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4-Bromo-*N*-(4-bromophenyl)benzenesulfonamide

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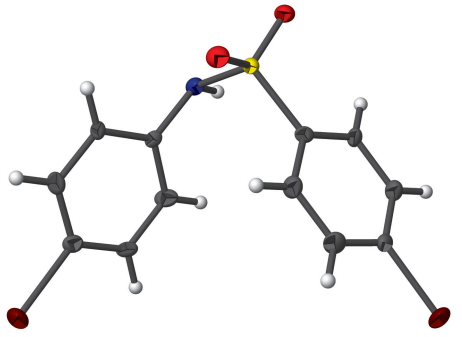
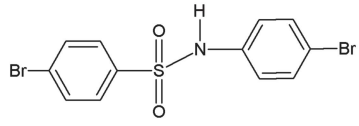
The molecule of the title compound, $C_{12}H_9Br_2NO_2S$, is U shaped with the central C—S—N—C segment having a torsion angle of 63.2 (4)°. Further, the dihedral angle between the benzene rings is 38.5 (2)°. The crystal structure features strong N—H···O hydrogen bonds that form infinite [100] *C*(4) chains. Molecules in adjacent chains are interlinked *via* C—H···O interactions which run along the *b* axis, forming *C*(7) chains. This results in a two-dimensional network in the *ab* plane; adjacent networks are connected by short Br···Br contacts [3.5092 (8) Å] propagating along the diagonal of the *ac* plane, so that a three-dimensional supramolecular architecture ensues.

3D view



Chemical scheme



Structure description

Sulfonamide drugs were the first chemotherapeutic agents to be used to cure and prevent bacterial infection in human beings (Shiva Prasad *et al.*, 2011). They play a vital role as key constituents in a number of biologically active molecules being known to exhibit a wide variety of biological activities such as antibacterial (Subhakara Reddy *et al.*, 2012), insecticidal (Himel *et al.*, 1971), antifungal (Hanafy *et al.*, 2007), antihepatitis (Yan-Fang *et al.*, 2010), anti-inflamatory (Küçükgüzel *et al.*, 2013), antitumor (Ghorab *et al.*, 2011), anticancer (Al-Said *et al.*, 2011), anti-HIV (Sahu *et al.*, 2007) and antitubercular activities (Vora *et al.*, 2012). In recent years, extensive research has been carried out on the synthesis and evaluation of the pharmacological activities of molecules containing the sulfonamide moiety, and they have been reported to be important pharmacophores (Mohan *et al.*, 2013). In this context and as part of our continued investigations of *N*-(4-substitutedphenyl)-4-bromobenzenesulfonamides (Vinola *et al.*, 2015), we report herein the crystal structure of *N*-(4-bromophenyl)-4-bromobenzenesulfonamide.

OPEN ACCESS

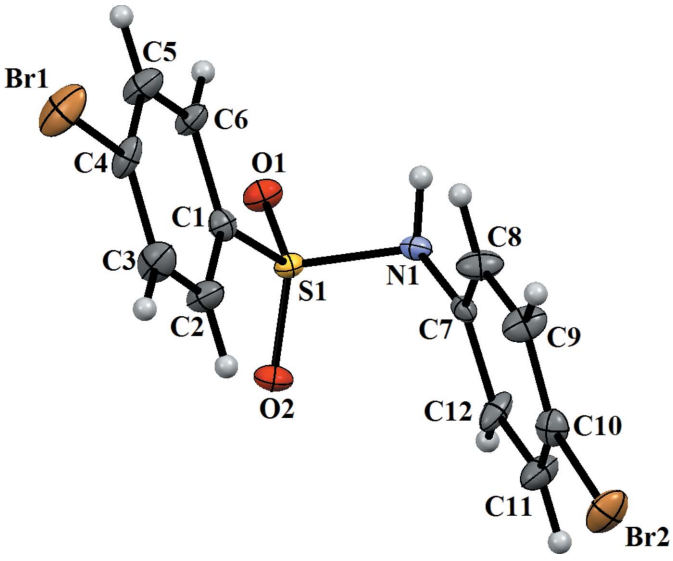


Figure 1
A view of the molecular structure of the compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

The molecule of the title compound (I) (Fig. 1) is U shaped, the central segment having a C1—S1—N1—C7 torsion angle of 63.2 (4)°. Further, the dihedral angle between the benzene rings is 38.5 (2)°.

The crystal structure features strong N1—H1···O2 hydrogen bonds (Table 1) that result in infinite [100] *C*(4) chains (Fig. 2). Molecules in adjacent chains are interlinked *via* C9—H9···O1 interactions which run along the *b* axis, forming *C*(7) chains. This results in a two-dimensional network in the *ab* plane (Fig. 3); adjacent networks are connected by short Br1···Br2 contacts [3.5092 (8) Å] propagating along the diagonal of the *ac* plane, so that a three-dimensional supramolecular architecture ensues (Fig. 4).

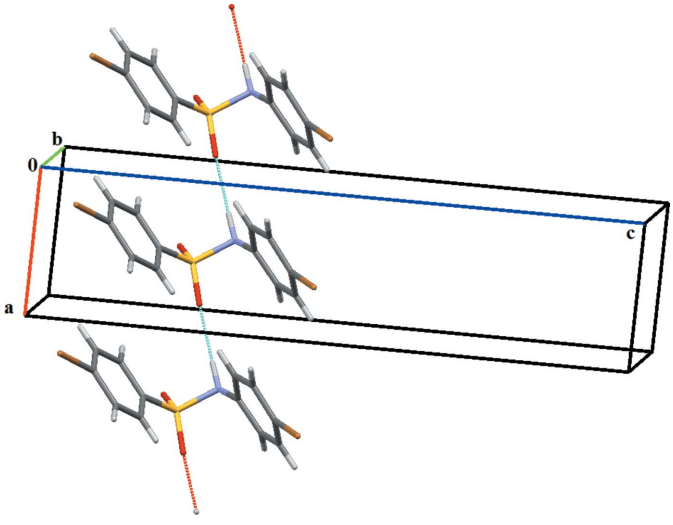


Figure 2
Crystal packing of the title compound, displaying the N—H···O chains running along [100].

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.90 (3)	2.06 (2)	2.945 (5)	170 (5)
C9—H9···O1^{ii}	0.93	2.48	3.238 (6)	138

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

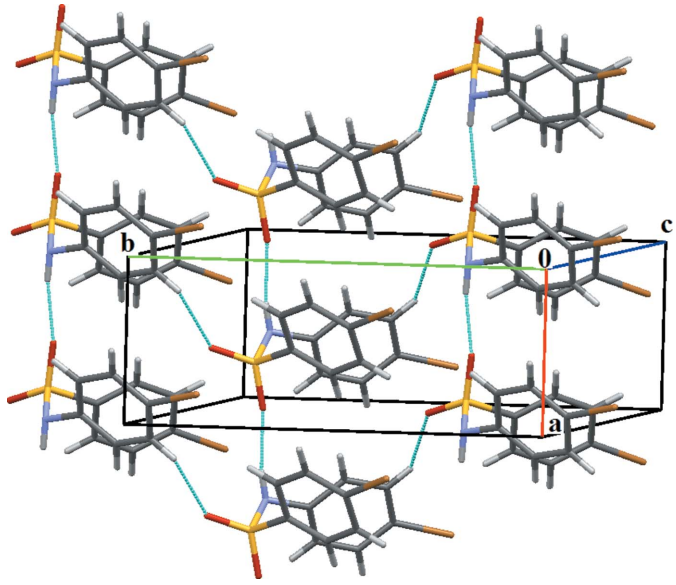


Figure 3
Formation of a two-dimensional network in the *ab* plane *via* N—H···O hydrogen bonds and C—H···O interactions.

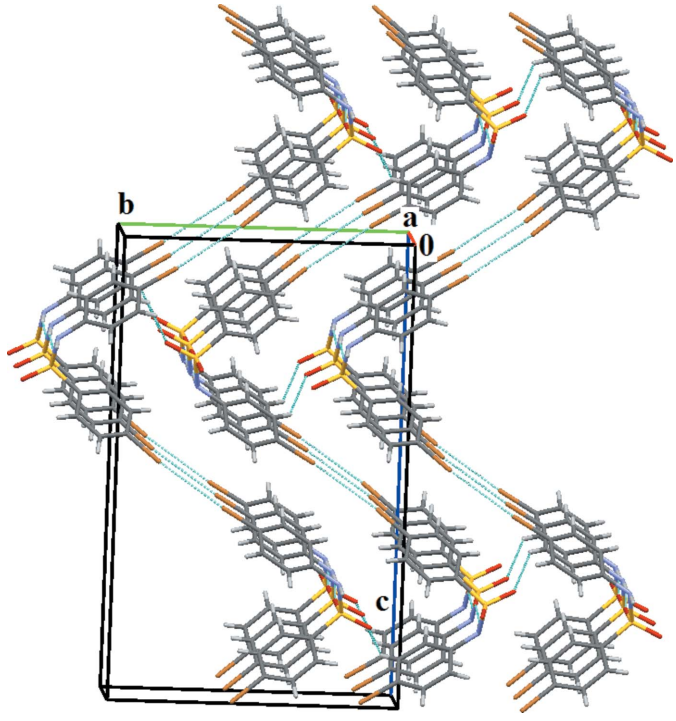


Figure 4
The three-dimensional architecture in the crystal structure.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_9Br_2NO_2S$
M_r	391.08
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, b, c (Å)	5.0643 (3), 12.8006 (7), 20.5540 (11)
β (°)	91.076 (2)
V (Å^3)	1332.20 (13)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	9.14
Crystal size (mm)	0.28 × 0.27 × 0.22
Data collection	
Diffractometer	Bruker APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.184, 0.238
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9589, 2142, 2092
R_{int}	0.050
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.585
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.061, 0.181, 1.16
No. of reflections	2142
No. of parameters	167
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.15, −1.26

Computer programs: *APEX2*, *SAINT-Plus* and *XPREP* (Bruker, 2009), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

The dihedral angle between the benzene rings in 4-bromo-*N*-(4-nitrophenyl)-benzenesulfonamide (II) (Vinola *et al.*, 2015) is slightly less than that in (I), being 32.6 (6)°. The central segment C1—S1—N1—C7 has a torsion angle of −64.2 (3)° in (II), compared to 63.2 (4)° in (I). Similar to (I), the crystal structure of (II) displays a three-dimensional architecture. A structure-directing N—H···O hydrogen bond and three different structure-directing C—H···O interactions along with weak C—Br···O interactions, consolidate the crystal structure of (II) into a three dimensional architecture.

Synthesis and crystallization

Compound (I) was prepared according to the literature method (Vinola *et al.*, 2015). The purity of the compound was checked by determining its melting point. Prismatic single crystals of (I) suitable for X-ray diffraction study were obtained by slow evaporation of an ethanolic solution of (I) at room temperature.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. To improve considerably the values of *R*1, *wR*2, and GOOF, the bad (132), (105) and (024) reflections were omitted from the final refinement.

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full crystallographic data

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4-Bromo-*N*-(4-bromophenyl)benzenesulfonamide

Crystal data

$C_{12}H_9Br_2NO_2S$
M_r = 391.08
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 5.0643 (3) Å
b = 12.8006 (7) Å
c = 20.5540 (11) Å
β = 91.076 (2)°
V = 1332.20 (13) Å^3
Z = 4
F(000) = 760

Prism
D_x = 1.950 Mg m^{-3}
Melting point: 413 K
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 167 reflections
θ = 4.1–64.5°
μ = 9.14 mm^{-1}
T = 296 K
Prism, colourless
0.28 × 0.27 × 0.22 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 1 pixels mm^{-1}
phi and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.184, T_{max} = 0.238

9589 measured reflections
2142 independent reflections
2092 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 64.5°, θ_{min} = 4.1°
h = −5→5
k = −14→14
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.181
S = 1.16
2142 reflections
167 parameters
1 restraint
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1331P)^2 + 1.7178P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.15 e Å^{-3}
$\Delta\rho_{min}$ = −1.26 e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5612 (9)	0.6558 (3)	0.1853 (2)	0.0144 (9)
C2	0.6912 (9)	0.5603 (4)	0.1886 (2)	0.0212 (10)
H2	0.8316	0.5506	0.2178	0.025*
C3	0.6100 (11)	0.4798 (4)	0.1481 (3)	0.0246 (11)
H3	0.6959	0.4157	0.1497	0.030*
C4	0.3999 (10)	0.4956 (3)	0.1053 (2)	0.0195 (10)
C5	0.2722 (10)	0.5905 (4)	0.1016 (2)	0.0225 (10)
H5	0.1330	0.5999	0.0720	0.027*
C6	0.3507 (10)	0.6715 (4)	0.1418 (2)	0.0174 (10)
H6	0.2646	0.7356	0.1398	0.021*
C7	0.5220 (8)	0.6491 (3)	0.3422 (2)	0.0122 (9)
C8	0.3527 (9)	0.5641 (4)	0.3350 (3)	0.0227 (10)
H8	0.2089	0.5684	0.3063	0.027*
C9	0.3960 (10)	0.4734 (4)	0.3701 (3)	0.0218 (10)
H9	0.2810	0.4172	0.3655	0.026*
C10	0.6108 (9)	0.4672 (3)	0.4119 (2)	0.0187 (10)
C11	0.7797 (10)	0.5524 (4)	0.4209 (2)	0.0230 (10)
H11	0.9224	0.5483	0.4500	0.028*
C12	0.7313 (10)	0.6431 (4)	0.3860 (2)	0.0201 (10)
H12	0.8412	0.7006	0.3921	0.024*
N1	0.4784 (7)	0.7427 (3)	0.30503 (18)	0.0142 (8)
O1	0.5684 (6)	0.8555 (2)	0.21164 (16)	0.0192 (7)
O2	0.9253 (6)	0.7423 (2)	0.25806 (16)	0.0189 (7)
S1	0.65243 (19)	0.75847 (8)	0.23895 (5)	0.0125 (4)
Br1	0.28679 (13)	0.38445 (4)	0.05096 (3)	0.0339 (3)
Br2	0.68273 (11)	0.34108 (4)	0.45686 (2)	0.0271 (3)
H1	0.312 (4)	0.751 (4)	0.290 (2)	0.010 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.015 (2)	0.013 (2)	0.016 (2)	−0.0018 (16)	0.0027 (17)	0.0005 (16)
C2	0.023 (2)	0.021 (2)	0.019 (2)	0.0029 (19)	−0.0061 (18)	−0.0004 (19)
C3	0.035 (3)	0.017 (2)	0.022 (3)	0.0076 (19)	−0.002 (2)	−0.0035 (19)
C4	0.033 (3)	0.015 (2)	0.011 (2)	−0.0025 (19)	−0.0005 (18)	−0.0009 (17)
C5	0.031 (3)	0.019 (2)	0.018 (3)	0.0024 (19)	−0.0083 (19)	0.0000 (19)

C6	0.024 (2)	0.016 (2)	0.012 (2)	0.0034 (17)	−0.0042 (18)	−0.0014 (16)
C7	0.010 (2)	0.014 (2)	0.013 (2)	0.0003 (16)	0.0024 (17)	−0.0034 (16)
C8	0.019 (2)	0.017 (2)	0.032 (3)	−0.0026 (18)	−0.0089 (19)	0.0001 (19)
C9	0.026 (2)	0.013 (2)	0.026 (3)	−0.0046 (18)	−0.0082 (19)	−0.0004 (18)
C10	0.023 (2)	0.014 (2)	0.019 (2)	0.0010 (17)	0.0021 (18)	−0.0022 (17)
C11	0.026 (2)	0.025 (2)	0.018 (2)	−0.0054 (19)	−0.0073 (19)	0.0050 (19)
C12	0.028 (3)	0.020 (2)	0.012 (2)	−0.0113 (19)	−0.0055 (19)	0.0026 (18)
N1	0.0108 (18)	0.0145 (17)	0.017 (2)	0.0023 (13)	−0.0016 (15)	−0.0013 (15)
O1	0.0214 (17)	0.0144 (15)	0.0215 (18)	−0.0005 (12)	−0.0043 (13)	0.0010 (13)
O2	0.0117 (15)	0.0205 (15)	0.0245 (18)	−0.0008 (11)	−0.0030 (12)	−0.0001 (13)
S1	0.0116 (6)	0.0123 (6)	0.0135 (6)	−0.0008 (3)	−0.0022 (4)	0.0006 (4)
Br1	0.0600 (5)	0.0180 (5)	0.0234 (5)	0.0004 (2)	−0.0122 (3)	−0.00819 (19)
Br2	0.0410 (5)	0.0172 (5)	0.0228 (4)	0.00183 (18)	−0.0048 (3)	0.00593 (18)

Geometric parameters (Å, º)

C1—C2	1.389 (7)	C7—N1	1.435 (6)
C1—C6	1.393 (7)	C8—C9	1.382 (7)
C1—S1	1.772 (4)	C8—H8	0.9300
C2—C3	1.381 (7)	C9—C10	1.375 (7)
C2—H2	0.9300	C9—H9	0.9300
C3—C4	1.382 (7)	C10—C11	1.396 (7)
C3—H3	0.9300	C10—Br2	1.892 (5)
C4—C5	1.377 (7)	C11—C12	1.385 (7)
C4—Br1	1.892 (5)	C11—H11	0.9300
C5—C6	1.380 (7)	C12—H12	0.9300
C5—H5	0.9300	N1—S1	1.646 (4)
C6—H6	0.9300	N1—H1	0.895 (10)
C7—C12	1.380 (7)	O1—S1	1.425 (3)
C7—C8	1.391 (6)	O2—S1	1.445 (3)
C2—C1—C6	121.0 (4)	C7—C8—H8	119.7
C2—C1—S1	120.3 (4)	C10—C9—C8	119.4 (4)
C6—C1—S1	118.7 (3)	C10—C9—H9	120.3
C3—C2—C1	119.4 (4)	C8—C9—H9	120.3
C3—C2—H2	120.3	C9—C10—C11	120.9 (4)
C1—C2—H2	120.3	C9—C10—Br2	119.8 (3)
C2—C3—C4	119.4 (4)	C11—C10—Br2	119.3 (4)
C2—C3—H3	120.3	C12—C11—C10	118.9 (5)
C4—C3—H3	120.3	C12—C11—H11	120.5
C5—C4—C3	121.3 (4)	C10—C11—H11	120.5
C5—C4—Br1	119.6 (4)	C7—C12—C11	120.8 (4)
C3—C4—Br1	119.2 (4)	C7—C12—H12	119.6
C4—C5—C6	120.0 (5)	C11—C12—H12	119.6
C4—C5—H5	120.0	C7—N1—S1	117.5 (3)
C6—C5—H5	120.0	C7—N1—H1	114 (3)
C5—C6—C1	118.9 (4)	S1—N1—H1	103 (3)
C5—C6—H6	120.5	O1—S1—O2	120.63 (19)

C1—C6—H6	120.5	O1—S1—N1	105.77 (19)
C12—C7—C8	119.3 (4)	O2—S1—N1	106.41 (19)
C12—C7—N1	120.2 (4)	O1—S1—C1	109.2 (2)
C8—C7—N1	120.5 (4)	O2—S1—C1	107.4 (2)
C9—C8—C7	120.7 (4)	N1—S1—C1	106.58 (19)
C9—C8—H8	119.7		

C6—C1—C2—C3	−0.1 (7)	Br2—C10—C11—C12	177.6 (4)
S1—C1—C2—C3	177.3 (4)	C8—C7—C12—C11	2.3 (7)
C1—C2—C3—C4	−0.4 (7)	N1—C7—C12—C11	−178.4 (4)
C2—C3—C4—C5	0.9 (8)	C10—C11—C12—C7	−0.9 (7)
C2—C3—C4—Br1	−179.1 (4)	C12—C7—N1—S1	82.2 (5)
C3—C4—C5—C6	−1.0 (8)	C8—C7—N1—S1	−98.5 (4)
Br1—C4—C5—C6	179.0 (4)	C7—N1—S1—O1	179.3 (3)
C4—C5—C6—C1	0.5 (7)	C7—N1—S1—O2	−51.3 (4)
C2—C1—C6—C5	0.0 (7)	C7—N1—S1—C1	63.2 (4)
S1—C1—C6—C5	−177.5 (4)	C2—C1—S1—O1	158.4 (4)
C12—C7—C8—C9	−1.5 (7)	C6—C1—S1—O1	−24.1 (4)
N1—C7—C8—C9	179.2 (4)	C2—C1—S1—O2	25.9 (4)
C7—C8—C9—C10	−0.8 (8)	C6—C1—S1—O2	−156.6 (4)
C8—C9—C10—C11	2.3 (8)	C2—C1—S1—N1	−87.8 (4)
C8—C9—C10—Br2	−176.8 (4)	C6—C1—S1—N1	89.7 (4)
C9—C10—C11—C12	−1.4 (7)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.90 (3)	2.06 (2)	2.945 (5)	170 (5)
C9—H9···O1^{ii}	0.93	2.48	3.238 (6)	138

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