



Received 23 October 2018

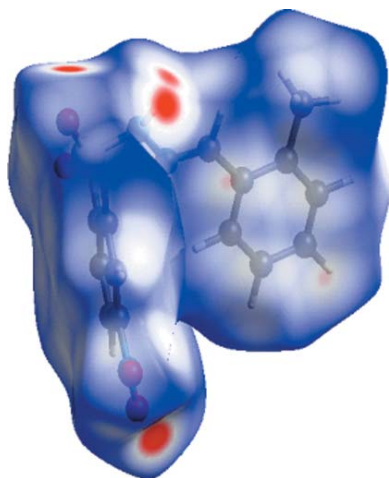
Accepted 28 October 2018

Edited by H. Stoeckli-Evans, University of
Neuchâtel, Switzerland**Keywords:** crystal structure; *N*-(arylidene)-aryl-
sulfonohydrazides; hydrogen bonding; ring
motif; Hirshfeld surface analysis; fingerprint
plots.**CCDC references:** 1578712; 1578713;
1578715**Supporting information:** this article has
supporting information at journals.iucr.org/e

Crystal structures and the Hirshfeld surface analysis of (*E*)-4-nitro-*N'*-(*o*-chloro, *o*- and *p*-methylbenzylidene)benzenesulfonohydrazides

Akshatha R. Salian,^a Sabine Foro^b and B. Thimme Gowda^{a,c,*}^aDepartment of Chemistry, Mangalore University, Mangalagangothri-574 199, India, ^bInstitute of Materials Science, Darmstadt University of Technology, Alarich-Weiss-Str. 2, D-64287, Darmstadt, Germany, and ^cKarnataka State Rural Development and Panchayat Raj University, Raitha Bhavan, Gadag-582101, India. *Correspondence e-mail: gowdabt@yahoo.com

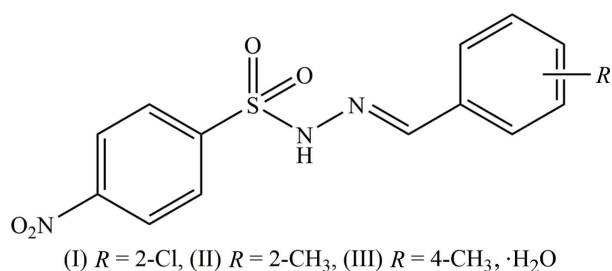
The crystal structures of (*E*)-*N'*-(2-chlorobenzylidene)-4-nitrobenzenesulfonohydrazide, C₁₃H₁₀ClN₃O₄S (I), (*E*)-*N'*-(2-methylbenzylidene)-4-nitrobenzenesulfonohydrazide, C₁₄H₁₃N₃O₄S (II), and (*E*)-*N'*-(4-methylbenzylidene)-4-nitrobenzenesulfonohydrazide monohydrate, C₁₄H₁₃N₃O₄S·H₂O (III), have been synthesized, characterized and their crystal structures determined to study the effects of the nature and sites of substitutions on the structural parameters and the hydrogen-bonding interactions. All three compounds crystallize in the monoclinic crystal system, with space group *P*2₁ for (I) and *P*2₁/*c* for (II) and (III). Compound (III) crystallizes as a monohydrate. All three compounds adopt an *E* configuration around the C=N bond. The molecules are bent at the S atom with C–S–N–N torsion angles of –59.0 (3), 58.0 (2) and –70.2 (1)° in (I), (II) and (III), respectively. The sulfonohydrazide parts are also non-linear, as is evident from the S–N–N–C torsional angles of 159.3 (3), –164.2 (1) and 152.3 (1)° in (I), (II) and (III), respectively, while the hydrazide parts are almost planar with the N–N=C–C torsion angles being –179.1 (3)° in (I), 176.7 (2)° in (II) and 175.0 (2)° in (III). The 4-nitro-substituted phenylsulfonyl and 2/4-substituted benzylidene rings are inclined to each other by 81.1 (1)° in (I), 81.4 (1)° in (II) and 74.4 (1)° in (III). The compounds show differences in hydrogen-bonding interactions. In the crystal of (I), molecules are linked *via* N–H···O hydrogen bonds, forming *C*(4) chains along the *a*-axis direction that are interconnected by weak C–H···O hydrogen bonds, generating layers parallel to the *ac* plane. In the crystal of (II), the amino H atom shows bifurcated N–H···O(O) hydrogen bonding with both O atoms of the nitro group generating *C*(9) chains along the *b*-axis direction. The chains are linked by weak C–H···O hydrogen bonds, forming a three-dimensional framework. In the crystal of (III), molecules are linked by O_w–H···O, N–H···O_w and C–H···O hydrogen bonds, forming layers lying parallel to the *bc* plane. The fingerprint plots generated for the three compounds show that for (I) and (II) the O···H/H···O contacts make the largest contributions, while for the *para*-substituted compound (III), H···H contacts are the major contributors to the Hirshfeld surfaces.



1. Chemical context

Sulfonyl hydrazides have been used extensively to synthesize new Schiff bases owing to the presence of two chemically and biologically important sulfonyl and hydrazine moieties (Murtaza *et al.*, 2016). Reactions of hydrazines with other functional groups produce compounds with unique physical and chemical characteristics (Xavier *et al.*, 2012). Hydrazones derived from the condensation reactions of hydrazides with aldehydes show excellent biological properties (Küçükgül *et*

al., 2006). As a result of the ease of the electron-transport mechanism through the π -conjugated framework, the azomethine-bridged benzene derivatives exhibit excellent optical non-linearities (Manivannan & Dhanuskodi, 2004). Organic polymers containing the azomethine group are known to have good mechanical strength (Morgan *et al.*, 1987) and high thermal stability (Catanescu *et al.*, 2001). As part of our continuing studies to explore the effect of the nature and site of substituents on the crystal structures of sulfonyl hydrazide derivatives (Salian *et al.*, 2018), we report herein the synthesis, crystal structures and Hirshfeld surface analyses of the title compounds, (*E*)-4-nitro-*N'*-(2-chlorobenzylidene)benzenesulfonylhydrazide (I), (*E*)-4-nitro-*N'*-(2-methylbenzylidene)benzenesulfonylhydrazide (II) and (*E*)-4-nitro-*N'*-(4-methylbenzylidene)benzenesulfonylhydrazide monohydrate (III).



2. Structural commentary

The title compounds crystallize in the monoclinic crystal system, in space group $P2_1$ for (I) and $P2_1/c$ for (II) and (III). The molecular structures of the three compounds are shown in Figs. 1, 2 and 3. Compound (III) crystallizes as a monohydrate. In all three compounds the configuration about the C=N bond is *E*, with C7=N2 bond lengths of 1.269 (5), 1.275 (2) and 1.263 (3) Å in (I), (II) and (III), respectively. The respective N1–N2 bond lengths of 1.404 (4), 1.400 (2) and 1.398 (2) Å are consistent with the azine bond lengths of

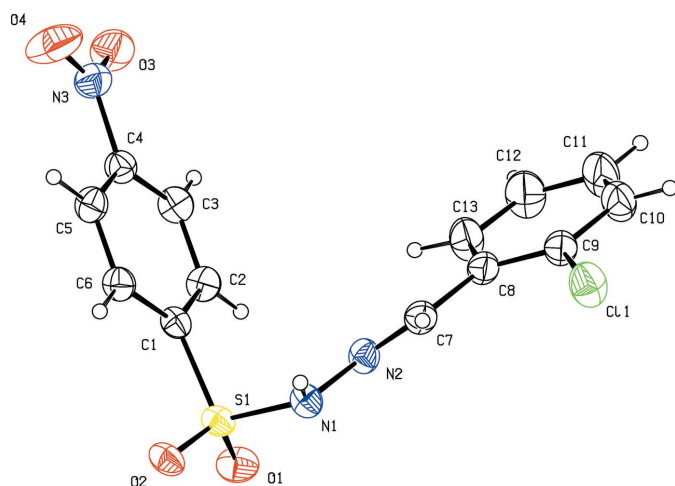


Figure 1

Molecular structure of compound (I), showing the atom labelling and displacement ellipsoids drawn at the 30% probability level.

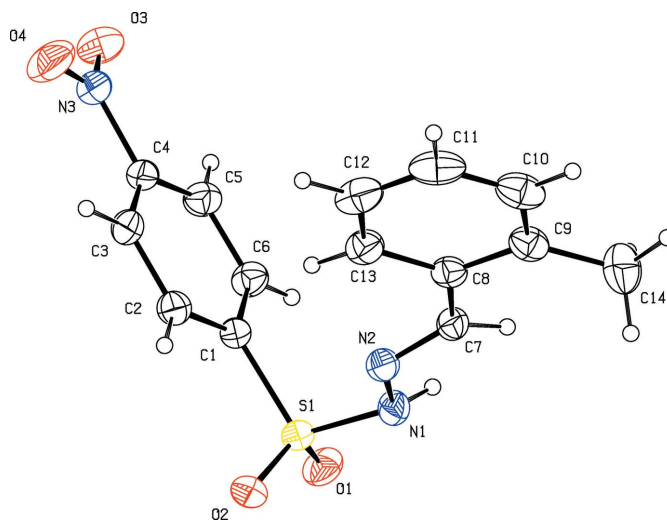


Figure 2

Molecular structure of compound (II), showing the atom labelling and displacement ellipsoids drawn at the 30% probability level.

1.40 Å in similar structures (Salian *et al.*, 2018), indicating the delocalization of π -electron density over the hydrazone portion of the molecules. The conformation of the N–H and C–H bonds in (I) and (II), with respect to the *ortho*-substituents, are *syn* to each other (Figs. 1 and 2). In the central parts of each molecule the S1–N1–N2=C7 torsion angles deviate from linearity with values of 159.3 (3)° in (I), –164.2 (1)° in (II) and 152.3 (1)° in (III), while the hydrazide parts are almost planar with the N1–N2=C7–C8 torsion angles being –179.1 (3), 176.7 (2) and 175.0 (2)° in (I), (II) and (III), respectively. The dihedral angles between the 4-nitrobenzene ring (C1–C6) and benzene ring (C8–C13) are 81.1 (1), 81.4 (1) and 74.4 (1)°, respectively. The plane of the nitro group (N3/O3/O4) is inclined to the 4-nitrobenzene ring (C1–C6) by 9.3 (5)° in (I) and 9.1 (3)° in (II), but is significantly out of plane in (III) with a dihedral angle of 16.1 (2)°.

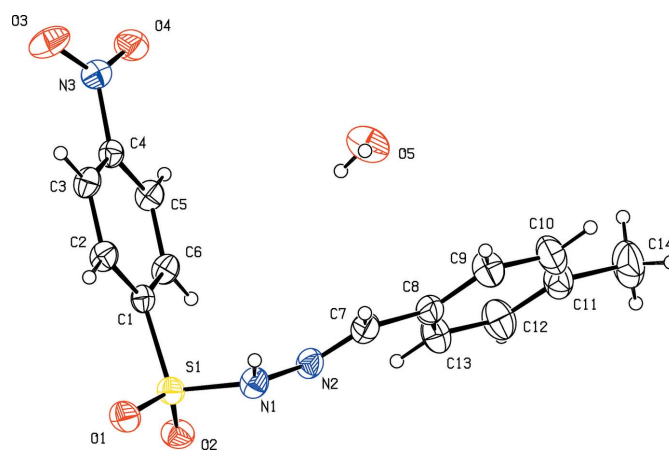


Figure 3

Molecular structure of compound (III), showing the atom labelling and displacement ellipsoids drawn at the 30% probability level.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O1^i$	0.86 (2)	2.02 (3)	2.853 (4)	163 (4)
$C3-H3\cdots O2^{ii}$	0.93	2.46	3.290 (4)	149

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

3. Supramolecular features

In the crystals of the title compounds there are significant difference in the hydrogen-bonding interactions. In the crystal of the *ortho*-chloro-substituted compound (I), molecules are linked *via* $N-H\cdots O$ hydrogen bonds, forming C_4 chains along the a -axis direction (Table 1 and Fig. 4). These chains are interconnected by weak $C-H\cdots O$ hydrogen bonds, generating layers parallel to the ab plane (Table 1 and Fig. 5). In the crystal of the *ortho*-methyl-substituted compound (II), the amino H atom shows bifurcated $N-H\cdots O(O)$ hydrogen

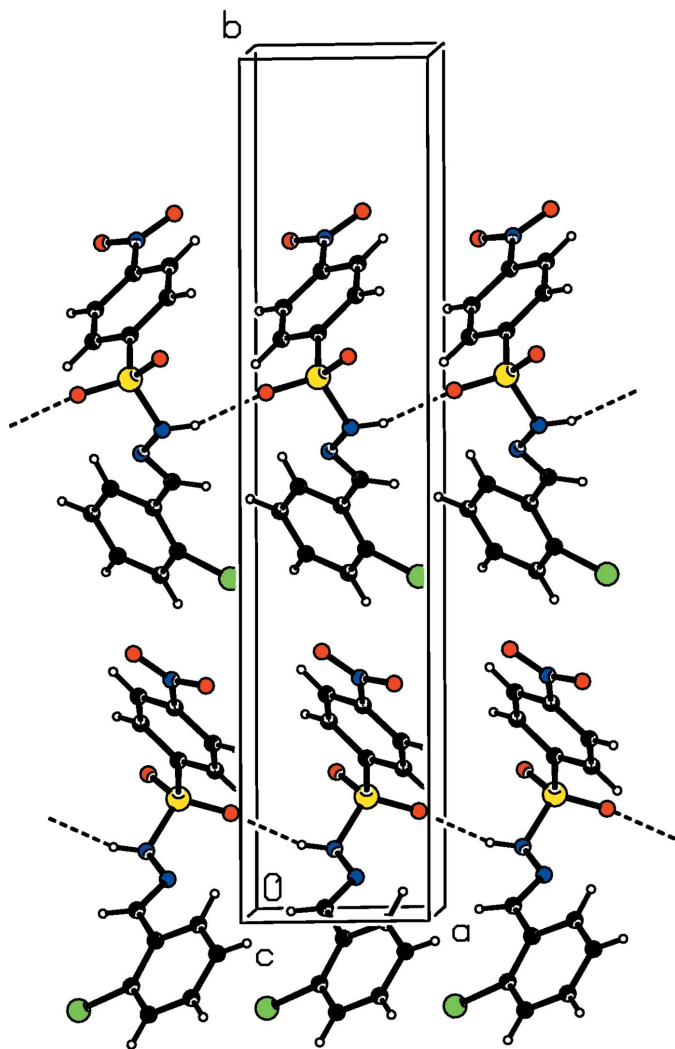


Figure 4
A partial view along the c axis of the crystal packing of compound (I), with hydrogen bonds shown as dashed lines.

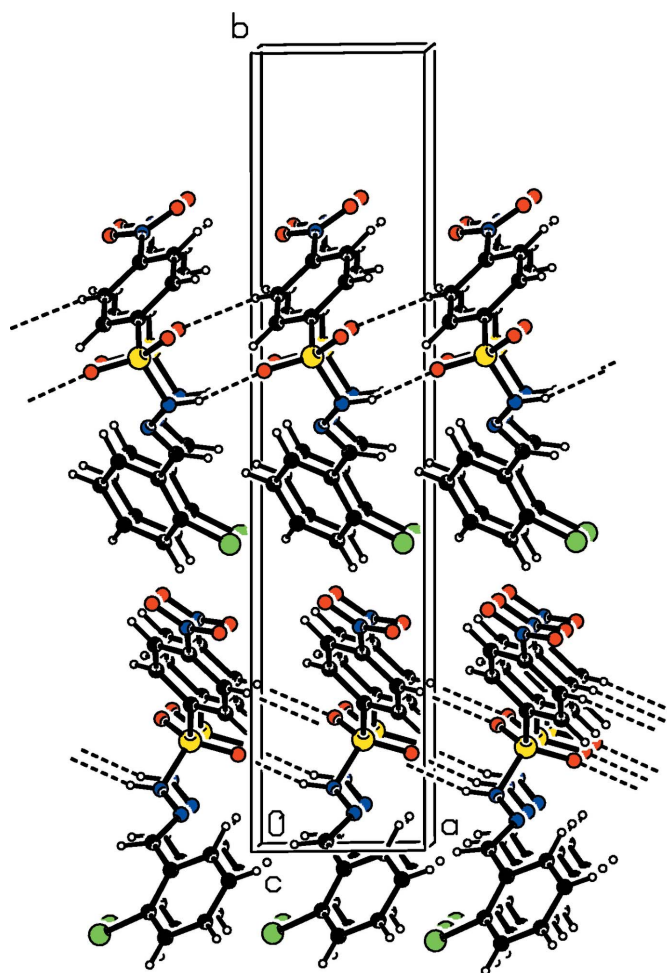


Figure 5
The crystal packing of compound (I), viewed along the c axis, with hydrogen bonds shown as dashed lines.

bonding with both the O atoms of the nitro group, generating chains with a $C(9)$ motif that propagate along the b -axis direction (Table 2 and Fig. 6). These chains are linked by $C-H\cdots O$ hydrogen bonds, resulting in the formation of a three-dimensional framework (Table 2 and Fig. 7). Finally, in the crystal of the *para*-methyl-substituted compound (III), the

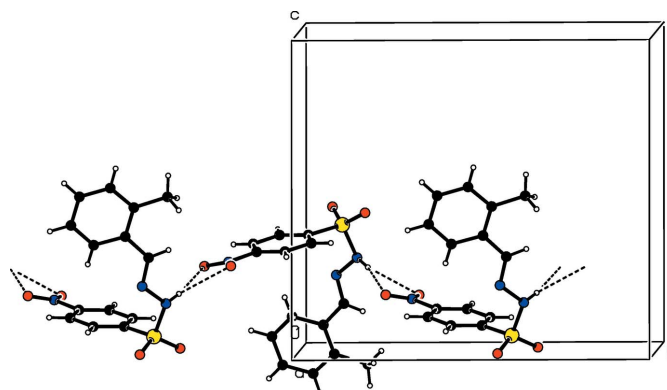


Figure 6
A partial view along the a axis of the crystal packing of compound (II), with hydrogen bonds shown as dashed lines.

Table 2
 Hydrogen-bond geometry (Å, °) for (II).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O3^i$	0.84 (2)	2.51 (2)	3.230 (2)	144 (2)
$N1-H1N\cdots O4^i$	0.84 (2)	2.44 (2)	3.260 (3)	164 (2)
$C2-H2\cdots O2^{ii}$	0.93	2.58	3.284 (2)	133
$C12-H12\cdots O1^{iii}$	0.93	2.44	3.341 (3)	164

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

Table 3
 Hydrogen-bond geometry (Å, °) for (III).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O5^i$	0.85 (2)	2.00 (2)	2.848 (2)	173 (2)
$O5-H51\cdots O2^{ii}$	0.81 (2)	2.29 (2)	3.006 (2)	148 (3)
$O5-H52\cdots O1^{iii}$	0.80 (2)	2.17 (2)	2.949 (2)	166 (3)
$C5-H5\cdots O1^{iii}$	0.93	2.52	3.167 (2)	127

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

presence of the water molecule of crystallization has an important effect on the crystal packing. The molecules of compound (III) are bridged by the water molecule, *via* $Ow-$

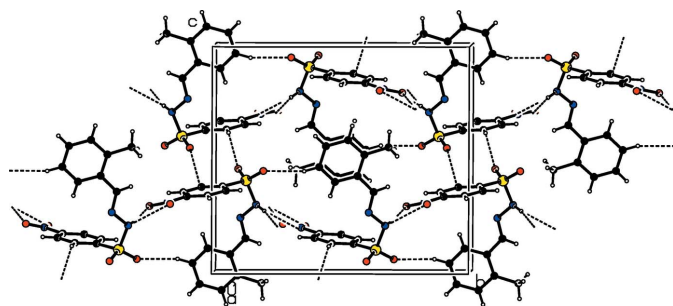
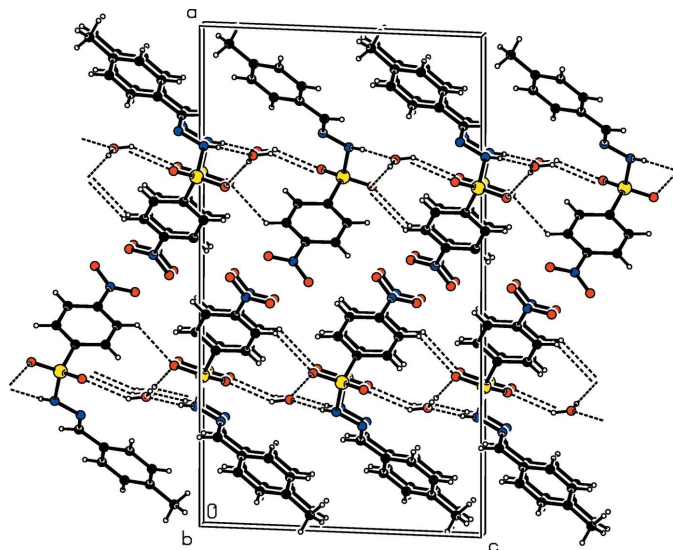

Figure 7
 The crystal packing of compound (II), viewed along the a axis, with hydrogen bonds shown as dashed lines.

Figure 8
 The crystal packing of compound (III), viewed along the b axis, with hydrogen bonds shown as dashed lines.

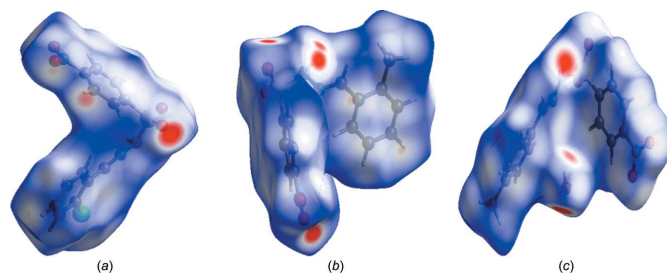
Table 4
 Hirshfeld contact interactions (%).

Contact type	(I)	(II)	(III)
$O\cdots H/H\cdots O$	35.0	37.3	32.0
$H\cdots H$	17.5	28.4	37.2
$C\cdots H/H\cdots C$	17.3	13.4	11.0
$O\cdots C/C\cdots O$	4.3	1.8	9.4
$C\cdots C$	1.0	7.8	0.3
$N\cdots H/H\cdots N$	4.3	7.3	5.0
$N\cdots C/C\cdots N$	2.2	0.1	1.2
$O\cdots N/N\cdots O$	1.1	1.4	1.4
$O\cdots O$	1.9	2.4	0.0
$S\cdots C/C\cdots S$	0.0	0.1	0.1
$Cl\cdots C/C\cdots Cl$	4.7	-	-
$Cl\cdots H/H\cdots Cl$	6.1	-	-
$Cl\cdots O/O\cdots Cl$	3.1	-	-
$Cl\cdots N/N\cdots Cl$	1.4	-	-

$H\cdots O$ and $N-H\cdots Ow$ hydrogen bonds, forming layers lying parallel to the bc plane that are reinforced by $C-H\cdots O$ hydrogen bonds (Table 3 and Fig. 8).

4. Hirshfeld surface analysis

The type of intermolecular contacts and their quantitative contribution to the crystal packing in all the three compounds were studied by Hirshfeld surfaces and two-dimensional fingerprint plots, which were generated using *Crystal-Explorer3.1* (Wolff *et al.*, 2012). The Hirshfeld surfaces mapped over d_{norm} are in the scale of $-0.56-1.43$ a.u. The bright-red spots on the Hirshfeld surfaces mapped over d_{norm} indicate the strong $N-H\cdots O$ interactions present in the crystal structure (McKinnon *et al.*, 2004; Spackman & Jayatilaka, 2009); these correspond to $N1-H1N\cdots O1^i$ in (I) (Fig. 9a; Table 1), $N1-H1N\cdots O3^i$ and $N1-H1N\cdots O4^i$ in (II) (Fig. 9b; Table 2) and $N1-H1N\cdots O5^i$, $O5-H51\cdots O2^{ii}$ and $O5-H52\cdots O1^{iii}$ in (III) (Fig. 9c; Table 3). The fingerprint plots corresponding to the various contacts contributing more than 10% (along with the $C\cdots C$ contacts) to the Hirshfeld surfaces are shown in Fig. 10. Table 4 lists all the contacts present in the crystal structures of the three compounds, and their respective percentage contributions to the Hirshfeld surfaces. The $O\cdots H/H\cdots O$ contacts correspond to the $N-H\cdots O/O-H\cdots O$ interactions at $d_e + d_i \sim 2.2$ Å in (I) and (III) and at 2.6 Å in (II), which is very close to the hydrogen-bonding


Figure 9
 View of the Hirshfeld surface mapped over d_{norm} for (a) (I), (b) (II) and (c) (III).

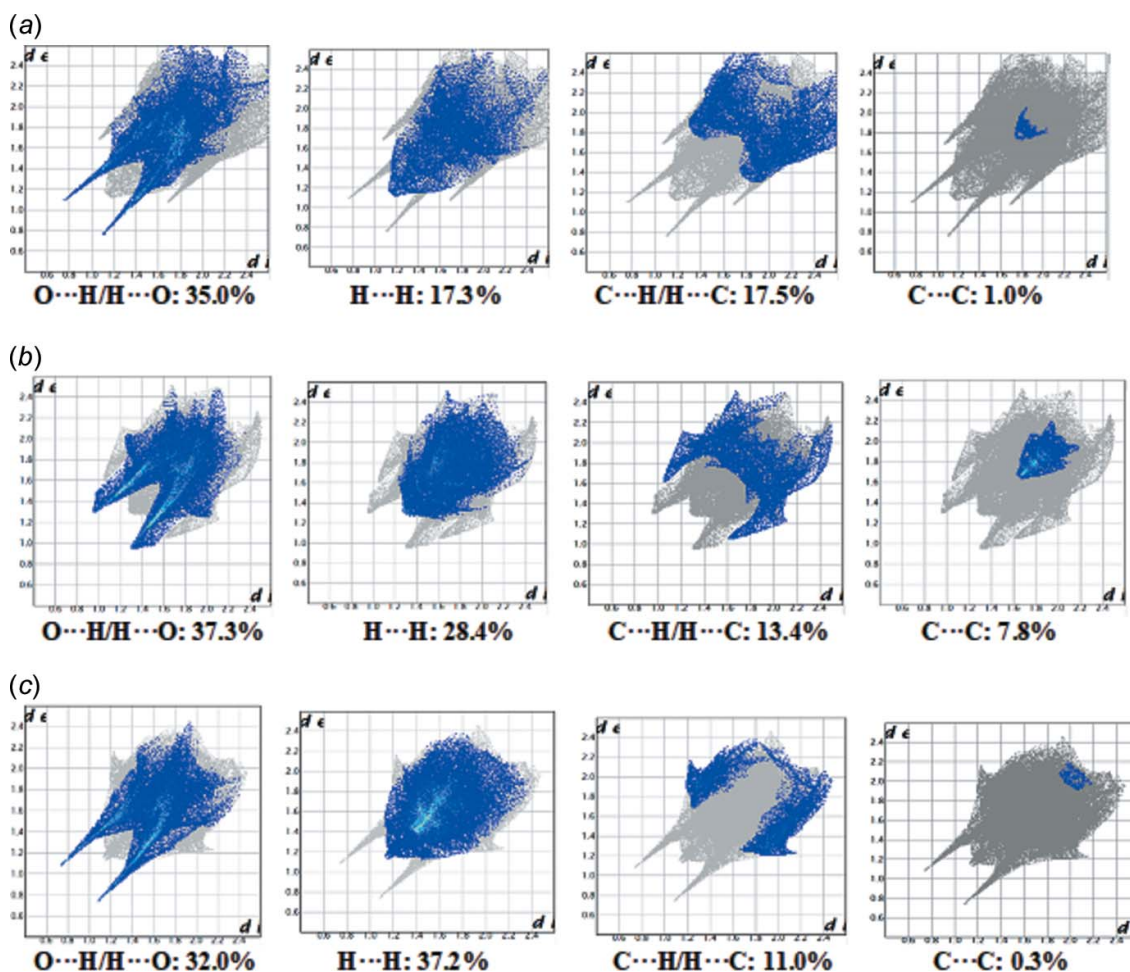


Figure 10
Two-dimensional fingerprint plots showing the contributions of the different types of interactions in (a) (I), (b) (II) and (c) (III).

distances observed in these compounds [Tables 1, 2 and 3 for (I), (II) and (III), respectively]. These interactions are the major contributor in (I) and (II) [35.0% in (I) and 37.3% in (II)] followed by H...H contacts [17.5% in (I) and 28.4% in (II)]. In (III), H...H interactions make the largest contribution to the Hirshfeld surface (37.2%), followed by O...H/H...O contacts (32.0%). The H...H interactions appear as a short single peak at $d_e + d_i \sim 2.2$ Å in the fingerprint plot of (III) (see Fig. 10c). The distinct pair of wings corresponds to C...H/H...C contacts, which are the third largest contributor to the Hirshfeld surfaces in all three compounds [17.3% in (I), 13.4% in (II) and 11.0% in (III)]. A significant difference is in the percentage contribution from C...C contacts found for the three compounds. They are characterized by two overlapping broad peaks in the fingerprint plot for (II) (see Fig. 10b), accounting for 7.8% of the Hirshfeld surface with $d_e + d_i \sim 3.4$ Å, whereas for (I) and (III) these interactions make negligible contributions of 1.0 and 0.3%, respectively. The O...C/C...O contacts contribute 4.3% in (I), 1.8% in (II) and 9.4% in (III). N...H/H...N contacts contribute 4.3, 7.3 and 5.0% in (I), (II) and (III), respectively. In (I), the Cl...H/H...Cl, Cl...C/C...Cl, Cl...O/O...Cl and Cl...N/N...Cl contacts contribute 6.1, 4.7, 3.1 and 1.4%, respectively, to the

Hirshfeld surfaces. The percentage contributions for the various interactions in the title compounds are compared in Table 4.

5. Database survey

Structures similar to the title compounds that have been reported in the literature include *N'*-[(*E*)-4-methylbenzylidene]-4-methylbenzenesulfonohydrazide (Tabatabaee *et al.*, 2007), (*E*)-*N'*-(4-chlorobenzylidene)-*p*-toluenesulfonohydrazide 0.15-hydrate (Kia *et al.*, 2009a), (*E*)-*N'*-(4-chlorobenzylidene)-4-methylbenzenesulfonohydrazide (Balaji *et al.*, 2014), (*E*)-*N'*-(4-bromobenzylidene)-*p*-toluenesulfonohydrazide (Kia *et al.*, 2009b), (*E*)-*N'*-(4-nitrobenzylidene)benzenesulfonohydrazide (Hussain *et al.*, 2017a), (*E*)-4-methyl-*N'*-(4-nitrobenzylidene)benzenesulfonohydrazide (Hussain *et al.*, 2017b). (*E*)-*N'*-(2-methylbenzylidene)-4-chlorobenzene-sulfonohydrazide and (*E*)-*N'*-(4-methylbenzylidene)-4-chlorobenzene-sulfonohydrazide (Salian *et al.*, 2018). In all the structures, intermolecular N—H...O hydrogen bonds link neighbouring molecules to form chains, which are linked by π – π interactions, further stabilizing the crystal structures. The chains are linked *via* C—H...O hydrogen bonds, forming

Table 5
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	C ₁₃ H ₁₀ ClN ₃ O ₄ S	C ₁₄ H ₁₃ N ₃ O ₄ S	C ₁₄ H ₁₃ N ₃ O ₄ S·H ₂ O
<i>M_r</i>	339.75	319.33	337.35
Crystal system, space group	Monoclinic, <i>P</i> ₂ ₁	Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>	Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>
Temperature (K)	293	293	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	4.9498 (6), 22.340 (3), 7.0003 (9)	7.190 (1), 15.288 (2), 13.596 (2)	22.589 (2), 5.4424 (4), 12.7180 (9)
β (°)	104.40 (1)	97.68 (2)	92.146 (6)
<i>V</i> (Å ³)	749.76 (17)	1481.1 (4)	1562.4 (2)
<i>Z</i>	2	4	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.42	0.24	0.24
Crystal size (mm)	0.24 × 0.24 × 0.12	0.50 × 0.48 × 0.24	0.40 × 0.36 × 0.16
Data collection			
Diffractometer	Oxford Diffraction Xcalibur single crystal X-ray diffractometer with Sapphire CCD detector	Oxford Diffraction Xcalibur single crystal X-ray diffractometer with Sapphire CCD detector	Oxford Diffraction Xcalibur single crystal X-ray diffractometer with Sapphire CCD detector
Absorption correction	Multi-scan (<i>CrysAlis RED</i> ; Oxford Diffraction, 2009)	Multi-scan (<i>CrysAlis RED</i> ; Oxford Diffraction, 2009)	Multi-scan (<i>CrysAlis RED</i> ; Oxford Diffraction, 2009)
<i>T</i> _{min} , <i>T</i> _{max}	0.907, 0.952	0.889, 0.945	0.911, 0.963
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	4582, 2696, 2457	9810, 2719, 2271	9384, 2870, 2178
<i>R</i> _{int}	0.015	0.020	0.021
(sin θ/λ) _{max} (Å ⁻¹)	0.602	0.602	0.602
Refinement			
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.030, 0.078, 1.01	0.038, 0.097, 1.05	0.034, 0.092, 1.02
No. of reflections	2696	2719	2870
No. of parameters	202	203	218
No. of restraints	2	2	6
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.14, -0.20	0.28, -0.32	0.19, -0.28
Absolute structure	Flack <i>x</i> determined using 1102 quotients [(<i>I</i> ⁺) - (<i>I</i> ⁻)] / [(<i>I</i> ⁺) + (<i>I</i> ⁻)] (Parsons <i>et al.</i> , 2013)	–	–
Absolute structure parameter	0.05 (3)	–	–

Computer programs: *CrysAlis CCD* and *CrysAlis RED* (Oxford Diffraction, 2009), *SHELXS2013* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

layers. This situation is similar to that in the recently reported structures of (*E*)-*N'*-benzylidene-4-chlorobenzenesulfonylhydrazine and the 2-methylbenzylidene derivative, (*E*)-*N'*-(2-methylbenzylidene)-4-chlorobenzenesulfonylhydrazine (Salian *et al.*, 2018).

6. Synthesis and crystallization

Synthesis of 4-nitrobenzenesulfonylhydrazide

Hydrazine hydrate (99%, 5 ml) was added to 4-nitrobenzenesulfonyl chloride (0.01 mol), dissolved in ethanol (30 ml) at 273 K under constant stirring. The stirring continued for 15 min at 273 K and then at 303 K for 3 h. The reaction mixture was then concentrated by evaporating off the excess ethanol. The solid product obtained, *i.e.* 4-nitrobenzenesulfonylhydrazide, was washed with cold water and dried.

Synthesis of the title compounds (I), (II) and (III)

The title compounds were synthesized by refluxing the mixtures of 4-nitrobenzenesulfonylhydrazide (0.01 mol) and 0.01 mol of 2-chlorobenzaldehyde for (I), 2-methylbenzaldehyde for (II), and 4-methylbenzaldehyde for (III), in

ethanol (30 ml) and two drops of glacial acetic acid for 4 h. The reaction mixtures were cooled to room temperature and concentrated by evaporating off the excess of solvent. The solid products obtained were washed with cold water, dried and recrystallized to constant melting points from ethanol. Purity of the compounds was checked by TLC. All three compounds were characterized by measuring their IR, ¹H and ¹³C NMR spectra.

(*E*)-*N'*-(2-chlorobenzylidene)-4-nitrobenzenesulfonylhydrazide (I)

Colourless prismatic crystals; m.p: 438–439 K; IR (cm⁻¹): 3182.5 (N–H *asym. stretch*), 1604.8 (C=N), 1311.6 (S=O *asym. stretch*) and 1168.9 (S=O *sym. stretch*).

¹H NMR (400 MHz, DMSO-*d*₆): δ 7.16–7.18 (*m*, 2H, Ar-H), 7.45–7.47 (*m*, 2H, Ar-H), 7.91 (*s*, 1H), 8.15 (*d*, 2H, Ar-H), 8.41 (*d*, 2H, Ar-H), 11.71 (*s*, 1H); ¹³C NMR (400 MHz, DMSO-*d*₆): δ 124.41, 126.67, 127.34, 128.75, 129.67, 130.55, 131.46, 133.08, 143.80, 144.27, 149.88.

(*E*)-*N'*-(2-methylbenzylidene)-4-nitrobenzenesulfonylhydrazide (II)

Yellow rod-shaped crystals; m.p: 417–418 K; IR (cm^{-1}): 3217.3 (N–H *asym. stretch*), 1602.9 (C=N), 1332.1 (S=O *asym. stretch*) and 1172.7 (S=O *sym. stretch*).

^1H NMR (400 MHz, DMSO- d_6): δ 2.31 (*s*, 3H), 7.34–7.37 (*m*, 3H, Ar-H), 7.40 (*t*, 1H, Ar-H), 7.81 (*d*, 1H, Ar-H), 8.16 (*d*, 1H, Ar-H), 8.31 (*s*, 1H), 8.44 (*d*, 2H, Ar-H), 12.10 (*s*, 1H). ^{13}C NMR (400 MHz, DMSO- d_6): δ 20.97, 124.21, 125.90, 126.78, 128.75, 129.01, 130.61, 131.68, 139.99, 144.48, 148.13, 149.74.

(E)-N'-(4-methylbenzylidene)-4-nitrobenzenesulfonohydrazide (III)

Yellow prismatic crystals; m.p: 447–448 K; IR (cm^{-1}): 3291.5 (N–H *asym. stretch*), 1606.7 (C=N), 1305.8 (S=O *asym. stretch*) and 1165.0 (S=O *sym. stretch*).

^1H NMR (400 MHz, DMSO- d_6): δ 2.31 (*s*, 3H), 7.34 (*d*, 2H, Ar-H), 7.60 (*d*, 2H, Ar-H), 8.16 (*d*, 2H, Ar-H), 8.30 (*s*, 1H), 8.42 (*d*, 2H, Ar-H), 12.03 (*s*, 1H). ^{13}C NMR (400 MHz, DMSO- d_6): δ 20.96, 123.81, 126.65, 128.59, 128.96, 130.47, 139.94, 144.64, 147.96, 149.58.

Single crystals of the title compounds used for the single-crystal X-ray diffraction analyses were obtained by slow evaporation of the solvent in their DMF solutions at room temperature.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. The C-bound H atoms were positioned with idealized geometry and refined using a riding model with the aromatic C–H = 0.93 Å. The amino H atoms were refined with an N–H distance restraint of 0.86 (2) Å. For (III), the H atoms of the water molecule were refined with the O–H distance restrained to 0.82 (2) Å. All H atoms were refined with isotropic displacement parameters set at $1.2U_{\text{eq}}$ (C-aromatic, N, O) and $1.5U_{\text{eq}}$ (C-methyl). For (I), the low angle reflection (0 $\bar{2}$ 1) had a poor agreement with its calculated value and was omitted from the refinement.

Acknowledgements

The authors thank SAIF Panjab University for extending the services of their NMR facility and Mangalore University for providing all the facilities required.

Funding information

ARS thanks the Department of Science and Technology, Government of India, New Delhi, for a research fellowship under its DST-PURSE Program and BTG thanks the University Grants Commission, Government of India, New Delhi, for a special grant under a UGC–BSR one-time grant to faculty.

References

Balaji, J., John Francis Xavier, J., Prabu, S. & Srinivasan, P. (2014). *Acta Cryst.* **E70**, o1250–o1251.
 Catanescu, O., Grigoras, M., Colotin, G., Dobreanu, A., Hurduc, N. & Simionescu, C. I. (2001). *Eur. Polym. J.* **37**, 2213–2216.
 Hussain, M. M., Rahman, M. M., Arshad, M. N. & Asiri, A. M. (2017a). *ACS Omega*, **2**, 420–431.
 Hussain, M. M., Rahman, M. M., Arshad, M. N. & Asiri, A. M. (2017b). *Sci. Rep.* **7**, 5832.
 Kia, R., Etemadi, B., Fun, H.-K. & Kargar, H. (2009b). *Acta Cryst.* **E65**, o821–o822.
 Kia, R., Fun, H.-K. & Kargar, H. (2009a). *Acta Cryst.* **E65**, o1119–o1120.
 Küçükgülzel, G., Kocatepe, A., De Clercq, E., Şahin, F. & Güllüce, M. (2006). *Eur. J. Med. Chem.* **41**, 353–359.
 Manivannan, S. & Dhanuskodi, S. (2004). *J. Cryst. Growth*, **262**, 473–478.
 McKinnon, J. J., Spackman, M. A. & Mitchell, A. S. (2004). *Acta Cryst.* **B60**, 627–668.
 Morgan, P. W., Kwolek, S. L. & Pletcher, T. C. (1987). *Macromolecules*, **20**, 729–739.
 Murtaza, S., Shamim, S., Kousar, N., Tahir, M. N., Sirajuddin, M. & Rana, U. A. (2016). *J. Mol. Struct.* **1107**, 99–108.
 Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.
 Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* **B69**, 249–259.
 Salian, A. R., Foro, S. & Gowda, B. T. (2018). *Acta Cryst.* **E74**, 1613–1618.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
 Spackman, M. A. & Jayatilaka, D. (2009). *CrystEngComm*, **11**, 19–32.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
 Tabatabaee, M., Anari-Abbasnejad, M., Nozari, N., Sadegheian, S. & Ghasemzadeh, M. (2007). *Acta Cryst.* **E63**, o2099–o2100.
 Wolff, S. K., Grimwood, D. J., McKinnon, J. J., Turner, M. J., Jayatilaka, D. & Spackman, M. A. (2012). *CrystalExplorer3.1*. University of Western Australia.
 Xavier, A. J., Thakur, M. & Marie, J. M. (2012). *J. Chem. Pharm. Res.* **4**, 986–990.

supporting information

Acta Cryst. (2018). E74, 1710-1716 [https://doi.org/10.1107/S2056989018015207]

Crystal structures and the Hirshfeld surface analysis of (*E*)-4-nitro-*N'*-(*o*-chloro, *o*- and *p*-methylbenzylidene)benzenesulfonohydrazides

Akshatha R. Salian, Sabine Foro and B. Thimme Gowda

Computing details

For all structures, data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015).

(*E*)-*N'*-(2-Chlorobenzylidene)-4-nitrobenzenesulfonohydrazide (I)

Crystal data

C₁₃H₁₀ClN₃O₄S
M_r = 339.75
 Monoclinic, *P*2₁
a = 4.9498 (6) Å
b = 22.340 (3) Å
c = 7.0003 (9) Å
 β = 104.40 (1)°
V = 749.76 (17) Å³
Z = 2

F(000) = 348
D_x = 1.505 Mg m⁻³
 Mo *K*α radiation, λ = 0.71073 Å
 Cell parameters from 1264 reflections
 θ = 3.0–27.7°
 μ = 0.42 mm⁻¹
T = 293 K
 Prism, colourless
 0.24 × 0.24 × 0.12 mm

Data collection

Oxford Diffraction Xcalibur single crystal X-ray diffractometer with Sapphire CCD detector
 Radiation source: Enhance (Mo) X-ray Source
 Rotation method data acquisition using ω scans.
 Absorption correction: multi-scan
 (CrysAlis RED; Oxford Diffraction, 2009)
T_{min} = 0.907, *T_{max}* = 0.952
 4582 measured reflections

2696 independent reflections
 2457 reflections with *I* > 2σ(*I*)
R_{int} = 0.015
 θ_{\max} = 25.4°, θ_{\min} = 3.0°
h = -3→5
k = -26→26
l = -8→7

Refinement

Refinement on *F*²
 Least-squares matrix: full
R[*F*² > 2σ(*F*²)] = 0.030
wR(*F*²) = 0.078
S = 1.01
 2696 reflections
 202 parameters
 2 restraints
 Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
 Hydrogen site location: mixed
 H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0489P)^2 + 0.0544P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.14 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.20 \text{ e \AA}^{-3}$

Absolute structure: Flack x determined using
 1102 quotients $[(I^-)-(I^+)]/[(I^-)+(I^+)]$ (Parsons *et al.*, 2013)
 Absolute structure parameter: 0.05 (3)

Special details

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

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.0897 (2)	-0.10843 (5)	0.44188 (16)	0.0737 (3)
S1	0.59163 (16)	0.12968 (4)	0.08796 (12)	0.0429 (2)
O1	0.8691 (5)	0.11233 (13)	0.0900 (4)	0.0603 (7)
O2	0.4169 (5)	0.15582 (12)	-0.0845 (3)	0.0519 (6)
O3	0.8117 (8)	0.27393 (16)	0.9402 (5)	0.0788 (9)
O4	0.4147 (8)	0.3096 (2)	0.7925 (5)	0.1064 (14)
N1	0.4239 (6)	0.07005 (13)	0.1288 (4)	0.0445 (7)
H1N	0.248 (5)	0.0750 (18)	0.109 (5)	0.053*
N2	0.5519 (6)	0.04007 (13)	0.3042 (4)	0.0472 (7)
N3	0.6159 (8)	0.27887 (16)	0.7968 (5)	0.0619 (9)
C1	0.6093 (7)	0.17729 (15)	0.2929 (5)	0.0411 (7)
C2	0.8188 (7)	0.16808 (16)	0.4642 (5)	0.0480 (8)
H2	0.9556	0.1393	0.4672	0.058*
C3	0.8216 (7)	0.20189 (17)	0.6288 (5)	0.0509 (9)
H3	0.9590	0.1963	0.7451	0.061*
C4	0.6175 (8)	0.24394 (16)	0.6174 (5)	0.0467 (8)
C5	0.4109 (8)	0.25443 (17)	0.4481 (6)	0.0517 (9)
H5	0.2762	0.2836	0.4456	0.062*
C6	0.4083 (8)	0.22071 (16)	0.2829 (5)	0.0479 (8)
H6	0.2728	0.2271	0.1662	0.057*
C7	0.3937 (8)	0.00577 (16)	0.3719 (6)	0.0498 (9)
H7	0.2072	0.0013	0.3054	0.060*
C8	0.5085 (8)	-0.02677 (16)	0.5566 (5)	0.0504 (9)
C9	0.3835 (9)	-0.07856 (17)	0.6049 (6)	0.0553 (9)
C10	0.4901 (10)	-0.1087 (2)	0.7781 (6)	0.0719 (11)
H10	0.4014	-0.1428	0.8084	0.086*
C11	0.7282 (13)	-0.0882 (2)	0.9061 (7)	0.0875 (17)
H11	0.8007	-0.1084	1.0237	0.105*
C12	0.8616 (12)	-0.0377 (2)	0.8620 (7)	0.0861 (16)
H12	1.0268	-0.0247	0.9471	0.103*
C13	0.7476 (11)	-0.00665 (19)	0.6908 (6)	0.0715 (13)
H13	0.8324	0.0285	0.6645	0.086*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0766 (7)	0.0678 (6)	0.0675 (6)	-0.0191 (6)	0.0002 (5)	0.0046 (6)
S1	0.0340 (4)	0.0534 (5)	0.0430 (4)	0.0058 (4)	0.0128 (3)	0.0063 (4)
O1	0.0368 (13)	0.080 (2)	0.0683 (16)	0.0089 (12)	0.0213 (12)	-0.0014 (14)
O2	0.0519 (14)	0.0638 (15)	0.0395 (13)	0.0067 (12)	0.0103 (11)	0.0142 (12)
O3	0.084 (2)	0.083 (2)	0.0589 (18)	-0.0005 (18)	-0.0023 (17)	-0.0094 (17)
O4	0.110 (3)	0.128 (3)	0.076 (2)	0.055 (3)	0.011 (2)	-0.027 (2)
N1	0.0362 (15)	0.0500 (16)	0.0443 (16)	0.0064 (13)	0.0041 (13)	0.0079 (12)
N2	0.0466 (17)	0.0437 (16)	0.0472 (17)	0.0088 (14)	0.0039 (14)	0.0061 (13)
N3	0.072 (2)	0.0595 (19)	0.053 (2)	0.0030 (18)	0.0142 (19)	-0.0050 (17)
C1	0.0350 (17)	0.0456 (17)	0.0433 (18)	0.0022 (14)	0.0110 (14)	0.0077 (14)
C2	0.0355 (18)	0.054 (2)	0.052 (2)	0.0097 (15)	0.0059 (16)	0.0040 (16)
C3	0.0401 (19)	0.059 (2)	0.048 (2)	0.0028 (16)	0.0006 (16)	0.0039 (17)
C4	0.048 (2)	0.047 (2)	0.045 (2)	-0.0013 (16)	0.0096 (17)	0.0016 (16)
C5	0.048 (2)	0.050 (2)	0.056 (2)	0.0108 (16)	0.0100 (19)	0.0057 (18)
C6	0.044 (2)	0.052 (2)	0.0443 (19)	0.0118 (16)	0.0048 (16)	0.0082 (16)
C7	0.057 (2)	0.046 (2)	0.043 (2)	0.0017 (16)	0.0059 (17)	-0.0016 (16)
C8	0.065 (2)	0.0430 (19)	0.0394 (18)	0.0024 (18)	0.0053 (17)	-0.0017 (15)
C9	0.070 (3)	0.0474 (19)	0.0442 (19)	-0.0012 (18)	0.0055 (18)	-0.0009 (16)
C10	0.101 (3)	0.058 (2)	0.051 (2)	-0.007 (3)	0.007 (2)	0.007 (2)
C11	0.132 (5)	0.068 (3)	0.046 (2)	-0.005 (3)	-0.011 (3)	0.011 (2)
C12	0.111 (4)	0.074 (3)	0.051 (2)	-0.015 (3)	-0.021 (2)	0.000 (2)
C13	0.095 (4)	0.054 (2)	0.052 (3)	-0.018 (2)	-0.006 (2)	0.003 (2)

Geometric parameters (Å, °)

C11—C9	1.743 (4)	C4—C5	1.379 (5)
S1—O1	1.424 (3)	C5—C6	1.378 (5)
S1—O2	1.424 (2)	C5—H5	0.9300
S1—N1	1.632 (3)	C6—H6	0.9300
S1—C1	1.770 (3)	C7—C8	1.468 (5)
O3—N3	1.214 (4)	C7—H7	0.9300
O4—N3	1.204 (4)	C8—C13	1.390 (6)
N1—N2	1.404 (4)	C8—C9	1.393 (5)
N1—H1N	0.86 (2)	C9—C10	1.373 (6)
N2—C7	1.269 (5)	C10—C11	1.370 (7)
N3—C4	1.481 (5)	C10—H10	0.9300
C1—C6	1.379 (5)	C11—C12	1.380 (7)
C1—C2	1.391 (5)	C11—H11	0.9300
C2—C3	1.375 (5)	C12—C13	1.378 (6)
C2—H2	0.9300	C12—H12	0.9300
C3—C4	1.367 (5)	C13—H13	0.9300
C3—H3	0.9300		
O1—S1—O2	120.09 (15)	C6—C5—H5	120.7
O1—S1—N1	107.89 (16)	C4—C5—H5	120.7

O2—S1—N1	104.77 (15)	C1—C6—C5	119.1 (3)
O1—S1—C1	107.59 (16)	C1—C6—H6	120.5
O2—S1—C1	109.75 (16)	C5—C6—H6	120.5
N1—S1—C1	105.87 (15)	N2—C7—C8	119.2 (3)
N2—N1—S1	113.8 (2)	N2—C7—H7	120.4
N2—N1—H1N	115 (3)	C8—C7—H7	120.4
S1—N1—H1N	114 (3)	C13—C8—C9	117.3 (3)
C7—N2—N1	115.4 (3)	C13—C8—C7	120.9 (4)
O4—N3—O3	123.9 (4)	C9—C8—C7	121.8 (3)
O4—N3—C4	117.3 (4)	C10—C9—C8	121.8 (4)
O3—N3—C4	118.8 (4)	C10—C9—C11	117.7 (3)
C6—C1—C2	121.4 (3)	C8—C9—C11	120.5 (3)
C6—C1—S1	119.4 (3)	C11—C10—C9	119.4 (4)
C2—C1—S1	119.0 (3)	C11—C10—H10	120.3
C3—C2—C1	119.4 (3)	C9—C10—H10	120.3
C3—C2—H2	120.3	C10—C11—C12	120.7 (4)
C1—C2—H2	120.3	C10—C11—H11	119.7
C4—C3—C2	118.3 (3)	C12—C11—H11	119.7
C4—C3—H3	120.8	C11—C12—C13	119.3 (5)
C2—C3—H3	120.8	C11—C12—H12	120.3
C3—C4—C5	123.1 (3)	C13—C12—H12	120.3
C3—C4—N3	118.2 (3)	C12—C13—C8	121.4 (4)
C5—C4—N3	118.7 (3)	C12—C13—H13	119.3
C6—C5—C4	118.6 (3)	C8—C13—H13	119.3
O1—S1—N1—N2	56.0 (3)	C3—C4—C5—C6	0.5 (6)
O2—S1—N1—N2	-175.0 (2)	N3—C4—C5—C6	-178.2 (3)
C1—S1—N1—N2	-59.0 (3)	C2—C1—C6—C5	-1.9 (5)
S1—N1—N2—C7	159.3 (3)	S1—C1—C6—C5	174.9 (3)
O1—S1—C1—C6	151.0 (3)	C4—C5—C6—C1	0.8 (5)
O2—S1—C1—C6	18.7 (3)	N1—N2—C7—C8	-179.1 (3)
N1—S1—C1—C6	-93.9 (3)	N2—C7—C8—C13	22.2 (5)
O1—S1—C1—C2	-32.1 (3)	N2—C7—C8—C9	-158.0 (4)
O2—S1—C1—C2	-164.4 (3)	C13—C8—C9—C10	0.2 (6)
N1—S1—C1—C2	83.0 (3)	C7—C8—C9—C10	-179.6 (4)
C6—C1—C2—C3	1.7 (5)	C13—C8—C9—C11	-177.9 (3)
S1—C1—C2—C3	-175.1 (3)	C7—C8—C9—C11	2.3 (5)
C1—C2—C3—C4	-0.4 (5)	C8—C9—C10—C11	-1.1 (7)
C2—C3—C4—C5	-0.7 (6)	C11—C9—C10—C11	177.1 (4)
C2—C3—C4—N3	178.0 (3)	C9—C10—C11—C12	-0.2 (8)
O4—N3—C4—C3	-170.0 (4)	C10—C11—C12—C13	2.3 (9)
O3—N3—C4—C3	8.2 (5)	C11—C12—C13—C8	-3.2 (9)
O4—N3—C4—C5	8.7 (6)	C9—C8—C13—C12	2.0 (7)
O3—N3—C4—C5	-173.0 (4)	C7—C8—C13—C12	-178.2 (5)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N \cdots O1 ⁱ	0.86 (2)	2.02 (3)	2.853 (4)	163 (4)
C3—H3 \cdots O2 ⁱⁱ	0.93	2.46	3.290 (4)	149

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

(*E*)-*N'*-(2-Methylbenzylidene)-4-nitrobenzenesulfonylhydrazide (II)

Crystal data

$C_{14}H_{13}N_3O_4S$

$M_r = 319.33$

Monoclinic, $P2_1/c$

$a = 7.190$ (1) Å

$b = 15.288$ (2) Å

$c = 13.596$ (2) Å

$\beta = 97.68$ (2)°

$V = 1481.1$ (4) Å³

$Z = 4$

$F(000) = 664$

$D_x = 1.432$ Mg m⁻³

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

Cell parameters from 2833 reflections

$\theta = 2.6$ – 27.8 °

$\mu = 0.24$ mm⁻¹

$T = 293$ K

Rod, yellow

$0.50 \times 0.48 \times 0.24$ mm

Data collection

Oxford Diffraction Xcalibur single crystal X-ray diffractometer with Sapphire CCD detector

Radiation source: Enhance (Mo) X-ray Source

Rotation method data acquisition using ω scans.

Absorption correction: multi-scan

(CrysAlis RED; Oxford Diffraction, 2009)

$T_{\min} = 0.889$, $T_{\max} = 0.945$

9810 measured reflections

2719 independent reflections

2271 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 25.4$ °, $\theta_{\min} = 2.7$ °

$h = -8 \rightarrow 5$

$k = -18 \rightarrow 18$

$l = -11 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.097$

$S = 1.05$

2719 reflections

203 parameters

2 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0372P)^2 + 0.8005P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.28$ e Å⁻³

$\Delta\rho_{\min} = -0.32$ e Å⁻³

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1811 (3)	0.53913 (12)	0.11782 (14)	0.0392 (4)
C2	0.2549 (3)	0.45557 (13)	0.12179 (16)	0.0472 (5)

H2	0.3779	0.4459	0.1104	0.057*
C3	0.1428 (3)	0.38663 (13)	0.14295 (16)	0.0515 (5)
H3	0.1894	0.3298	0.1470	0.062*
C4	-0.0386 (3)	0.40354 (13)	0.15791 (14)	0.0458 (5)
C5	-0.1143 (3)	0.48574 (14)	0.15301 (16)	0.0506 (5)
H5	-0.2384	0.4948	0.1628	0.061*
C6	-0.0019 (3)	0.55474 (13)	0.13323 (16)	0.0475 (5)
H6	-0.0489	0.6115	0.1303	0.057*
C7	0.5612 (3)	0.62282 (12)	0.35371 (15)	0.0414 (4)
H7	0.5251	0.6771	0.3756	0.050*
C8	0.6602 (3)	0.56164 (13)	0.42534 (15)	0.0419 (5)
C9	0.7375 (3)	0.58901 (15)	0.52053 (16)	0.0513 (5)
C10	0.8215 (3)	0.52648 (19)	0.58632 (19)	0.0651 (7)
H10	0.8717	0.5436	0.6500	0.078*
C11	0.8321 (3)	0.4407 (2)	0.5599 (2)	0.0701 (8)
H11	0.8900	0.4004	0.6053	0.084*
C12	0.7574 (3)	0.41387 (16)	0.4665 (2)	0.0651 (7)
H12	0.7657	0.3555	0.4483	0.078*
C13	0.6700 (3)	0.47378 (14)	0.39960 (18)	0.0513 (5)
H13	0.6172	0.4553	0.3369	0.062*
C14	0.7337 (4)	0.68279 (18)	0.5534 (2)	0.0781 (8)
H14A	0.6070	0.7041	0.5427	0.117*
H14B	0.8104	0.7174	0.5157	0.117*
H14C	0.7813	0.6868	0.6226	0.117*
N1	0.4195 (2)	0.66562 (10)	0.20225 (12)	0.0431 (4)
H1N	0.347 (3)	0.6988 (13)	0.2284 (16)	0.052*
N2	0.5234 (2)	0.60339 (10)	0.26201 (12)	0.0413 (4)
N3	-0.1566 (3)	0.32996 (13)	0.18268 (14)	0.0592 (5)
O1	0.2086 (2)	0.69660 (9)	0.05038 (11)	0.0591 (4)
O2	0.4735 (2)	0.59482 (10)	0.04571 (11)	0.0590 (4)
O3	-0.3229 (3)	0.34312 (13)	0.18613 (15)	0.0833 (6)
O4	-0.0808 (3)	0.25947 (12)	0.19986 (16)	0.0867 (6)
S1	0.32586 (7)	0.62811 (3)	0.09402 (4)	0.04312 (16)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0478 (11)	0.0349 (10)	0.0357 (10)	-0.0027 (8)	0.0079 (8)	0.0008 (8)
C2	0.0516 (12)	0.0409 (11)	0.0520 (12)	0.0030 (9)	0.0168 (10)	-0.0018 (9)
C3	0.0726 (14)	0.0318 (10)	0.0524 (13)	-0.0001 (10)	0.0170 (11)	-0.0017 (9)
C4	0.0584 (12)	0.0446 (11)	0.0351 (10)	-0.0133 (9)	0.0086 (9)	-0.0002 (8)
C5	0.0455 (11)	0.0544 (13)	0.0526 (13)	-0.0045 (9)	0.0095 (9)	0.0029 (10)
C6	0.0487 (12)	0.0400 (11)	0.0542 (13)	0.0035 (9)	0.0088 (10)	0.0048 (9)
C7	0.0414 (10)	0.0371 (10)	0.0472 (11)	0.0000 (8)	0.0116 (8)	-0.0037 (9)
C8	0.0340 (10)	0.0463 (11)	0.0473 (12)	0.0009 (8)	0.0121 (8)	0.0023 (9)
C9	0.0409 (11)	0.0646 (14)	0.0494 (13)	-0.0001 (10)	0.0097 (9)	0.0019 (11)
C10	0.0462 (13)	0.096 (2)	0.0527 (14)	0.0022 (13)	0.0074 (10)	0.0156 (14)
C11	0.0456 (13)	0.085 (2)	0.0818 (19)	0.0105 (12)	0.0173 (13)	0.0388 (16)

C12	0.0535 (13)	0.0495 (13)	0.097 (2)	0.0086 (11)	0.0280 (13)	0.0170 (13)
C13	0.0481 (12)	0.0456 (12)	0.0628 (14)	0.0025 (9)	0.0172 (10)	0.0021 (10)
C14	0.0869 (19)	0.0817 (19)	0.0621 (17)	-0.0032 (15)	-0.0039 (14)	-0.0177 (14)
N1	0.0500 (10)	0.0349 (9)	0.0449 (10)	0.0041 (7)	0.0077 (8)	-0.0020 (7)
N2	0.0413 (9)	0.0383 (9)	0.0457 (10)	0.0024 (7)	0.0103 (7)	-0.0003 (7)
N3	0.0799 (11)	0.0527 (12)	0.0460 (10)	-0.0222 (10)	0.0126 (10)	-0.0024 (9)
O1	0.0738 (10)	0.0416 (8)	0.0592 (10)	-0.0047 (7)	-0.0012 (8)	0.0139 (7)
O2	0.0715 (10)	0.0577 (9)	0.0545 (9)	-0.0116 (8)	0.0326 (8)	-0.0072 (7)
O3	0.0790 (10)	0.0840 (13)	0.0928 (14)	-0.0339 (10)	0.0331 (11)	-0.0027 (11)
O4	0.1088 (16)	0.0507 (11)	0.1012 (15)	-0.0198 (10)	0.0163 (12)	0.0168 (10)
S1	0.0545 (3)	0.0361 (3)	0.0402 (3)	-0.0054 (2)	0.0117 (2)	0.0023 (2)

Geometric parameters (Å, °)

C1—C6	1.381 (3)	C9—C14	1.503 (3)
C1—C2	1.381 (3)	C10—C11	1.364 (4)
C1—S1	1.7687 (19)	C10—H10	0.9300
C2—C3	1.380 (3)	C11—C12	1.375 (4)
C2—H2	0.9300	C11—H11	0.9300
C3—C4	1.371 (3)	C12—C13	1.381 (3)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.368 (3)	C13—H13	0.9300
C4—N3	1.475 (3)	C14—H14A	0.9600
C5—C6	1.377 (3)	C14—H14B	0.9600
C5—H5	0.9300	C14—H14C	0.9600
C6—H6	0.9300	N1—N2	1.400 (2)
C7—N2	1.275 (2)	N1—S1	1.6380 (17)
C7—C8	1.465 (3)	N1—H1N	0.839 (15)
C7—H7	0.9300	N3—O4	1.216 (3)
C8—C13	1.392 (3)	N3—O3	1.220 (3)
C8—C9	1.402 (3)	O1—S1	1.4233 (15)
C9—C10	1.391 (3)	O2—S1	1.4157 (15)
C6—C1—C2	121.50 (18)	C9—C10—H10	119.1
C6—C1—S1	119.39 (15)	C10—C11—C12	120.1 (2)
C2—C1—S1	119.10 (15)	C10—C11—H11	120.0
C3—C2—C1	118.86 (19)	C12—C11—H11	120.0
C3—C2—H2	120.6	C11—C12—C13	119.8 (2)
C1—C2—H2	120.6	C11—C12—H12	120.1
C4—C3—C2	118.70 (19)	C13—C12—H12	120.1
C4—C3—H3	120.6	C12—C13—C8	120.5 (2)
C2—C3—H3	120.6	C12—C13—H13	119.7
C5—C4—C3	123.12 (19)	C8—C13—H13	119.7
C5—C4—N3	118.3 (2)	C9—C14—H14A	109.5
C3—C4—N3	118.5 (2)	C9—C14—H14B	109.5
C4—C5—C6	118.25 (19)	H14A—C14—H14B	109.5
C4—C5—H5	120.9	C9—C14—H14C	109.5
C6—C5—H5	120.9	H14A—C14—H14C	109.5

C5—C6—C1	119.56 (19)	H14B—C14—H14C	109.5
C5—C6—H6	120.2	N2—N1—S1	114.00 (12)
C1—C6—H6	120.2	N2—N1—H1N	118.6 (15)
N2—C7—C8	121.51 (18)	S1—N1—H1N	112.8 (15)
N2—C7—H7	119.2	C7—N2—N1	115.88 (16)
C8—C7—H7	119.2	O4—N3—O3	123.7 (2)
C13—C8—C9	119.5 (2)	O4—N3—C4	117.6 (2)
C13—C8—C7	119.03 (19)	O3—N3—C4	118.7 (2)
C9—C8—C7	121.37 (19)	O2—S1—O1	120.69 (10)
C10—C9—C8	118.2 (2)	O2—S1—N1	107.39 (9)
C10—C9—C14	119.2 (2)	O1—S1—N1	105.46 (9)
C8—C9—C14	122.5 (2)	O2—S1—C1	107.72 (9)
C11—C10—C9	121.8 (2)	O1—S1—C1	108.17 (9)
C11—C10—H10	119.1	N1—S1—C1	106.63 (9)
C6—C1—C2—C3	-0.9 (3)	C10—C11—C12—C13	-0.6 (3)
S1—C1—C2—C3	178.33 (16)	C11—C12—C13—C8	1.3 (3)
C1—C2—C3—C4	0.9 (3)	C9—C8—C13—C12	-1.0 (3)
C2—C3—C4—C5	-0.1 (3)	C7—C8—C13—C12	-178.05 (19)
C2—C3—C4—N3	-178.67 (19)	C8—C7—N2—N1	176.70 (15)
C3—C4—C5—C6	-0.8 (3)	S1—N1—N2—C7	-164.16 (14)
N3—C4—C5—C6	177.81 (19)	C5—C4—N3—O4	-170.4 (2)
C4—C5—C6—C1	0.8 (3)	C3—C4—N3—O4	8.3 (3)
C2—C1—C6—C5	0.0 (3)	C5—C4—N3—O3	8.7 (3)
S1—C1—C6—C5	-179.19 (16)	C3—C4—N3—O3	-172.7 (2)
N2—C7—C8—C13	-17.0 (3)	N2—N1—S1—O2	-57.23 (15)
N2—C7—C8—C9	165.96 (18)	N2—N1—S1—O1	172.86 (13)
C13—C8—C9—C10	-0.1 (3)	N2—N1—S1—C1	58.01 (15)
C7—C8—C9—C10	176.89 (18)	C6—C1—S1—O2	-158.52 (16)
C13—C8—C9—C14	179.5 (2)	C2—C1—S1—O2	22.28 (19)
C7—C8—C9—C14	-3.5 (3)	C6—C1—S1—O1	-26.54 (19)
C8—C9—C10—C11	0.9 (3)	C2—C1—S1—O1	154.26 (16)
C14—C9—C10—C11	-178.7 (2)	C6—C1—S1—N1	86.46 (17)
C9—C10—C11—C12	-0.5 (4)	C2—C1—S1—N1	-92.74 (17)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1N...O3 ⁱ	0.84 (2)	2.51 (2)	3.230 (2)	144 (2)
N1—H1N...O4 ⁱ	0.84 (2)	2.44 (2)	3.260 (3)	164 (2)
C2—H2...O2 ⁱⁱ	0.93	2.58	3.284 (2)	133
C12—H12...O1 ⁱⁱⁱ	0.93	2.44	3.341 (3)	164

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

(E)-*N'*-(4-Methylbenzylidene)-4-nitrobenzenesulfonohydrazide monohydrate (III)*Crystal data*C₁₄H₁₃N₃O₄S·H₂O $M_r = 337.35$ Monoclinic, $P2_1/c$ $a = 22.589$ (2) Å $b = 5.4424$ (4) Å $c = 12.7180$ (9) Å $\beta = 92.146$ (6)° $V = 1562.4$ (2) Å³ $Z = 4$ $F(000) = 704$ $D_x = 1.434$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3331 reflections

 $\theta = 3.1$ – 27.8 ° $\mu = 0.24$ mm⁻¹ $T = 293$ K

Prism, yellow

 $0.40 \times 0.36 \times 0.16$ mm*Data collection*

Oxford Diffraction Xcalibur single crystal X-ray diffractometer with Sapphire CCD detector

Radiation source: Enhance (Mo) X-ray Source

Rotation method data acquisition using ω scans.

Absorption correction: multi-scan

(CrysAlis RED; Oxford Diffraction, 2009)

 $T_{\min} = 0.911$, $T_{\max} = 0.963$

9384 measured reflections

2870 independent reflections

2178 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.021$ $\theta_{\max} = 25.4$ °, $\theta_{\min} = 3.2$ ° $h = -19 \rightarrow 27$ $k = -6 \rightarrow 6$ $l = -14 \rightarrow 15$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.034$ $wR(F^2) = 0.092$ $S = 1.02$

2870 reflections

218 parameters

6 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 0.3615P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.19$ e Å⁻³ $\Delta\rho_{\min} = -0.28$ e Å⁻³*Special details*

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.29814 (2)	-0.41337 (9)	0.00460 (3)	0.04151 (15)
O1	0.31514 (6)	-0.4922 (3)	-0.09707 (10)	0.0540 (4)
O2	0.28665 (6)	-0.5896 (2)	0.08395 (10)	0.0533 (4)
O3	0.50400 (7)	0.4229 (3)	0.11440 (12)	0.0697 (5)
O4	0.45497 (7)	0.4367 (3)	0.25661 (11)	0.0619 (4)
N1	0.23739 (7)	-0.2563 (3)	-0.01813 (12)	0.0465 (4)
H1N	0.2396 (9)	-0.153 (3)	-0.0681 (14)	0.056*
N2	0.21027 (7)	-0.1771 (3)	0.07293 (12)	0.0470 (4)
N3	0.46427 (7)	0.3533 (3)	0.16974 (13)	0.0463 (4)

C1	0.35122 (7)	-0.2010 (3)	0.05425 (13)	0.0367 (4)
C2	0.38690 (8)	-0.0791 (4)	-0.01472 (14)	0.0439 (4)
H2	0.3852	-0.1181	-0.0860	0.053*
C3	0.42513 (8)	0.1012 (4)	0.02325 (14)	0.0440 (4)
H3	0.4500	0.1839	-0.0215	0.053*
C4	0.42543 (7)	0.1550 (3)	0.12892 (14)	0.0383 (4)
C5	0.39051 (8)	0.0344 (4)	0.19866 (14)	0.0431 (4)
H5	0.3923	0.0746	0.2698	0.052*
C6	0.35286 (8)	-0.1470 (3)	0.16104 (13)	0.0424 (4)
H6	0.3289	-0.2321	0.2065	0.051*
C7	0.17972 (8)	0.0167 (4)	0.06417 (16)	0.0505 (5)
H7	0.1791	0.1035	0.0012	0.061*
C8	0.14529 (8)	0.1070 (4)	0.15155 (16)	0.0501 (5)
C9	0.11387 (10)	0.3238 (4)	0.14097 (19)	0.0669 (6)
H9	0.1164	0.4165	0.0799	0.080*
C10	0.07863 (11)	0.4042 (5)	0.2206 (2)	0.0754 (7)
H10	0.0578	0.5505	0.2118	0.090*
C11	0.07354 (10)	0.2750 (5)	0.31180 (19)	0.0670 (6)
C12	0.10618 (11)	0.0627 (5)	0.32272 (19)	0.0725 (7)
H12	0.1044	-0.0267	0.3848	0.087*
C13	0.14130 (10)	-0.0210 (4)	0.24460 (17)	0.0626 (6)
H13	0.1627	-0.1658	0.2545	0.075*
C14	0.03449 (12)	0.3685 (6)	0.3974 (2)	0.0970 (10)
H14A	0.0205	0.2322	0.4374	0.146*
H14B	0.0013	0.4553	0.3660	0.146*
H14C	0.0569	0.4774	0.4430	0.146*
O5	0.25535 (9)	0.4283 (3)	0.31149 (13)	0.0753 (5)
H51	0.2502 (12)	0.445 (5)	0.2487 (14)	0.090*
H52	0.2685 (12)	0.296 (4)	0.328 (2)	0.090*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0456 (3)	0.0425 (3)	0.0370 (3)	-0.0023 (2)	0.00972 (18)	-0.0038 (2)
O1	0.0622 (9)	0.0571 (8)	0.0437 (7)	-0.0028 (7)	0.0164 (6)	-0.0144 (7)
O2	0.0653 (9)	0.0448 (7)	0.0506 (8)	-0.0067 (7)	0.0131 (7)	0.0041 (6)
O3	0.0700 (10)	0.0800 (11)	0.0592 (9)	-0.0336 (9)	0.0065 (8)	0.0081 (8)
O4	0.0664 (9)	0.0616 (9)	0.0579 (8)	-0.0062 (8)	0.0040 (7)	-0.0154 (7)
N1	0.0452 (9)	0.0583 (10)	0.0363 (9)	-0.0017 (8)	0.0052 (7)	-0.0027 (7)
N2	0.0416 (9)	0.0569 (10)	0.0430 (9)	-0.0024 (8)	0.0087 (7)	-0.0077 (8)
N3	0.0474 (9)	0.0467 (9)	0.0444 (8)	-0.0028 (7)	-0.0023 (7)	0.0059 (7)
C1	0.0349 (9)	0.0406 (9)	0.0350 (9)	0.0037 (8)	0.0060 (7)	0.0004 (8)
C2	0.0456 (10)	0.0545 (11)	0.0323 (9)	-0.0030 (9)	0.0102 (8)	-0.0018 (9)
C3	0.0415 (10)	0.0532 (11)	0.0380 (10)	-0.0026 (9)	0.0117 (8)	0.0063 (9)
C4	0.0356 (9)	0.0402 (9)	0.0393 (9)	0.0028 (7)	0.0023 (7)	0.0036 (7)
C5	0.0458 (10)	0.0536 (12)	0.0303 (9)	-0.0018 (9)	0.0045 (8)	0.0007 (8)
C6	0.0417 (10)	0.0518 (11)	0.0341 (9)	-0.0030 (9)	0.0084 (8)	0.0057 (8)
C7	0.0420 (11)	0.0591 (12)	0.0505 (12)	-0.0031 (10)	0.0043 (9)	-0.0030 (10)

C8	0.0425 (11)	0.0548 (12)	0.0533 (12)	-0.0013 (9)	0.0039 (9)	-0.0123 (10)
C9	0.0678 (15)	0.0632 (14)	0.0700 (15)	0.0114 (12)	0.0066 (12)	-0.0006 (12)
C10	0.0706 (16)	0.0697 (15)	0.0857 (19)	0.0242 (13)	0.0016 (14)	-0.0173 (14)
C11	0.0539 (13)	0.0848 (17)	0.0624 (15)	0.0074 (13)	0.0029 (11)	-0.0267 (13)
C12	0.0834 (17)	0.0808 (17)	0.0542 (14)	0.0141 (15)	0.0159 (12)	-0.0067 (13)
C13	0.0661 (14)	0.0672 (14)	0.0548 (13)	0.0171 (12)	0.0086 (11)	-0.0087 (11)
C14	0.0808 (18)	0.127 (3)	0.0847 (19)	0.0258 (18)	0.0152 (15)	-0.0400 (18)
O5	0.1131 (15)	0.0646 (10)	0.0485 (9)	0.0000 (10)	0.0078 (10)	-0.0005 (9)

Geometric parameters (Å, °)

S1—O2	1.4230 (13)	C6—H6	0.9300
S1—O1	1.4284 (13)	C7—C8	1.465 (3)
S1—N1	1.6335 (17)	C7—H7	0.9300
S1—C1	1.7652 (18)	C8—C13	1.379 (3)
O3—N3	1.221 (2)	C8—C9	1.381 (3)
O4—N3	1.220 (2)	C9—C10	1.383 (3)
N1—N2	1.398 (2)	C9—H9	0.9300
N1—H1N	0.850 (15)	C10—C11	1.365 (3)
N2—C7	1.263 (3)	C10—H10	0.9300
N3—C4	1.473 (2)	C11—C12	1.375 (3)
C1—C2	1.383 (2)	C11—C14	1.514 (3)
C1—C6	1.389 (2)	C12—C13	1.372 (3)
C2—C3	1.382 (3)	C12—H12	0.9300
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.375 (2)	C14—H14A	0.9600
C3—H3	0.9300	C14—H14B	0.9600
C4—C5	1.375 (2)	C14—H14C	0.9600
C5—C6	1.377 (3)	O5—H51	0.808 (16)
C5—H5	0.9300	O5—H52	0.802 (17)
O2—S1—O1	120.13 (8)	C1—C6—H6	120.5
O2—S1—N1	107.70 (8)	N2—C7—C8	121.14 (19)
O1—S1—N1	104.45 (8)	N2—C7—H7	119.4
O2—S1—C1	109.10 (8)	C8—C7—H7	119.4
O1—S1—C1	108.56 (8)	C13—C8—C9	117.7 (2)
N1—S1—C1	105.98 (8)	C13—C8—C7	122.4 (2)
N2—N1—S1	113.94 (12)	C9—C8—C7	119.9 (2)
N2—N1—H1N	117.2 (14)	C8—C9—C10	120.4 (2)
S1—N1—H1N	113.8 (15)	C8—C9—H9	119.8
C7—N2—N1	116.04 (17)	C10—C9—H9	119.8
O4—N3—O3	124.16 (17)	C11—C10—C9	121.9 (2)
O4—N3—C4	118.13 (15)	C11—C10—H10	119.0
O3—N3—C4	117.71 (16)	C9—C10—H10	119.0
C2—C1—C6	121.61 (17)	C10—C11—C12	117.2 (2)
C2—C1—S1	119.44 (13)	C10—C11—C14	120.5 (2)
C6—C1—S1	118.77 (13)	C12—C11—C14	122.3 (3)
C3—C2—C1	119.35 (16)	C13—C12—C11	121.9 (2)

C3—C2—H2	120.3	C13—C12—H12	119.1
C1—C2—H2	120.3	C11—C12—H12	119.1
C4—C3—C2	118.22 (16)	C12—C13—C8	120.8 (2)
C4—C3—H3	120.9	C12—C13—H13	119.6
C2—C3—H3	120.9	C8—C13—H13	119.6
C3—C4—C5	123.13 (17)	C11—C14—H14A	109.5
C3—C4—N3	118.79 (15)	C11—C14—H14B	109.5
C5—C4—N3	118.07 (16)	H14A—C14—H14B	109.5
C4—C5—C6	118.66 (16)	C11—C14—H14C	109.5
C4—C5—H5	120.7	H14A—C14—H14C	109.5
C6—C5—H5	120.7	H14B—C14—H14C	109.5
C5—C6—C1	119.01 (16)	H51—O5—H52	113 (3)
C5—C6—H6	120.5		
O2—S1—N1—N2	46.42 (15)	C3—C4—C5—C6	-0.8 (3)
O1—S1—N1—N2	175.19 (13)	N3—C4—C5—C6	178.33 (16)
C1—S1—N1—N2	-70.24 (14)	C4—C5—C6—C1	-0.3 (3)
S1—N1—N2—C7	152.32 (14)	C2—C1—C6—C5	0.8 (3)
O2—S1—C1—C2	153.79 (14)	S1—C1—C6—C5	-174.26 (14)
O1—S1—C1—C2	21.22 (17)	N1—N2—C7—C8	175.00 (16)
N1—S1—C1—C2	-90.50 (15)	N2—C7—C8—C13	-4.2 (3)
O2—S1—C1—C6	-31.06 (16)	N2—C7—C8—C9	177.82 (19)
O1—S1—C1—C6	-163.64 (14)	C13—C8—C9—C10	-1.6 (3)
N1—S1—C1—C6	84.65 (15)	C7—C8—C9—C10	176.5 (2)
C6—C1—C2—C3	-0.1 (3)	C8—C9—C10—C11	0.2 (4)
S1—C1—C2—C3	174.89 (14)	C9—C10—C11—C12	1.5 (4)
C1—C2—C3—C4	-1.0 (3)	C9—C10—C11—C14	-180.0 (2)
C2—C3—C4—C5	1.4 (3)	C10—C11—C12—C13	-1.7 (4)
C2—C3—C4—N3	-177.66 (16)	C14—C11—C12—C13	179.8 (2)
O4—N3—C4—C3	163.87 (17)	C11—C12—C13—C8	0.3 (4)
O3—N3—C4—C3	-16.5 (2)	C9—C8—C13—C12	1.4 (3)
O4—N3—C4—C5	-15.3 (2)	C7—C8—C13—C12	-176.6 (2)
O3—N3—C4—C5	164.39 (17)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots O5 ⁱ	0.85 (2)	2.00 (2)	2.848 (2)	173 (2)
O5—H51 \cdots O2 ⁱⁱ	0.81 (2)	2.29 (2)	3.006 (2)	148 (3)
O5—H52 \cdots O1 ⁱⁱⁱ	0.80 (2)	2.17 (2)	2.949 (2)	166 (3)
C5—H5 \cdots O1 ⁱⁱⁱ	0.93	2.52	3.167 (2)	127

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