

Crystal structures of three *N*-acylhydrazone isomersH. Purandara,^{a,b} Sabine Foro^c and B. Thimme Gowda^{a,d,*}

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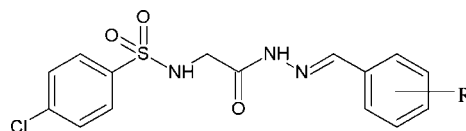
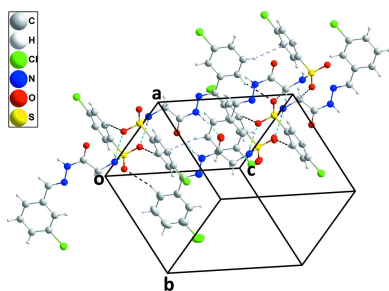
Keywords: crystal structure; *N*-acylhydrazone; dihedral angle; inversion dimers; C—H···O interaction.

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The crystal structures of three isomers of (*E*)-4-chloro-*N*-{2-[2-(chlorobenzylidene)hydrazinyl]-2-oxoethyl}benzenesulfonamide, namely, (*E*)-4-chloro-*N*-{2-[2-(2-chlorobenzylidene)hydrazinyl]-2-oxoethyl}benzenesulfonamide (I), (*E*)-4-chloro-*N*-{2-[2-(3-chlorobenzylidene)hydrazinyl]-2-oxoethyl}benzenesulfonamide (II) and (*E*)-4-chloro-*N*-{2-[2-(4-chlorobenzylidene)hydrazinyl]-2-oxoethyl}benzenesulfonamide (III), with the general formula C₁₅H₁₃Cl₂N₃O₃S are described, with the chloro group in *ortho*, *meta* and *para* positions in the benzylidene benzene ring. All the three isomeric compounds crystallize in the centrosymmetric triclinic $P\bar{1}$ space group with one molecule each in the asymmetric unit and two molecules in the unit cell. The dihedral angles between the two phenyl rings are 11.09 (14), 53.79 (18) and 72.37 (11)° in (I), (II) and (III), respectively. The central part of the molecule (—C—N—N=C—) is almost linear with C—N—N—C torsion angles of 179.1 (2), −169.5 (3) and 178.5 (2)° for (I), (II) and (III), respectively. In all the three crystals, the molecules form inversion dimers with $R_2^2(8)$ ring motifs, which are further augmented by C—H···O interactions.

1. Chemical context

The properties of molecules in solution and the solid state are strongly influenced by weak non-covalent interactions. Weak molecular interactions are investigated routinely in the areas of molecular recognition (Brouwer *et al.*, 1999), self-assembly (Seth *et al.*, 2011), supramolecular chemistry and general host-guest interactions (Kim *et al.*, 2000; Sharma *et al.*, 2009). Analysis of intermolecular interactions and estimation of their energies provide greater insights into molecular conformations (Cao *et al.*, 2020; Jablonski, 2020). The nature and site of substituents influence the extent of polarization of electron distribution in covalent compounds. In our previous work (Purandara *et al.*, 2017*a,b*), the presence of the electron-withdrawing nitro group on the benzene ring was found to decrease the electronic density, rendering aromatic C—H protons acidic, whereas a methyl substituent did not activate aromatic protons for participation in intermolecular C—H···O interactions. In a continuation of these efforts to study the effect of substituents on weak molecular interactions, we report herein the synthesis, characterization and crystal structures of three isomeric molecules.



(I) R = 2-Cl, (II) R = 3-Cl, (III) R = 4-Cl



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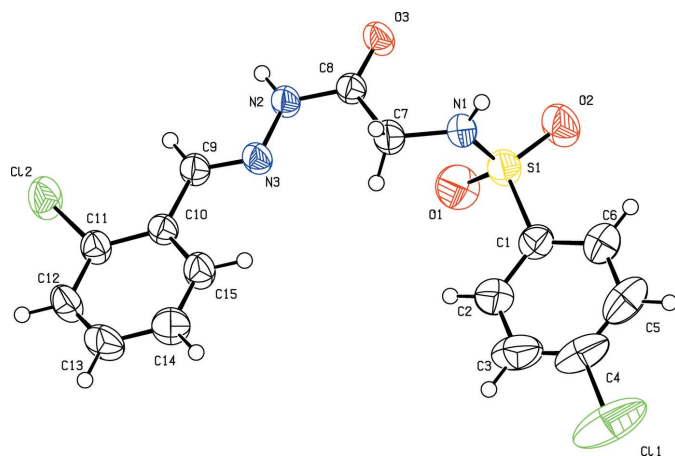


Figure 1
Molecular structure of (I), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level.

2. Structural commentary

All three isomers (I)–(III) (Figs. 1–3) crystallize in the centrosymmetric triclinic system with space group $P\bar{1}$ and with one molecule in the asymmetric unit and two molecules in the unit cell. The conformation of both the sulfonamide and hydrazine N–H bonds are *syn* with respect to the C=O bonds in all the three compounds. Similarly, the imine C–H bond in the amide part is also *syn* with respect to the amide N–H bond. All four such bonds in the central part are *syn* to each other. The C8–O3 and C9–N3 bond lengths of 1.224 (3)–1.236 (3) Å and 1.271 (3)–1.275 (4) Å, respectively, are in the ranges of normal C=O and C=N bond lengths, indicating double-bond character and thus confirming the keto tautomeric form and are comparable with those in related *N*-acylhydrazone structures (Purandara *et al.*, 2017). The delocalization of π -electron density over the C9/N3/N2/C8/O3 fragment is indicated by the shortening of the C8–N2 [1.337 (3)–1.342 (4) Å] distances compared to the normal C–N single bond length of 1.40 Å (Allen *et al.*, 1987). The sulfonamide bonds are *synclinal*, *anticlinal* and *antiperiplanar* with the S1–N1–C7–C8 torsion angle being 83.6 (3),

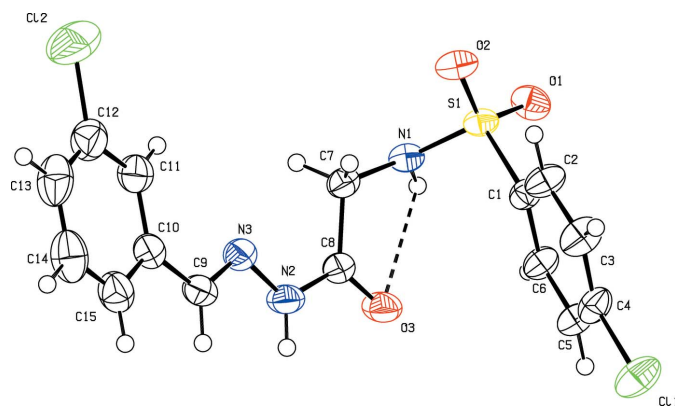


Figure 2
The molecular structure of (II), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level. The intramolecular hydrogen bond is depicted by a dashed line.

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 O3 ⁱ	0.84 (2)	2.01 (2)	2.823 (3)	162 (3)
N2–H2N \cdots O3 ⁱⁱ	0.85 (2)	2.06 (2)	2.897 (2)	167 (2)
C12–H12 \cdots O2 ⁱⁱⁱ	0.93	2.53	3.439 (3)	166

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

–107.2 (3) and 171.09 (18)°, in compounds (I), (II) and (III), respectively. The major twist in the molecule occurs about the S1–N1 bond [C1–S1–N1–C7 = 97.6 (2) (I), 65.6 (2) (II) and –80.1 (2)° (III)], giving the molecule an approximate overall L-shape. All the molecules adopt an *E* configuration around the C9=N3 bond as indicated by the N2–N3–C9–C10 torsion angles of 179.6 (2), 179.1 (3) and 180.0 (2)° for (I), (II) and (III), respectively. The central fragment of the molecule, (C9/N3/N2/C8/O3) is nearly coplanar with the phenyl ring (C10–C15), as indicated by the dihedral angles between their best planes of 4.2 (2) in (I), 11.9 (3) in (II) and 7.0 (3)° in (III). The dihedral angles between the two phenyl rings, C1–C6 and C10–C15 are 11.1 (1), 53.8 (1) and 72.4 (1)° for (I), (II) and (III), respectively, indicating non-planarity of the three molecules. An intramolecular hydrogen bond, N1–H1N \cdots O3, is observed in (II) and (III), generating an *S*(5) ring motif.

3. Supramolecular features

In the crystal of (I), the carbonyl oxygen (O3) shows bifurcated hydrogen bonding. In one part, the molecules are linked by a pair of N2–H2N \cdots O3 hydrogen bonds involving the amide NH atom, forming inversion dimers with an $R_2^2(8)$ ring motif. In the other part, the molecules are linked by a pair of N1–H1N \cdots O3 hydrogen bonds with the sulfonamide NH atom of another molecule, forming rings with an $R_2^2(10)$ graph-set motif, leading to a layered structure with the mean planes of the layers inclined to the *ab* plane by 16.1 (5)° (Table 1, Fig. 4). In the crystal of (II), the molecules are linked by two pairs of N–H \cdots O hydrogen bonds (N1–H1N \cdots O2 and

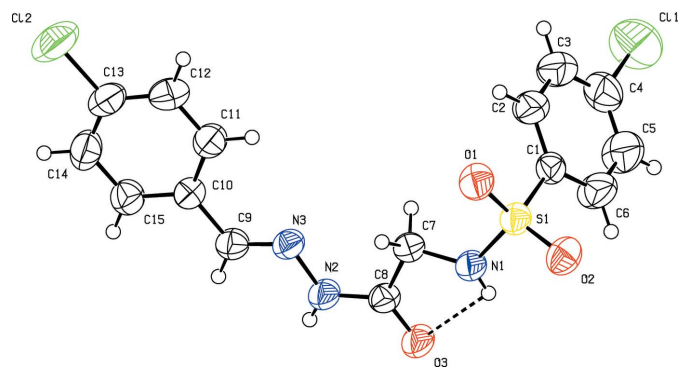


Figure 3
The molecular structure of (III), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level. The intramolecular hydrogen bond is depicted by a dashed line.

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

C_g is the centroid of the C1–C6 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O1^i$	0.84 (2)	2.23 (2)	3.041 (3)	161 (3)
$N2-H2N\cdots O3^{ii}$	0.86 (2)	1.97 (2)	2.828 (3)	171 (3)
$C6-H6\cdots O1^i$	0.93	2.53	3.422 (4)	161
$C7-H7B\cdots O2^{iii}$	0.97	2.47	3.434 (4)	172
$C15-H15\cdots O2^{iv}$	0.93	2.58	3.474 (4)	162
$C14-H14\cdots C_g^v$	0.93	2.84	3.675 (5)	150

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

$N2-H2N\cdots O3$, involving both the sulfonyl and carbonyl O atoms with both sulfonamide and amide N–H bonds ($N1-H1N$ and $N2-H2N$), forming inversion dimers with $R_2^2(8)$ ring motifs. These interactions are further strengthened by C–H \cdots O hydrogen bonds. Thus, three-center $N1-H1N/C6-$

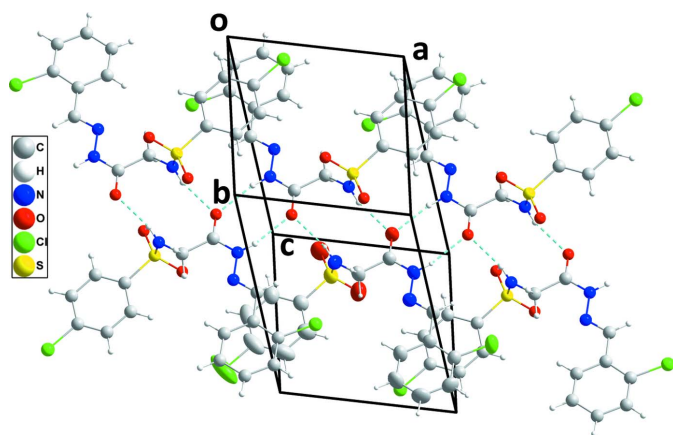


Figure 4
A view of a portion of one chain of inversion dimers in (I) connected by N–H \cdots O hydrogen bonds (dashed lines) and extending along the a -axis direction.

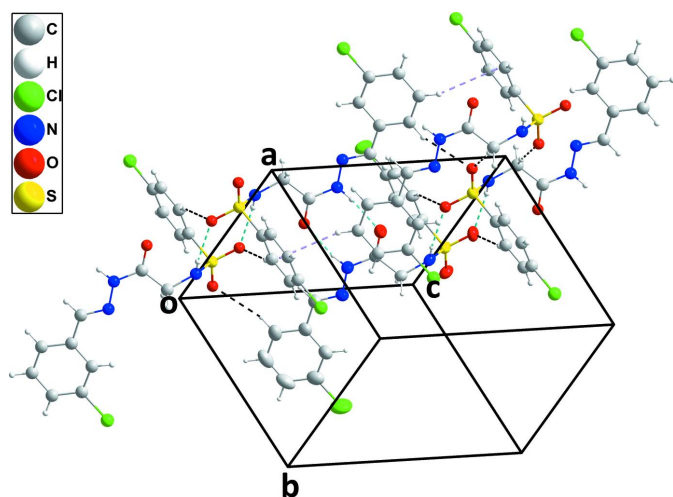


Figure 5
A partial packing diagram for (II) with N–H \cdots O and C–H \cdots O hydrogen bonds depicted, respectively, by light-blue and black dashed lines. The C–H \cdots π (ring) interactions are depicted by violet dashed lines.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O2^i$	0.82 (2)	2.25 (2)	3.032 (3)	162 (3)
$N1-H1N\cdots O3$	0.82 (2)	2.23 (3)	2.609 (3)	109 (2)
$N2-H2N\cdots O3^{ii}$	0.86 (2)	1.98 (2)	2.829 (3)	170 (3)
$C15-H15\cdots O1^{iii}$	0.93	2.45	3.330 (3)	157

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

$H6\cdots O1$ hydrogen bonds result in molecular chains containing the $R_2^1(7)$ ring motif (Fig. 5). These rings are extended along the principal diagonal of the ac plane via $C7-H7\cdots O2$ hydrogen bonds, forming $R_2^2(10)$ ring motifs, and by $C15-H15\cdots O2$ interactions. In addition, the crystal structure is reinforced by C–H \cdots π (ring) interactions (Fig. 5), details of which are summarized in Table 2. In the crystal of (III), the molecules are also linked by two pairs of N–H \cdots O hydrogen bonds ($N1-H1N\cdots O2$ and $N2-H2N\cdots O3$), forming inversion dimers with $R_2^2(8)$ ring motifs. These dimers are connected by intermolecular $C15-H15\cdots O1$ interactions, forming ribbons two molecules wide and extending along the principal diagonal of the ab plane (Table 3, Fig. 6). The presence of the chlorine atom on the phenyl ring (C10–C15) of (I)–(III) makes the aromatic protons acidic, resulting in the formation of C–H \cdots O hydrogen bonds with the sulfonyl O atom.

4. Database survey

Comparison of structures (I)–(III) with those of related *N*-acylhydrazone derivatives (Purandara *et al.*, 2017, 2018) shows that the site of substitution of an electron-withdrawing group on the aromatic ring plays a major role in stabilizing the crystal packing by linking the molecules through various weak interactions.

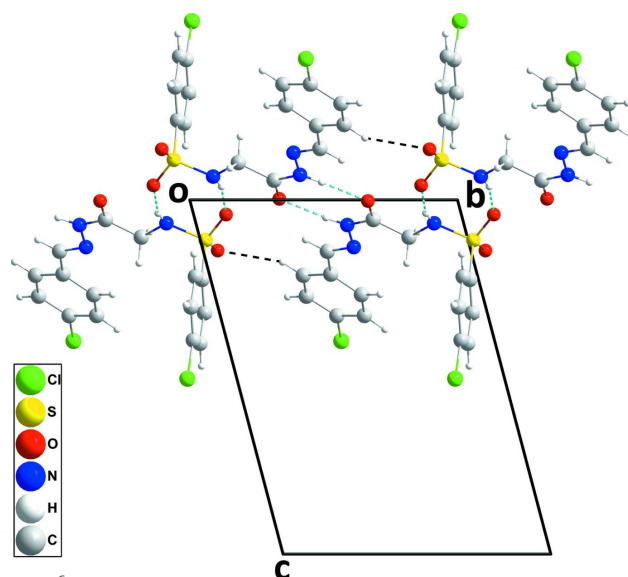


Figure 6
A portion of one chain in (III) viewed along the a -axis direction with hydrogen bonds depicted as in Fig. 5.

Table 4
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	C ₁₅ H ₁₃ Cl ₂ N ₃ O ₃ S	C ₁₅ H ₁₃ Cl ₂ N ₃ O ₃ S	C ₁₅ H ₁₃ Cl ₂ N ₃ O ₃ S
<i>M_r</i>	386.24	386.24	386.24
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$	Triclinic, <i>P</i> $\bar{1}$	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	293	293	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	7.7426 (7), 10.429 (1), 10.934 (1)	9.491 (1), 9.976 (1), 10.446 (1)	6.7234 (9), 10.281 (1), 13.611 (2)
α , β , γ (°)	85.51 (1), 76.92 (1), 81.04 (1)	67.22 (1), 66.80 (1), 86.32 (1)	74.98 (1), 87.11 (1), 75.34 (1)
<i>V</i> (Å ³)	848.64 (14)	833.59 (17)	879.0 (2)
<i>Z</i>	2	2	2
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.52	0.53	0.51
Crystal size (mm)	0.48 × 0.36 × 0.10	0.36 × 0.14 × 0.08	0.46 × 0.42 × 0.20
Data collection			
Diffractometer	Oxford Diffraction Xcalibur with Sapphire CCD	Oxford Diffraction Xcalibur with Sapphire CCD	Oxford Diffraction Xcalibur with Sapphire CCD
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_{min}</i> – <i>T_{max}</i>	0.787, 0.949	0.831, 0.959	0.801, 0.906
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	5699, 3417, 2359	5759, 3354, 2688	5737, 3598, 2600
<i>R_{int}</i>	0.018	0.018	0.019
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.625	0.625	0.625
Refinement			
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> [<i>F</i> ²], <i>S</i>	0.046, 0.102, 1.04	0.054, 0.108, 1.22	0.048, 0.120, 1.04
No. of reflections	3417	3354	3598
No. of parameters	223	223	223
No. of restraints	2	2	2
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
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.24, -0.29	0.27, -0.39	0.33, -0.30

Computer programs: *CrysAlis CCD* and *CrysAlis RED* (Oxford Diffraction, 2009), *SHELXS2013/1* (Sheldrick, 2008), *SHELXL2014/6* (Sheldrick, 2015) and *PLATON* (Spek, 2020).

5. Synthesis and crystallization

General procedure for the synthesis of *N*-(4-chlorobenzene-sulfonyl) glycine hydrazone derivatives (I)–(III)

A mixture of *N*-(4-chlorobenzene-sulfonyl) glycyl hydrazide (0.01 mol) and the appropriate chlorobenzaldehyde (0.01 mol) in anhydrous methanol (30 mL) and two drops of glacial acetic acid was refluxed for 8 h. After cooling, the precipitate was collected by vacuum filtration, washed with cold methanol and dried. It was recrystallized to constant melting point from methanol. The purity of the compound was checked by TLC and characterized by its IR and NMR spectra. Single crystals suitable for the X-ray diffraction study were grown from DMF solution by slow evaporation of the solvent.

Compound (I): Prism-like yellow single crystals; m.p. 506–507 K; IR (KBr, γ , cm⁻¹): 3190.3 (N–H), 1672.3 (C=O), 1608.6 (C=N), 1334.7 (S=O, asym) and 1159.2 cm⁻¹ (S=O, sym); ¹H NMR (400 MHz, DMSO-*d*₆, δ ppm): 3.64, 4.14 (*d*, 2H), 7.36–7.45 (*m*, 2H, Ar-H), 7.47–7.50 (*m*, 1H, Ar-H), 7.61–7.67 (*m*, 2H, Ar-H), 7.83–7.95 (*m*, 3H, Ar-H), 8.12 (*s*, 1H), 8.13 (*s*, 1H), 11.64 (*s*, 1H). ¹³C NMR (400 MHz, DMSO-*d*₆, δ ppm): 43.27, 44.49, 126.82, 127.53, 128.54, 129.11, 129.82, 131.25, 133.02, 137.24, 139.19, 139.81, 143.18, 164.23, 169.08.

Compound (II): Prism-like colourless single crystals; m.p. 469–470 K; IR (KBr, γ , cm⁻¹): 3265.5 (N–H), 1687.7

(C=O), 1589.3 (C=N), 1340.5 (S=O, asym) and 1168.9 cm⁻¹ (S=O, sym); ¹H NMR (400 MHz, DMSO-*d*₆, δ ppm): 3.64, 4.11 (*2d*, 2H), 7.39–7.44 (*m*, 2H, Ar-H), 7.54–7.65 (*m*, 4H, Ar-H), 7.84–7.87 (*m*, 2H, Ar-H), 7.91, 8.15 (*2s*, 1H), 8.01, 8.21 (*2t*, 1H), 11.51, 11.54 (*2s*, 1H). ¹³C NMR (400 MHz, DMSO-*d*₆, δ ppm): 43.24, 44.42, 125.52, 126.14, 128.44, 128.91, 129.47, 130.31, 133.70, 136.05, 137.34, 139.36, 142.18, 145.60, 164.17, 168.96.

Compound (III): Rod-like colourless single crystals; m.p. 473–475 K; IR (KBr, γ , cm⁻¹): 3246.2 (N–H), 1685.8 (C=O), 1591.3 (C=N), 1344.4 (S=O, asym) and 1168.9 cm⁻¹ (S=O, sym); ¹H NMR (400 MHz, DMSO-*d*₆, δ ppm): 3.62, 4.11 (*2d*, 2H), 7.48–7.51 (*m*, 2H, Ar-H), 7.63–7.71 (*m*, 4H, Ar-H), 7.81–7.85 (*m*, 2H, Ar-H), 7.92, 8.14 (*2s*, 1H), 8.01 (*t*, 1H), 11.49, 11.53 (*2s*, 1H). ¹³C NMR (400 MHz, DMSO-*d*₆, δ ppm): 43.21, 128.50, 128.70, 128.86, 129.14, 132.87, 134.34, 137.20, 139.50, 142.53, 145.80, 164.07, 168.96.

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 4. H atoms bonded to C were positioned with idealized geometry using a riding model with C–H = 0.93 Å (aromatic) and 0.97 Å (methylene). The amino H atoms were refined with the N–H distances restrained to

0.86 (2) Å. All H atoms were assigned isotropic displacement parameters $1.2 \times U_{eq}$ of the parent atom. In compound (III), the $\bar{1}13$ reflection had a poor agreement with its calculated value and was omitted from the final refinement.

Acknowledgements

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supporting information

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Crystal structures of three *N*-acylhydrazone isomers

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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: *SHELXS2013/1* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014/6* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2020); software used to prepare material for publication: *SHELXL2014/6* (Sheldrick, 2015).

(*E*)-4-Chloro-*N*-{2-[2-(2-chlorobenzylidene)hydrazinyl]-2-oxoethyl}benzenesulfonamide (I)

Crystal data

$C_{15}H_{13}Cl_2N_3O_3S$

$M_r = 386.24$

Triclinic, $P\bar{1}$

$a = 7.7426$ (7) Å

$b = 10.429$ (1) Å

$c = 10.934$ (1) Å

$\alpha = 85.51$ (1)°

$\beta = 76.92$ (1)°

$\gamma = 81.04$ (1)°

$V = 848.64$ (14) Å³

$Z = 2$

$F(000) = 396$

$D_x = 1.512$ Mg m⁻³

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

Cell parameters from 2012 reflections

$\theta = 2.8$ – 27.8 °

$\mu = 0.52$ mm⁻¹

$T = 293$ K

Prism, yellow

$0.48 \times 0.36 \times 0.10$ mm

Data collection

Oxford Diffraction Xcalibur with Sapphire

CCD

diffractometer

Radiation source: Enhance (Mo) X-ray Source

Rotation method data acquisition using ω scans.

Absorption correction: multi-scan

(*CrysAlis RED*; Oxford Diffraction, 2009)

$T_{\min} = 0.787$, $T_{\max} = 0.949$

5699 measured reflections

3417 independent reflections

2359 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\max} = 26.4$ °, $\theta_{\min} = 2.8$ °

$h = -9 \rightarrow 9$

$k = -10 \rightarrow 13$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.102$

$S = 1.04$

3417 reflections

223 parameters

2 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0321P)^2 + 0.3938P]$

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

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

$\Delta\rho_{\max} = 0.24$ e Å⁻³

$\Delta\rho_{\min} = -0.29$ e Å⁻³

Special details

Experimental. CrysAlis RED (Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1746 (3)	0.6076 (2)	0.8440 (2)	0.0487 (6)
C2	0.1897 (4)	0.7192 (3)	0.8985 (3)	0.0687 (8)
H2	0.3023	0.7399	0.8985	0.082*
C3	0.0378 (5)	0.7997 (3)	0.9529 (3)	0.0852 (11)
H3	0.0473	0.8756	0.9888	0.102*
C4	-0.1270 (5)	0.7675 (4)	0.9540 (3)	0.0801 (11)
C5	-0.1448 (4)	0.6551 (4)	0.9042 (3)	0.0763 (10)
H5	-0.2579	0.6330	0.9083	0.092*
C6	0.0079 (4)	0.5742 (3)	0.8473 (3)	0.0595 (7)
H6	-0.0022	0.4982	0.8118	0.071*
C7	0.5597 (3)	0.6727 (2)	0.6046 (3)	0.0521 (7)
H7A	0.5504	0.7198	0.6794	0.063*
H7B	0.5376	0.7357	0.5378	0.063*
C8	0.7483 (3)	0.6002 (2)	0.5670 (2)	0.0386 (5)
C9	0.9738 (3)	0.8484 (2)	0.6184 (2)	0.0405 (6)
H9	1.0910	0.8074	0.5943	0.049*
C10	0.9368 (3)	0.9816 (2)	0.6622 (2)	0.0381 (5)
C11	1.0705 (3)	1.0515 (2)	0.6753 (2)	0.0430 (6)
C12	1.0322 (4)	1.1777 (2)	0.7149 (3)	0.0545 (7)
H12	1.1239	1.2217	0.7240	0.065*
C13	0.8572 (4)	1.2374 (3)	0.7408 (3)	0.0616 (8)
H13	0.8306	1.3224	0.7669	0.074*
C14	0.7213 (4)	1.1717 (3)	0.7281 (3)	0.0614 (8)
H14	0.6032	1.2124	0.7452	0.074*
C15	0.7610 (3)	1.0457 (2)	0.6902 (3)	0.0516 (7)
H15	0.6681	1.0019	0.6831	0.062*
N1	0.4241 (3)	0.5872 (2)	0.6292 (2)	0.0526 (6)
H1N	0.383 (3)	0.569 (3)	0.569 (2)	0.063*
N2	0.8820 (3)	0.66560 (18)	0.5716 (2)	0.0420 (5)
H2N	0.990 (2)	0.631 (2)	0.544 (2)	0.050*
N3	0.8430 (2)	0.79018 (18)	0.61392 (19)	0.0411 (5)
O1	0.5090 (3)	0.5131 (2)	0.8282 (2)	0.0751 (6)
O2	0.3180 (3)	0.39150 (17)	0.7388 (2)	0.0732 (6)
O3	0.7778 (2)	0.48784 (15)	0.53116 (17)	0.0483 (4)
Cl1	-0.31736 (16)	0.87209 (13)	1.01893 (11)	0.1407 (6)
Cl2	1.29502 (9)	0.98193 (7)	0.63926 (8)	0.0690 (3)
S1	0.36939 (9)	0.51159 (7)	0.76264 (7)	0.0529 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0476 (15)	0.0454 (15)	0.0497 (16)	-0.0040 (12)	-0.0062 (12)	0.0005 (12)
C2	0.0685 (19)	0.062 (2)	0.071 (2)	-0.0128 (16)	-0.0002 (16)	-0.0158 (17)
C3	0.105 (3)	0.062 (2)	0.071 (2)	0.004 (2)	0.011 (2)	-0.0138 (18)
C4	0.079 (2)	0.077 (2)	0.056 (2)	0.026 (2)	0.0122 (17)	0.0121 (18)
C5	0.0474 (17)	0.105 (3)	0.064 (2)	0.0034 (18)	-0.0040 (15)	0.019 (2)
C6	0.0504 (16)	0.0663 (19)	0.0597 (18)	-0.0082 (14)	-0.0104 (14)	0.0043 (15)
C7	0.0380 (13)	0.0404 (15)	0.0759 (19)	-0.0050 (11)	-0.0040 (13)	-0.0151 (13)
C8	0.0374 (12)	0.0324 (13)	0.0450 (14)	-0.0041 (10)	-0.0076 (11)	-0.0029 (11)
C9	0.0360 (12)	0.0333 (13)	0.0513 (15)	-0.0035 (10)	-0.0079 (11)	-0.0032 (11)
C10	0.0392 (12)	0.0289 (12)	0.0452 (14)	-0.0035 (10)	-0.0077 (11)	-0.0018 (11)
C11	0.0402 (13)	0.0371 (13)	0.0525 (16)	-0.0063 (10)	-0.0113 (11)	-0.0021 (12)
C12	0.0604 (17)	0.0397 (15)	0.0690 (19)	-0.0164 (13)	-0.0179 (14)	-0.0070 (14)
C13	0.075 (2)	0.0322 (14)	0.077 (2)	-0.0029 (14)	-0.0153 (16)	-0.0124 (14)
C14	0.0508 (16)	0.0414 (16)	0.086 (2)	0.0051 (13)	-0.0068 (15)	-0.0124 (15)
C15	0.0414 (14)	0.0391 (14)	0.0737 (19)	-0.0058 (11)	-0.0096 (13)	-0.0072 (13)
N1	0.0412 (12)	0.0617 (15)	0.0576 (15)	-0.0181 (11)	-0.0039 (10)	-0.0171 (12)
N2	0.0340 (10)	0.0305 (11)	0.0600 (14)	-0.0033 (9)	-0.0053 (10)	-0.0093 (10)
N3	0.0405 (11)	0.0298 (10)	0.0529 (13)	-0.0052 (9)	-0.0086 (9)	-0.0057 (9)
O1	0.0577 (12)	0.0822 (15)	0.0895 (16)	0.0029 (11)	-0.0313 (11)	-0.0095 (13)
O2	0.0696 (13)	0.0384 (11)	0.1099 (18)	-0.0079 (10)	-0.0132 (12)	-0.0116 (11)
O3	0.0393 (9)	0.0342 (9)	0.0725 (12)	-0.0030 (7)	-0.0115 (8)	-0.0153 (9)
Cl1	0.1177 (9)	0.1400 (10)	0.1047 (8)	0.0695 (8)	0.0332 (7)	0.0151 (7)
Cl2	0.0391 (4)	0.0617 (5)	0.1092 (7)	-0.0066 (3)	-0.0181 (4)	-0.0182 (4)
S1	0.0428 (4)	0.0433 (4)	0.0722 (5)	-0.0029 (3)	-0.0113 (3)	-0.0093 (3)

Geometric parameters (Å, °)

C1—C2	1.380 (4)	C9—C10	1.469 (3)
C1—C6	1.381 (4)	C9—H9	0.9300
C1—S1	1.769 (3)	C10—C11	1.394 (3)
C2—C3	1.377 (4)	C10—C15	1.398 (3)
C2—H2	0.9300	C11—C12	1.385 (3)
C3—C4	1.367 (5)	C11—Cl2	1.745 (2)
C3—H3	0.9300	C12—C13	1.376 (4)
C4—C5	1.369 (5)	C12—H12	0.9300
C4—Cl1	1.736 (3)	C13—C14	1.379 (4)
C5—C6	1.391 (4)	C13—H13	0.9300
C5—H5	0.9300	C14—C15	1.377 (3)
C6—H6	0.9300	C14—H14	0.9300
C7—N1	1.447 (3)	C15—H15	0.9300
C7—C8	1.518 (3)	N1—S1	1.604 (2)
C7—H7A	0.9700	N1—H1N	0.841 (16)
C7—H7B	0.9700	N2—N3	1.379 (3)
C8—O3	1.236 (3)	N2—H2N	0.852 (16)
C8—N2	1.337 (3)	O1—S1	1.4283 (19)

C9—N3	1.271 (3)	O2—S1	1.4312 (19)
C2—C1—C6	120.3 (3)	C11—C10—C9	123.2 (2)
C2—C1—S1	119.8 (2)	C15—C10—C9	120.2 (2)
C6—C1—S1	119.8 (2)	C12—C11—C10	122.1 (2)
C3—C2—C1	119.9 (3)	C12—C11—C12	117.61 (19)
C3—C2—H2	120.1	C10—C11—C12	120.32 (18)
C1—C2—H2	120.1	C13—C12—C11	119.4 (2)
C4—C3—C2	119.6 (3)	C13—C12—H12	120.3
C4—C3—H3	120.2	C11—C12—H12	120.3
C2—C3—H3	120.2	C12—C13—C14	120.2 (2)
C3—C4—C5	121.4 (3)	C12—C13—H13	119.9
C3—C4—C11	119.1 (3)	C14—C13—H13	119.9
C5—C4—C11	119.5 (3)	C15—C14—C13	119.7 (3)
C4—C5—C6	119.4 (3)	C15—C14—H14	120.1
C4—C5—H5	120.3	C13—C14—H14	120.1
C6—C5—H5	120.3	C14—C15—C10	121.9 (2)
C1—C6—C5	119.4 (3)	C14—C15—H15	119.0
C1—C6—H6	120.3	C10—C15—H15	119.0
C5—C6—H6	120.3	C7—N1—S1	122.21 (19)
N1—C7—C8	112.8 (2)	C7—N1—H1N	119 (2)
N1—C7—H7A	109.0	S1—N1—H1N	119 (2)
C8—C7—H7A	109.0	C8—N2—N3	119.52 (19)
N1—C7—H7B	109.0	C8—N2—H2N	119.0 (17)
C8—C7—H7B	109.0	N3—N2—H2N	121.4 (17)
H7A—C7—H7B	107.8	C9—N3—N2	117.61 (19)
O3—C8—N2	121.5 (2)	O1—S1—O2	120.84 (13)
O3—C8—C7	122.1 (2)	O1—S1—N1	107.43 (13)
N2—C8—C7	116.5 (2)	O2—S1—N1	107.08 (12)
N3—C9—C10	119.0 (2)	O1—S1—C1	107.99 (13)
N3—C9—H9	120.5	O2—S1—C1	107.31 (12)
C10—C9—H9	120.5	N1—S1—C1	105.16 (12)
C11—C10—C15	116.6 (2)		
C6—C1—C2—C3	-2.1 (4)	C11—C12—C13—C14	0.5 (4)
S1—C1—C2—C3	174.7 (2)	C12—C13—C14—C15	0.4 (5)
C1—C2—C3—C4	0.9 (5)	C13—C14—C15—C10	-0.8 (4)
C2—C3—C4—C5	1.3 (5)	C11—C10—C15—C14	0.4 (4)
C2—C3—C4—C11	-178.2 (2)	C9—C10—C15—C14	-178.1 (3)
C3—C4—C5—C6	-2.3 (5)	C8—C7—N1—S1	83.6 (3)
C11—C4—C5—C6	177.2 (2)	O3—C8—N2—N3	-179.0 (2)
C2—C1—C6—C5	1.1 (4)	C7—C8—N2—N3	2.6 (3)
S1—C1—C6—C5	-175.7 (2)	C10—C9—N3—N2	179.6 (2)
C4—C5—C6—C1	1.1 (4)	C8—N2—N3—C9	179.1 (2)
N1—C7—C8—O3	17.0 (4)	C7—N1—S1—O1	-17.3 (2)
N1—C7—C8—N2	-164.6 (2)	C7—N1—S1—O2	-148.5 (2)
N3—C9—C10—C11	177.3 (2)	C7—N1—S1—C1	97.6 (2)
N3—C9—C10—C15	-4.3 (4)	C2—C1—S1—O1	36.9 (3)

C15—C10—C11—C12	0.5 (4)	C6—C1—S1—O1	-146.3 (2)
C9—C10—C11—C12	179.0 (2)	C2—C1—S1—O2	168.7 (2)
C15—C10—C11—C12	-178.54 (19)	C6—C1—S1—O2	-14.6 (3)
C9—C10—C11—C12	0.0 (3)	C2—C1—S1—N1	-77.6 (2)
C10—C11—C12—C13	-0.9 (4)	C6—C1—S1—N1	99.2 (2)
Cl2—C11—C12—C13	178.1 (2)		

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

<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 O3 ⁱ	0.84 (2)	2.01 (2)	2.823 (3)	162 (3)
N2—H2N \cdots O3 ⁱⁱ	0.85 (2)	2.06 (2)	2.897 (2)	167 (2)
C12—H12 \cdots O2 ⁱⁱⁱ	0.93	2.53	3.439 (3)	166

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

(E)-4-Chloro-N-{2-[2-(3-chlorobenzylidene)hydrazinyl]-2-oxoethyl}benzenesulfonamide (II)*Crystal data*

$\text{C}_{15}\text{H}_{13}\text{Cl}_2\text{N}_3\text{O}_3\text{S}$

$M_r = 386.24$

Triclinic, $P\bar{1}$

$a = 9.491$ (1) \AA

$b = 9.976$ (1) \AA

$c = 10.446$ (1) \AA

$\alpha = 67.22$ (1) $^\circ$

$\beta = 66.80$ (1) $^\circ$

$\gamma = 86.32$ (1) $^\circ$

$V = 833.59$ (17) \AA^3

$Z = 2$

$F(000) = 396$

$D_x = 1.539$ Mg m^{-3}

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

Cell parameters from 2850 reflections

$\theta = 2.6$ – 27.9°

$\mu = 0.53$ mm^{-1}

$T = 293$ K

Prism, colourless

$0.36 \times 0.14 \times 0.08$ mm

Data collection

Oxford Diffraction Xcalibur with Sapphire

CCD

diffractometer

Radiation source: fine-focus sealed tube

Rotation method data acquisition using ω scans.

Absorption correction: multi-scan

(CrysAlis RED, Oxford Diffraction, 2009)

$T_{\min} = 0.831$, $T_{\max} = 0.959$

5759 measured reflections

3354 independent reflections

2688 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 2.6^\circ$

$h = -11 \rightarrow 11$

$k = -12 \rightarrow 12$

$l = -13 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.108$

$S = 1.22$

3354 reflections

223 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

$w = 1/[\sigma^2(F_o^2) + (0.0148P)^2 + 1.1114P]$

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

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.27$ e \AA^{-3}

$\Delta\rho_{\min} = -0.39$ e \AA^{-3}

Special details

Experimental. CrysAlis RED, Oxford Diffraction Ltd., 2009 Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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.

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2520 (3)	−0.1930 (3)	1.0295 (3)	0.0312 (6)
C2	0.1178 (3)	−0.2828 (3)	1.0900 (4)	0.0408 (7)
H2	0.0241	−0.2549	1.1415	0.049*
C3	0.1226 (4)	−0.4145 (4)	1.0739 (4)	0.0458 (8)
H3	0.0329	−0.4769	1.1166	0.055*
C4	0.2616 (4)	−0.4519 (3)	0.9939 (4)	0.0392 (7)
C5	0.3966 (4)	−0.3626 (3)	0.9327 (4)	0.0393 (7)
H5	0.4898	−0.3896	0.8789	0.047*
C6	0.3924 (3)	−0.2326 (3)	0.9519 (3)	0.0356 (7)
H6	0.4828	−0.1724	0.9131	0.043*
C7	0.2808 (3)	0.1427 (3)	0.7715 (3)	0.0356 (7)
H7A	0.2814	0.2480	0.7273	0.043*
H7B	0.1751	0.0995	0.8162	0.043*
C8	0.3771 (3)	0.0903 (3)	0.6490 (3)	0.0351 (7)
C9	0.1510 (4)	0.1675 (4)	0.4412 (3)	0.0414 (7)
H9	0.2108	0.1262	0.3740	0.050*
C10	0.0084 (4)	0.2250 (3)	0.4311 (3)	0.0400 (7)
C11	−0.0769 (4)	0.3041 (4)	0.5152 (4)	0.0469 (8)
H11	−0.0426	0.3245	0.5783	0.056*
C12	−0.2121 (4)	0.3515 (4)	0.5037 (4)	0.0540 (9)
C13	−0.2654 (4)	0.3247 (4)	0.4096 (5)	0.0600 (10)
H13	−0.3566	0.3589	0.4022	0.072*
C14	−0.1815 (5)	0.2467 (4)	0.3273 (5)	0.0603 (10)
H14	−0.2165	0.2274	0.2640	0.072*
C15	−0.0459 (4)	0.1966 (4)	0.3371 (4)	0.0497 (8)
H15	0.0097	0.1435	0.2808	0.060*
Cl1	0.26935 (11)	−0.61454 (10)	0.96808 (12)	0.0584 (3)
Cl2	−0.32203 (15)	0.44557 (16)	0.61290 (17)	0.0976 (5)
N1	0.3402 (3)	0.1031 (3)	0.8891 (3)	0.0318 (5)
H1N	0.436 (2)	0.101 (3)	0.860 (3)	0.038*
N2	0.3309 (3)	0.1124 (3)	0.5371 (3)	0.0436 (7)
H2N	0.378 (4)	0.072 (4)	0.474 (3)	0.052*
N3	0.1952 (3)	0.1727 (3)	0.5398 (3)	0.0394 (6)

O1	0.3340 (2)	-0.0350 (2)	1.1413 (2)	0.0411 (5)
O2	0.0898 (2)	0.0034 (2)	1.1055 (2)	0.0408 (5)
O3	0.4936 (2)	0.0311 (3)	0.6515 (2)	0.0475 (6)
S1	0.24739 (8)	-0.02589 (8)	1.05320 (8)	0.03071 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0333 (15)	0.0300 (15)	0.0385 (16)	0.0073 (12)	-0.0189 (13)	-0.0176 (13)
C2	0.0280 (16)	0.0415 (18)	0.057 (2)	0.0072 (13)	-0.0145 (15)	-0.0268 (16)
C3	0.0347 (17)	0.0386 (18)	0.069 (2)	0.0006 (14)	-0.0209 (17)	-0.0261 (17)
C4	0.0461 (19)	0.0299 (16)	0.0530 (19)	0.0096 (14)	-0.0279 (16)	-0.0204 (15)
C5	0.0347 (17)	0.0377 (17)	0.0496 (19)	0.0124 (13)	-0.0173 (15)	-0.0224 (15)
C6	0.0291 (15)	0.0343 (16)	0.0469 (18)	0.0061 (12)	-0.0159 (14)	-0.0192 (14)
C7	0.0354 (16)	0.0374 (17)	0.0375 (16)	0.0116 (13)	-0.0156 (14)	-0.0185 (14)
C8	0.0341 (16)	0.0371 (17)	0.0339 (16)	0.0086 (13)	-0.0129 (13)	-0.0153 (14)
C9	0.0435 (18)	0.050 (2)	0.0326 (16)	0.0112 (15)	-0.0145 (14)	-0.0195 (15)
C10	0.0422 (18)	0.0414 (18)	0.0329 (16)	0.0052 (14)	-0.0150 (14)	-0.0113 (14)
C11	0.047 (2)	0.054 (2)	0.0451 (19)	0.0126 (16)	-0.0246 (16)	-0.0199 (17)
C12	0.048 (2)	0.052 (2)	0.052 (2)	0.0109 (17)	-0.0181 (18)	-0.0131 (18)
C13	0.044 (2)	0.060 (2)	0.067 (3)	0.0048 (18)	-0.031 (2)	-0.006 (2)
C14	0.065 (3)	0.060 (2)	0.062 (2)	-0.004 (2)	-0.042 (2)	-0.011 (2)
C15	0.062 (2)	0.047 (2)	0.0440 (19)	0.0028 (17)	-0.0269 (18)	-0.0156 (16)
Cl1	0.0641 (6)	0.0405 (5)	0.0897 (7)	0.0132 (4)	-0.0367 (5)	-0.0401 (5)
Cl2	0.0847 (9)	0.1137 (11)	0.1072 (10)	0.0590 (8)	-0.0404 (8)	-0.0618 (9)
N1	0.0274 (12)	0.0354 (13)	0.0368 (14)	0.0033 (11)	-0.0109 (11)	-0.0205 (11)
N2	0.0416 (16)	0.0597 (18)	0.0408 (15)	0.0224 (13)	-0.0198 (13)	-0.0303 (14)
N3	0.0378 (14)	0.0457 (16)	0.0368 (14)	0.0134 (12)	-0.0165 (12)	-0.0183 (12)
O1	0.0400 (12)	0.0535 (14)	0.0406 (12)	0.0020 (10)	-0.0200 (10)	-0.0249 (11)
O2	0.0309 (11)	0.0474 (13)	0.0512 (13)	0.0071 (9)	-0.0115 (10)	-0.0320 (11)
O3	0.0404 (13)	0.0727 (16)	0.0418 (13)	0.0248 (12)	-0.0208 (11)	-0.0336 (12)
S1	0.0279 (4)	0.0352 (4)	0.0363 (4)	0.0050 (3)	-0.0127 (3)	-0.0219 (3)

Geometric parameters (Å, °)

C1—C2	1.377 (4)	C9—C10	1.462 (4)
C1—C6	1.385 (4)	C9—H9	0.9300
C1—S1	1.773 (3)	C10—C15	1.394 (4)
C2—C3	1.384 (4)	C10—C11	1.394 (4)
C2—H2	0.9300	C11—C12	1.372 (5)
C3—C4	1.374 (4)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.381 (5)
C4—C5	1.380 (4)	C12—Cl2	1.743 (4)
C4—Cl1	1.737 (3)	C13—C14	1.370 (6)
C5—C6	1.382 (4)	C13—H13	0.9300
C5—H5	0.9300	C14—C15	1.378 (5)
C6—H6	0.9300	C14—H14	0.9300
C7—N1	1.457 (4)	C15—H15	0.9300

C7—C8	1.510 (4)	N1—S1	1.618 (3)
C7—H7A	0.9700	N1—H1N	0.841 (17)
C7—H7B	0.9700	N2—N3	1.380 (3)
C8—O3	1.224 (3)	N2—H2N	0.863 (18)
C8—N2	1.342 (4)	O1—S1	1.433 (2)
C9—N3	1.275 (4)	O2—S1	1.430 (2)
C2—C1—C6	120.8 (3)	C15—C10—C9	118.8 (3)
C2—C1—S1	120.2 (2)	C11—C10—C9	122.2 (3)
C6—C1—S1	119.0 (2)	C12—C11—C10	119.3 (3)
C1—C2—C3	119.9 (3)	C12—C11—H11	120.4
C1—C2—H2	120.0	C10—C11—H11	120.4
C3—C2—H2	120.0	C11—C12—C13	121.9 (4)
C4—C3—C2	119.1 (3)	C11—C12—Cl2	119.4 (3)
C4—C3—H3	120.4	C13—C12—Cl2	118.7 (3)
C2—C3—H3	120.4	C14—C13—C12	118.7 (3)
C3—C4—C5	121.4 (3)	C14—C13—H13	120.6
C3—C4—Cl1	119.8 (2)	C12—C13—H13	120.6
C5—C4—Cl1	118.8 (2)	C13—C14—C15	120.9 (4)
C4—C5—C6	119.5 (3)	C13—C14—H14	119.6
C4—C5—H5	120.3	C15—C14—H14	119.6
C6—C5—H5	120.3	C14—C15—C10	120.3 (4)
C5—C6—C1	119.3 (3)	C14—C15—H15	119.9
C5—C6—H6	120.4	C10—C15—H15	119.9
C1—C6—H6	120.4	C7—N1—S1	119.9 (2)
N1—C7—C8	111.0 (2)	C7—N1—H1N	116 (2)
N1—C7—H7A	109.4	S1—N1—H1N	112 (2)
C8—C7—H7A	109.4	C8—N2—N3	120.8 (2)
N1—C7—H7B	109.4	C8—N2—H2N	117 (2)
C8—C7—H7B	109.4	N3—N2—H2N	121 (2)
H7A—C7—H7B	108.0	C9—N3—N2	115.1 (3)
O3—C8—N2	121.4 (3)	O2—S1—O1	119.53 (13)
O3—C8—C7	121.1 (3)	O2—S1—N1	107.80 (13)
N2—C8—C7	117.5 (3)	O1—S1—N1	105.65 (13)
N3—C9—C10	121.6 (3)	O2—S1—C1	107.23 (13)
N3—C9—H9	119.2	O1—S1—C1	107.84 (13)
C10—C9—H9	119.2	N1—S1—C1	108.40 (13)
C15—C10—C11	119.0 (3)		
C6—C1—C2—C3	0.2 (5)	C12—C12—C13—C14	-178.0 (3)
S1—C1—C2—C3	-178.6 (3)	C12—C13—C14—C15	-0.4 (6)
C1—C2—C3—C4	-1.7 (5)	C13—C14—C15—C10	-0.2 (6)
C2—C3—C4—C5	1.5 (5)	C11—C10—C15—C14	0.3 (5)
C2—C3—C4—Cl1	-178.3 (3)	C9—C10—C15—C14	178.7 (3)
C3—C4—C5—C6	0.0 (5)	C8—C7—N1—S1	-107.2 (3)
Cl1—C4—C5—C6	179.8 (2)	O3—C8—N2—N3	176.8 (3)
C4—C5—C6—C1	-1.4 (5)	C7—C8—N2—N3	-3.8 (5)
C2—C1—C6—C5	1.3 (5)	C10—C9—N3—N2	179.1 (3)

S1—C1—C6—C5	-179.8 (2)	C8—N2—N3—C9	-169.5 (3)
N1—C7—C8—O3	-4.1 (4)	C7—N1—S1—O2	-50.2 (2)
N1—C7—C8—N2	176.5 (3)	C7—N1—S1—O1	-179.0 (2)
N3—C9—C10—C15	-170.4 (3)	C7—N1—S1—C1	65.6 (2)
N3—C9—C10—C11	8.0 (5)	C2—C1—S1—O2	-13.7 (3)
C15—C10—C11—C12	0.2 (5)	C6—C1—S1—O2	167.4 (2)
C9—C10—C11—C12	-178.2 (3)	C2—C1—S1—O1	116.3 (3)
C10—C11—C12—C13	-0.8 (6)	C6—C1—S1—O1	-62.6 (3)
C10—C11—C12—C12	178.1 (3)	C2—C1—S1—N1	-129.8 (3)
C11—C12—C13—C14	0.9 (6)	C6—C1—S1—N1	51.3 (3)

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

Cg is the centroid of the C1–C6 ring.

<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	0.84 (2)	2.23 (2)	3.041 (3)	161 (3)
N2—H2N \cdots O3 ⁱⁱ	0.86 (2)	1.97 (2)	2.828 (3)	171 (3)
C6—H6 \cdots O1 ⁱ	0.93	2.53	3.422 (4)	161
C7—H7B \cdots O2 ⁱⁱⁱ	0.97	2.47	3.434 (4)	172
C15—H15 \cdots O2 ^{iv}	0.93	2.58	3.474 (4)	162
C14—H14 \cdots Cg ^v	0.93	2.84	3.675 (5)	150

Symmetry codes: (i) $-x+1, -y, -z+2$; (ii) $-x+1, -y, -z+1$; (iii) $-x, -y, -z+2$; (iv) $x, y, z-1$; (v) $-x, -y, -z+1$.**(*E*)-4-Chloro-*N*-{2-[2-(4-chlorobenzylidene)hydrazinyl]-2-oxoethyl}benzenesulfonamide (III)***Crystal data*C₁₅H₁₃Cl₂N₃O₃S $M_r = 386.24$ Triclinic, *P* $\bar{1}$ $a = 6.7234$ (9) \AA $b = 10.281$ (1) \AA $c = 13.611$ (2) \AA $\alpha = 74.98$ (1) $^\circ$ $\beta = 87.11$ (1) $^\circ$ $\gamma = 75.34$ (1) $^\circ$ $V = 879.0$ (2) \AA^3 $Z = 2$ $F(000) = 396$ $D_x = 1.459$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ \AA

Cell parameters from 1746 reflections

 $\theta = 2.9$ – 27.9 $^\circ$ $\mu = 0.51$ mm⁻¹ $T = 293$ K

Rod, colourless

 $0.46 \times 0.42 \times 0.20$ mm*Data collection*

Oxford Diffraction Xcalibur with Sapphire

CCD

diffractometer

Radiation source: fine-focus sealed tube

Rotation method data acquisition using ω scans.

Absorption correction: multi-scan

(CrysAlis RED, Oxford Diffraction, 2009)

 $T_{\min} = 0.801$, $T_{\max} = 0.906$

5737 measured reflections

3598 independent reflections

2600 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.019$ $\theta_{\max} = 26.4$ $^\circ$, $\theta_{\min} = 2.9$ $^\circ$ $h = -8$ → 8 $k = -12$ → 12 $l = -13$ → 16

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.048$ $wR(F^2) = 0.120$ $S = 1.03$

3598 reflections

223 parameters

2 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 0.5495P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$ *Special details***Experimental.** CrysAlis RED, Oxford Diffraction Ltd., 2009 Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.**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.**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 (\AA^2)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	-0.75370 (19)	0.81534 (16)	0.50474 (9)	0.1284 (5)
Cl2	1.20248 (15)	0.42711 (14)	0.39838 (7)	0.1119 (4)
S1	-0.27969 (10)	1.01993 (7)	0.11321 (5)	0.04891 (19)
O1	-0.1031 (3)	1.05461 (19)	0.14290 (15)	0.0609 (5)
O2	-0.4241 (3)	1.12132 (19)	0.04119 (15)	0.0628 (5)
O3	-0.1441 (3)	0.66440 (19)	0.00307 (15)	0.0601 (5)
N1	-0.1982 (3)	0.8899 (2)	0.06592 (17)	0.0518 (5)
H1N	-0.285 (4)	0.868 (3)	0.038 (2)	0.062*
N2	0.1718 (3)	0.5636 (2)	0.07085 (17)	0.0529 (5)
H2N	0.179 (4)	0.490 (2)	0.050 (2)	0.063*
N3	0.3257 (3)	0.5661 (2)	0.13326 (15)	0.0490 (5)
C1	-0.4154 (4)	0.9674 (3)	0.2247 (2)	0.0487 (6)
C2	-0.3286 (5)	0.9436 (4)	0.3189 (2)	0.0707 (8)
H2	-0.199533	0.958848	0.324412	0.085*
C3	-0.4330 (5)	0.8971 (4)	0.4049 (2)	0.0840 (10)
H3	-0.374571	0.880441	0.468907	0.101*
C4	-0.6231 (5)	0.8754 (4)	0.3961 (3)	0.0767 (9)
C5	-0.7090 (5)	0.8990 (4)	0.3026 (3)	0.0909 (11)
H5	-0.837854	0.883342	0.297257	0.109*
C6	-0.6066 (5)	0.9456 (4)	0.2165 (2)	0.0773 (9)
H6	-0.666116	0.962570	0.152735	0.093*
C7	-0.0149 (4)	0.7821 (3)	0.10526 (19)	0.0471 (6)

H7A	-0.022691	0.748572	0.178490	0.057*
H7B	0.105960	0.818535	0.089975	0.057*
C8	-0.0016 (4)	0.6659 (3)	0.05555 (18)	0.0466 (6)
C9	0.4825 (4)	0.4633 (3)	0.1450 (2)	0.0521 (6)
H9	0.486624	0.393537	0.112435	0.063*
C10	0.6568 (4)	0.4535 (3)	0.20951 (19)	0.0480 (6)
C11	0.6490 (4)	0.5479 (3)	0.2675 (2)	0.0606 (7)
H11	0.530130	0.618121	0.266545	0.073*
C12	0.8152 (5)	0.5385 (3)	0.3263 (2)	0.0682 (8)
H12	0.808568	0.601525	0.365387	0.082*
C13	0.9906 (4)	0.4354 (4)	0.3268 (2)	0.0648 (8)
C14	1.0018 (4)	0.3406 (3)	0.2719 (2)	0.0692 (8)
H14	1.120998	0.270324	0.273761	0.083*
C15	0.8334 (4)	0.3497 (3)	0.2128 (2)	0.0596 (7)
H15	0.840415	0.284985	0.175185	0.071*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1225 (9)	0.1721 (12)	0.0862 (7)	-0.0563 (9)	0.0375 (7)	-0.0130 (8)
Cl2	0.0737 (6)	0.1848 (12)	0.0779 (6)	-0.0439 (7)	-0.0247 (5)	-0.0185 (7)
S1	0.0460 (3)	0.0478 (4)	0.0565 (4)	-0.0123 (3)	-0.0034 (3)	-0.0181 (3)
O1	0.0516 (10)	0.0629 (12)	0.0799 (13)	-0.0225 (9)	-0.0016 (9)	-0.0303 (10)
O2	0.0616 (12)	0.0518 (11)	0.0695 (13)	-0.0097 (9)	-0.0111 (9)	-0.0076 (9)
O3	0.0520 (10)	0.0608 (11)	0.0742 (13)	-0.0063 (9)	-0.0165 (9)	-0.0329 (10)
N1	0.0454 (12)	0.0572 (13)	0.0591 (14)	-0.0092 (10)	-0.0070 (10)	-0.0277 (11)
N2	0.0500 (12)	0.0529 (13)	0.0604 (14)	-0.0070 (10)	-0.0122 (10)	-0.0258 (11)
N3	0.0473 (12)	0.0524 (13)	0.0495 (12)	-0.0115 (10)	-0.0077 (9)	-0.0161 (10)
C1	0.0438 (13)	0.0487 (14)	0.0566 (15)	-0.0078 (11)	-0.0012 (11)	-0.0215 (12)
C2	0.0552 (17)	0.103 (3)	0.0606 (18)	-0.0226 (16)	-0.0005 (14)	-0.0302 (18)
C3	0.076 (2)	0.119 (3)	0.0541 (19)	-0.019 (2)	-0.0019 (16)	-0.0212 (19)
C4	0.072 (2)	0.090 (2)	0.066 (2)	-0.0230 (18)	0.0196 (16)	-0.0157 (18)
C5	0.061 (2)	0.139 (3)	0.080 (2)	-0.044 (2)	0.0082 (17)	-0.023 (2)
C6	0.0577 (18)	0.118 (3)	0.0617 (19)	-0.0349 (18)	-0.0015 (15)	-0.0199 (19)
C7	0.0455 (13)	0.0516 (14)	0.0475 (14)	-0.0129 (11)	-0.0030 (11)	-0.0169 (12)
C8	0.0462 (13)	0.0511 (14)	0.0455 (14)	-0.0142 (11)	-0.0029 (11)	-0.0145 (11)
C9	0.0533 (15)	0.0527 (15)	0.0548 (15)	-0.0139 (12)	-0.0041 (12)	-0.0200 (12)
C10	0.0469 (13)	0.0483 (14)	0.0483 (14)	-0.0115 (11)	-0.0032 (11)	-0.0110 (11)
C11	0.0581 (16)	0.0583 (17)	0.0647 (18)	-0.0068 (13)	-0.0100 (13)	-0.0200 (14)
C12	0.075 (2)	0.077 (2)	0.0606 (18)	-0.0238 (17)	-0.0107 (15)	-0.0244 (16)
C13	0.0557 (17)	0.089 (2)	0.0481 (16)	-0.0261 (16)	-0.0076 (13)	-0.0056 (15)
C14	0.0499 (16)	0.075 (2)	0.0688 (19)	-0.0025 (14)	-0.0039 (14)	-0.0055 (16)
C15	0.0584 (16)	0.0557 (16)	0.0644 (18)	-0.0101 (13)	-0.0019 (13)	-0.0183 (14)

Geometric parameters (Å, °)

Cl1—C4	1.735 (3)	C4—C5	1.362 (5)
Cl2—C13	1.738 (3)	C5—C6	1.368 (4)

S1—O1	1.4275 (18)	C5—H5	0.9300
S1—O2	1.4297 (19)	C6—H6	0.9300
S1—N1	1.594 (2)	C7—C8	1.500 (3)
S1—C1	1.764 (3)	C7—H7A	0.9700
O3—C8	1.229 (3)	C7—H7B	0.9700
N1—C7	1.450 (3)	C9—C10	1.467 (3)
N1—H1N	0.818 (17)	C9—H9	0.9300
N2—C8	1.341 (3)	C10—C15	1.375 (4)
N2—N3	1.380 (3)	C10—C11	1.390 (4)
N2—H2N	0.861 (17)	C11—C12	1.377 (4)
N3—C9	1.273 (3)	C11—H11	0.9300
C1—C2	1.373 (4)	C12—C13	1.371 (4)
C1—C6	1.374 (4)	C12—H12	0.9300
C2—C3	1.375 (4)	C13—C14	1.360 (4)
C2—H2	0.9300	C14—C15	1.392 (4)
C3—C4	1.369 (5)	C14—H14	0.9300
C3—H3	0.9300	C15—H15	0.9300
O1—S1—O2	119.70 (12)	N1—C7—C8	107.76 (19)
O1—S1—N1	107.02 (11)	N1—C7—H7A	110.2
O2—S1—N1	106.60 (12)	C8—C7—H7A	110.2
O1—S1—C1	107.38 (12)	N1—C7—H7B	110.2
O2—S1—C1	107.41 (12)	C8—C7—H7B	110.2
N1—S1—C1	108.32 (12)	H7A—C7—H7B	108.5
C7—N1—S1	121.73 (16)	O3—C8—N2	121.3 (2)
C7—N1—H1N	117 (2)	O3—C8—C7	121.3 (2)
S1—N1—H1N	116 (2)	N2—C8—C7	117.4 (2)
C8—N2—N3	119.7 (2)	N3—C9—C10	120.7 (2)
C8—N2—H2N	119.9 (19)	N3—C9—H9	119.7
N3—N2—H2N	119.6 (19)	C10—C9—H9	119.7
C9—N3—N2	115.3 (2)	C15—C10—C11	118.6 (2)
C2—C1—C6	120.1 (3)	C15—C10—C9	119.8 (2)
C2—C1—S1	120.5 (2)	C11—C10—C9	121.6 (2)
C6—C1—S1	119.3 (2)	C12—C11—C10	120.8 (3)
C1—C2—C3	119.8 (3)	C12—C11—H11	119.6
C1—C2—H2	120.1	C10—C11—H11	119.6
C3—C2—H2	120.1	C13—C12—C11	119.3 (3)
C4—C3—C2	119.7 (3)	C13—C12—H12	120.3
C4—C3—H3	120.1	C11—C12—H12	120.3
C2—C3—H3	120.1	C14—C13—C12	121.2 (3)
C5—C4—C3	120.4 (3)	C14—C13—C12	119.7 (3)
C5—C4—C11	119.9 (3)	C12—C13—C12	119.1 (2)
C3—C4—C11	119.7 (3)	C13—C14—C15	119.5 (3)
C4—C5—C6	120.3 (3)	C13—C14—H14	120.3
C4—C5—H5	119.8	C15—C14—H14	120.3
C6—C5—H5	119.8	C10—C15—C14	120.6 (3)
C5—C6—C1	119.7 (3)	C10—C15—H15	119.7
C5—C6—H6	120.2	C14—C15—H15	119.7

C1—C6—H6	120.2		
O1—S1—N1—C7	35.4 (2)	S1—C1—C6—C5	177.1 (3)
O2—S1—N1—C7	164.6 (2)	S1—N1—C7—C8	171.09 (18)
C1—S1—N1—C7	-80.1 (2)	N3—N2—C8—O3	-177.6 (2)
C8—N2—N3—C9	178.5 (2)	N3—N2—C8—C7	2.2 (4)
O1—S1—C1—C2	-10.8 (3)	N1—C7—C8—O3	-9.0 (3)
O2—S1—C1—C2	-140.8 (2)	N1—C7—C8—N2	171.1 (2)
N1—S1—C1—C2	104.5 (2)	N2—N3—C9—C10	180.0 (2)
O1—S1—C1—C6	171.5 (2)	N3—C9—C10—C15	-173.1 (3)
O2—S1—C1—C6	41.5 (3)	N3—C9—C10—C11	6.4 (4)
N1—S1—C1—C6	-73.3 (3)	C15—C10—C11—C12	0.6 (4)
C6—C1—C2—C3	0.4 (5)	C9—C10—C11—C12	-179.0 (3)
S1—C1—C2—C3	-177.3 (3)	C10—C11—C12—C13	0.5 (5)
C1—C2—C3—C4	-0.2 (5)	C11—C12—C13—C14	-1.4 (5)
C2—C3—C4—C5	0.3 (6)	C11—C12—C13—C12	178.2 (2)
C2—C3—C4—C11	179.5 (3)	C12—C13—C14—C15	1.1 (5)
C3—C4—C5—C6	-0.5 (6)	C12—C13—C14—C15	-178.5 (2)
C11—C4—C5—C6	-179.7 (3)	C11—C10—C15—C14	-0.9 (4)
C4—C5—C6—C1	0.6 (6)	C9—C10—C15—C14	178.7 (3)
C2—C1—C6—C5	-0.6 (5)	C13—C14—C15—C10	0.1 (5)

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...O2 ⁱ	0.82 (2)	2.25 (2)	3.032 (3)	162 (3)
N1—H1N...O3	0.82 (2)	2.23 (3)	2.609 (3)	109 (2)
N2—H2N...O3 ⁱⁱ	0.86 (2)	1.98 (2)	2.829 (3)	170 (3)
C15—H15...O1 ⁱⁱⁱ	0.93	2.45	3.330 (3)	157

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