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Crystal structures, Hirshfeld surface analysis and interaction energies of (Z)-2-(4-methylbenzylidene)- and (Z)-2-(furfurylidene)-2H-benzo[b][1,4]-thiazin-3(4H)-one

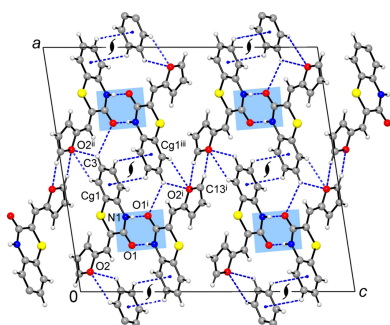
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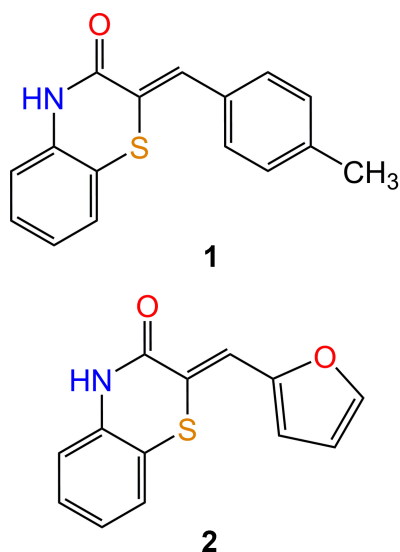
Two new 2-arylmethylidene derivatives of benzo-1,4-thiazin-3-one, namely, (Z)-2-(4-methylbenzylidene)-2H-benzo[b][1,4]thiazin-3(4H)-one, C₁₆H₁₃NOS, **1**, and (Z)-2-(furan-2-ylmethylidene)-2H-benzo[b][1,4]thiazin-3(4H)-one, C₁₃H₉NO₂S, **2**, are rare examples of a nearly planar structure of the 1,4-thiazin-3-one core stabilized by conjugation. Their supramolecular structures are very similar, being dominated by assembly of inversion dimers through highly directional reciprocal N—H...O bonds [N...O = 2.822 (2) Å for **1**; 2.881 (3) Å for **2**]. Weaker forces are represented by C—H...O, C—H... π and stacking interactions, with more interactions in the case of furfurylidene **2**, and they are important for consolidation of the structures. This is consistent with the results of Hirshfeld surface analysis and calculated interaction energies. Doubling the number of O atoms, when moving from **1** to **2**, results in even larger increase in fractions of O...H/H...O contacts [7.6 to 18.6%] due to extensive interactions with the furyl-O acceptor and this contributes to higher packing index in the case of **2**. The far superior energetics in the structures are related with the formation of hydrogen-bonded dimers [−73.3 and −72.9 kJ mol^{−1}, for **1** and **2**, respectively], followed by dispersion forces and weak C—H...O bonding. Identification of reliable 1,4-thiazin-3-one based supramolecular synthons is important for selective targeting for biomedical applications.

1. Chemical context

Heterocyclic compounds containing both sulfur and nitrogen atoms have garnered considerable interest due to their wide range of biological activities (Sebbar *et al.*, 2016a, 2020a; Armenise *et al.*, 2012). Among these, the 1,4-benzothiazine ring system stands out as a privileged scaffold in medicinal chemistry (Trapani *et al.*, 1985; Gupta *et al.*, 1985). Its distinctive physicochemical properties allow for versatile chemical modifications and intense interactions with biological targets (Tawada *et al.*, 1990; Sebbar *et al.*, 2020b). As a result, numerous 1,4-benzothiazine derivatives have exhibited significant pharmacological properties, including anti-oxidant (Zia-ur-Rehman *et al.*, 2009), antipyretic (Warren *et al.*, 1987), anti-cancer (Gupta *et al.*, 1991) and anti-inflammatory activities (Gowda *et al.*, 2011). 1,4-Benzothiazine compounds have also demonstrated their effectiveness as intermediates in the synthesis of new bioactive and anti-corrosion derivatives (Sebbar *et al.*, 2015; Ellouz *et al.*, 2017a; Hni *et al.*, 2019a)



possessing anti-diabetic (Tawada *et al.*, 1990) and anti-corrosion activities (Sebbar *et al.*, 2016b; Hni *et al.*, 2019b; Ellouz *et al.*, 2017b, 2018). Given the promising applications of 1,4-benzothiazine derivatives, we undertook the synthesis of new compounds belonging to this class. To this end 3,4-dihydro-2*H*-1,4-benzothiazin-3-one were condensed with 4-methylbenzaldehyde, **2a**, or furan-2-carbaldehyde, respectively, in the presence of excess sodium methoxide in dimethylformamide (DMF). The resulting products (*Z*)-2-(4-methylbenzylidene)-2*H*-benzo[*b*][1,4]thiazin-3(4*H*)-one, **1**, and (*Z*)-2-(furfurylidene)-3,4-dihydro-2*H*-1,4-benzothiazin-3-one, **2**, were characterized by single-crystal X-ray diffraction and Hirshfeld surface analysis.



2. Structural commentary

The molecular structures of the title compounds **1** and **2** are shown in Figs. 1 and 2, respectively. Both of them adopt a *Z*-configuration about the ethene bond. In **1**, the planes of the two aromatic counterparts, namely 1,4-benzothiazin-3(4*H*)-one and tolyl, sustain a relatively small dihedral angle of 11.09 (10)°, whereas in **2** the coplanar configuration of two ring systems is violated more significantly and an appreciable interplanar angle of 26.85 (9)° is developed due to the twist

about C9—C10 bond with torsion angle C8—C9—C10—C11 being 18.1 (4)°. The most salient feature of the molecular frameworks is the nearly planar geometries of the benzothiazinone cores themselves. The r.m.s. deviations from the S1/N1/C1/C6—C8 planes are 0.0247 (13) Å for **1** and 0.0482 (15) Å for **2**, and such flattening has only few precedents, for example 2-(nitromethylidene)-2*H*-1,4-benzothiazin-3(4*H*)-one (Bereztovskaya *et al.*, 2006). Any analogs with *sp*³ ring atoms C² adopt a half-chair conformation due to loss of conjugation along S¹—C²—C³(O)—N⁴ chain and they exist rather as cyclic amides, *e.g.* the 2*H*-1,4-benzothiazin-3(4*H*)-one with a dihedral angle between the S¹/C⁹/C¹⁰ and N⁴/C³/O/C² planes φ = 22.7° (Mague & Ouzidan, 2024). The latter parameter is more informative than the usually considered bent angle along the S¹...N⁴ axis, being indicative of the amide group involvement in the conjugation. For the title compounds **1** and **2**, the angles φ subtended by the S1/C1/C6 and N1/C7/O1/C8 planes are 3.36 (12) and 5.75 (14)°, respectively, and they suggest nearly coplanar arrangements. More interesting that N-substitution of 2-(methylidene)-species has the same destructive impact on conjugation as the involvement of the ring C*sp*³ atom. In this way, the heteroring in the 4-hexyl analog of **1** is non-planar to the same extent as in the above non-aromatic 2*H*-1,4-benzothiazin-3(4*H*)-one [φ = 20.85°; Sebbar *et al.*, 2020b]. One can postulate that the essential penalty to the conjugation in these 4-*R*-substituted species originates in steric *peri*-interaction with the 4-*R* group, similarly to dearomatization of 1-methyl-2-quinolones by *peri*-substituents (Chen *et al.*, 2013). In the present case, the ring may be more sensitive to such factor since even 5-*H* species experience this effect and therefore the category of planar 1,4-benzothiazin-3(4*H*)-ones is restricted to 2-(methylidene)- and N-unsubstituted species. The appreciable enhancement of conjugation in the title compounds when compared to their 4-substituted analogs is also visible from bond lengths in the C6—N1—C7—O1 chain. In particular, the C6—N1 and N1—C7 bonds [1.401 (2), 1.350 (3) and 1.409 (3), 1.342 (3) Å for **1** and **2**, respectively] are both shorter than in the 4-hexyl analog of **1** [1.4207 (17) and 1.3687 (17) Å; Sebbar *et al.*, 2020a], and this coincides with a certain elongation of the C7=O1 bonds, which are 1.2310 (15) in the latter case, but 1.242 (2) and 1.242 (3) Å in **1** and **2**, respectively.

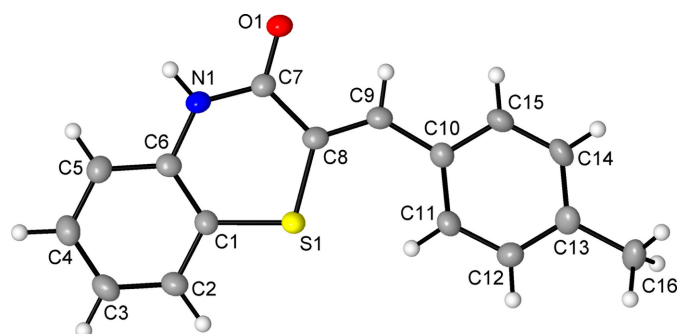


Figure 1

The molecular structure of compound **1**, with atom labelling and displacement ellipsoids drawn at the 50% probability level.

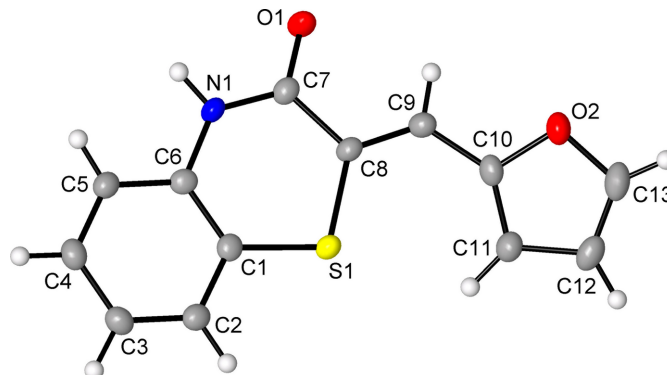


Figure 2

The molecular structure of compound **2**, with atom labelling and displacement ellipsoids drawn at the 50% probability level.

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

<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...O1 ⁱ	0.87 (3)	1.95 (3)	2.822 (2)	177 (3)
C4—H4...Cg(C1—C6) ⁱⁱ	0.98 (2)	2.94 (2)	3.687 (3)	134 (2)
C5—H5...O1 ⁱ	0.93 (2)	2.74 (2)	3.440 (3)	132.4 (16)
C16—H16B...Cg(C10—C15) ⁱⁱⁱ	0.96 (4)	2.90 (4)	3.813 (3)	158 (2)
C16—H16C...S1 ^{iv}	0.98 (3)	3.02 (3)	3.865 (3)	146 (2)

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, y+1, z$; (iv) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

3. Supramolecular features

The closely related supramolecular structures of the title compounds are primarily governed by relatively strong hydrogen bonding accompanied with a set of weak hydrogen bonds and stacking interactions. Two mutual N1—H...O1ⁱ bonds complemented by a pair of secondary C5—H...O1ⁱ bonds assemble the molecules into the inversion dimers [symmetry code (i) for **1**: $-x+1, -y, -z+1$; for **2**: $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+\frac{1}{2}$] (Fig. 3). The formation of such dimers dominates the crystal chemistry of many amide-related species, with a median of N...O length distribution at 2.95 Å (McMahon *et al.*, 2005). In the present cases these distances are shorter [2.822 (2) and 2.881 (3) Å for **1** and **2**, respectively;

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

<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...O1 ⁱ	0.89 (3)	2.00 (3)	2.881 (3)	177 (3)
C2—H2...O2 ⁱⁱ	0.96 (3)	2.81 (2)	3.446 (3)	124.2 (19)
C3—H3...O1 ⁱⁱⁱ	0.99 (3)	2.85 (3)	3.573 (3)	130.2 (19)
C3—H3...O2 ⁱⁱ	0.99 (3)	2.78 (3)	3.420 (3)	122.6 (19)
C4—H4...Cg(C1—C6) ^{iv}	0.97 (3)	3.07 (3)	3.688 (3)	123 (2)
C5—H5...O1 ⁱ	0.93 (3)	2.73 (2)	3.490 (3)	138.8 (18)
C11—H11...Cg(S1/N1/C1/C6—C8) ^v	0.98 (3)	3.06 (3)	3.749 (3)	129 (2)
C13—H13...O2 ^{vi}	0.93 (3)	2.68 (3)	3.359 (3)	130 (2)

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+\frac{1}{2}$; (ii) $x+\frac{1}{2}, -y, z$; (iii) $x+\frac{1}{2}, -y+1, z$; (iv) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $x, y-1, z$; (vi) $-x, -y, -z$.

Tables 1 and 2], as a consequence of stronger interactions between more polarized donors and acceptors NH^{δ+}...C—O^{δ−}, similarly to an even stronger bonding of 2-pyridone in its monoclinic polymorph [2.745 (2) and 2.792 (2) Å; Arman *et al.*, 2009]. With respect to the combined N—H...O and C—H...O bonding, the observed dimers may be best related to a similar motif in α -thiazine-indigo [N...O = 2.828 (3) and C...O = 3.492 (5) Å; Buchsbaum *et al.*, 2011]. At the same time, the comparable molecular configurations of 2*H*-benzo[*b*] [1,4]thiazin-3(4*H*)-one 1,1-dioxide (Irrou *et al.*, 2023) and thiomorpholin-3-one (Ramasubbu *et al.*, 1988) do not support formation of dimers. These hydrogen-bonding preferences of benzothiazinones are interesting in view of their selective targeting of Ser293 in the active region of acetylcholine esterase (Haji Ali *et al.*, 2022).

In **1**, the dimers are further stacked into the columns along the *b*-axis direction (Fig. 3). Within the column, one can distinguish mutual interactions of carbonyl groups, which are similar to lone-pair- π hole bonding in nitro compounds [C7...O1^{iv} = 3.173 (3) Å; symmetry code: (iv) $-x+1, -y+1$,

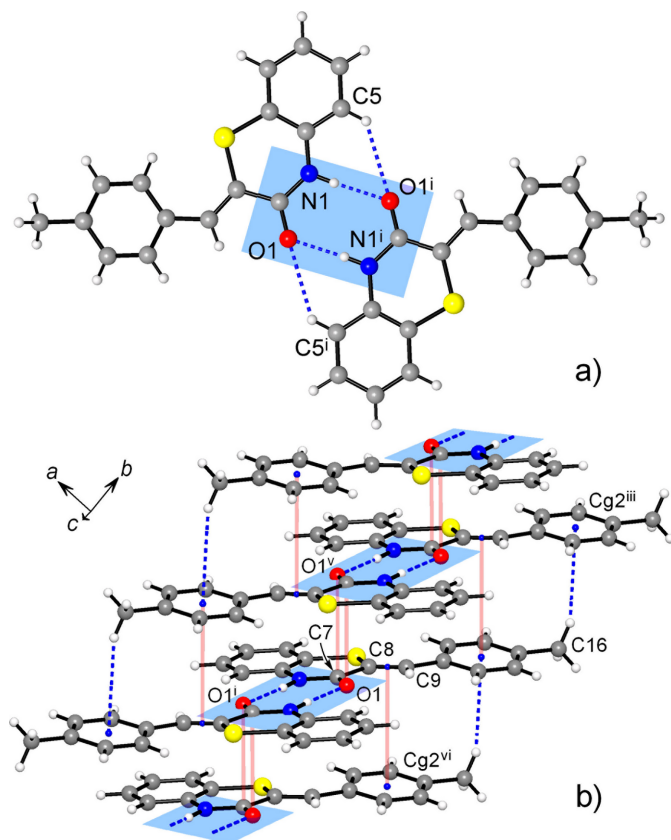


Figure 3
(a) Inversion-related hydrogen-bonded dimers in the structure of **1**; (b) Slipped stacking of the dimers generates columns along the *b*-axis direction, with a set of π - π and C—H... π interactions indicated in red and blue, respectively. [Symmetry codes: (i) $-x+1, -y, -z+1$; (iii) $x, y+1, z$; (v) $-x+1, -y+1, -z+1$; (vi) $x, y-1, z$.]

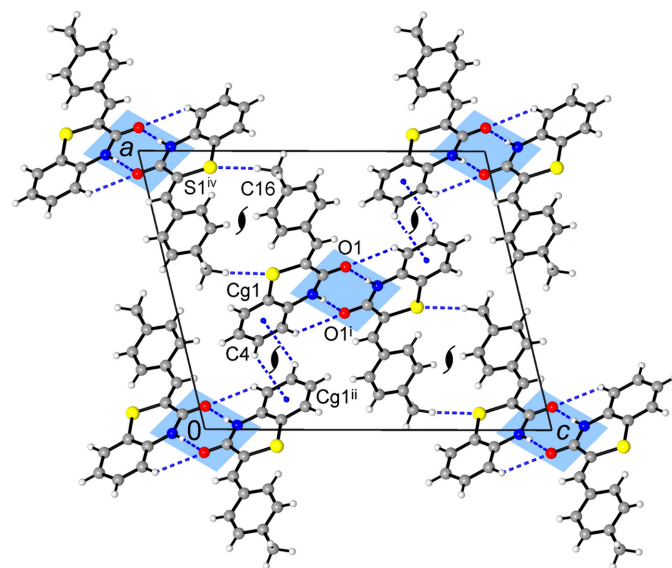


Figure 4
Projection of the structure **1** on the *ac* plane, showing mutual C—H... π bonding between columns of stacked dimers (which are orthogonal to the drawing plane) and weak interlayer C—H...S interactions. Both kinds of bonds generate helical motifs identified here by orthogonal 2₁ axes. [Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iv) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.]

$-z + 1]$ and π - π interactions of ethylene fragments and outer tolyl rings with a shortest contact $C8 \cdots C11^{vi} = 3.447(3) \text{ \AA}$ and a $Cg3 \cdots Cg2^{vi}$ distance of $3.680(2) \text{ \AA}$ [$Cg2$ and $Cg3$ are the centroids of the $C10$ - $C15$ and $C8$ / $C9$ groups, respectively; symmetry code (vi) $x, y - 1, z]$. In addition, two tolyl groups of translationally related molecules afford very weak, but directional $C-H \cdots \pi$ bonds [$C16 \cdots Cg2^{iii} = 3.813(3) \text{ \AA}$, $C-H \cdots Cg2^{iii} = 158(2)^\circ$; symmetry code: (iii) $x, y + 1, z]$. Similar in nature $C-H \cdots \pi$ interactions between carbo rings of the benzothiazinone moieties [$C4 \cdots Cg(C1-C6) = 3.687(3) \text{ \AA}$; Table 1] generate 2_1 -helices along the b -axis direction, which connect the above columns into the layers parallel to the $(10\bar{1})$ plane (Fig. 4).

The supramolecular morphology of **2** is very comparable, with some variations conditioned by specific bonding preferences of the furyl ring. Stacking of the dimers yields similar columns, propagating down the b -axis direction (Fig. 5). Instead of the $C=O/C=O$ interactions seen in **1**, the molecules afford inversion thiazinone stacks, with $O1 \cdots Cg(S1/N1/C1/C6-C8)^{vii}$ and $O1 \cdots \text{plane}^{vii}$ distances of $3.2937(19)$ and $3.226(2) \text{ \AA}$, respectively [symmetry code: (vii) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + \frac{1}{2}$]. That the $O1$ atoms are mutually situated above

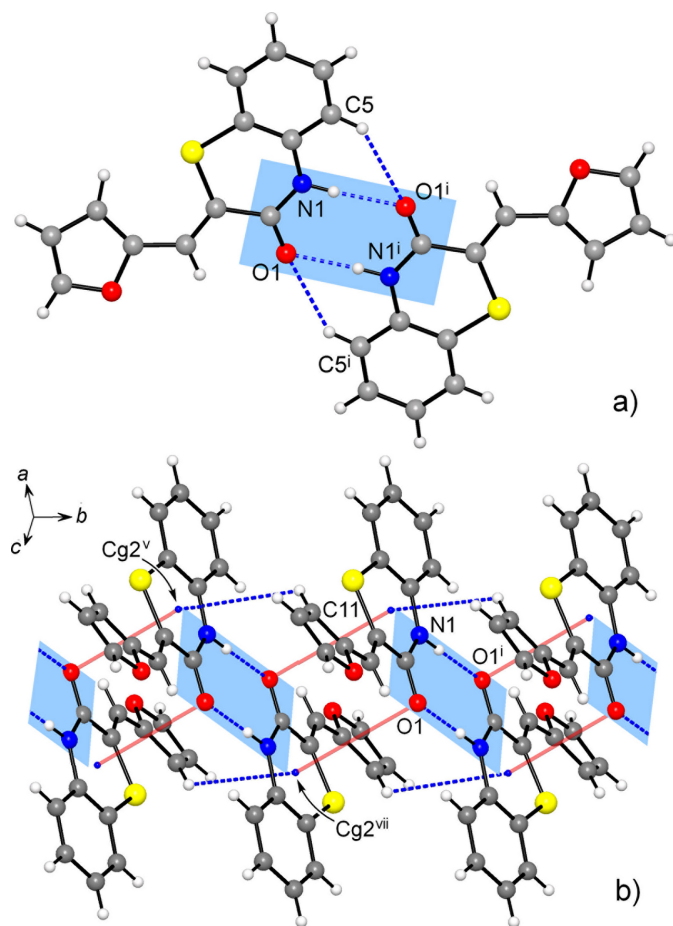


Figure 5
(a) Inversion-related hydrogen-bonded dimers in the structure of **2**; (b) Columns of stacked dimers, which feature importance of axial interactions at the thiazinone core (red: $C=O \cdots \pi$; blue: $C-H \cdots \pi$). [Symmetry codes: (i) $-x + \frac{1}{2}, -y + \frac{3}{2}, -z + \frac{1}{2}$; (v) $x, y - 1, z$; (vii) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + \frac{1}{2}$]

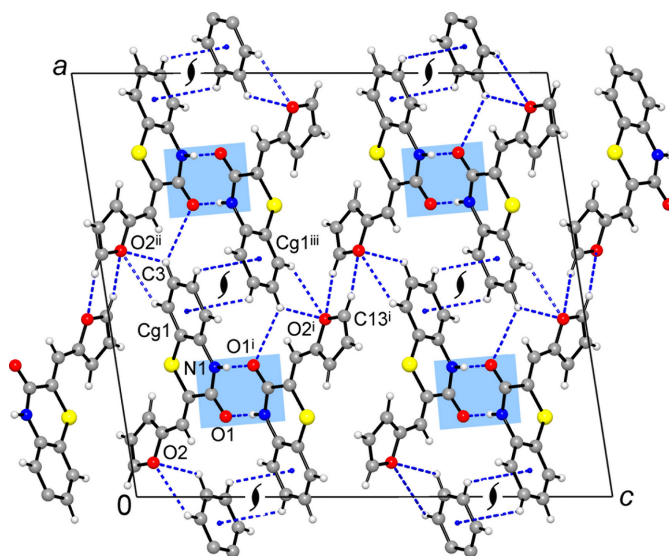


Figure 6
The structure of **2** viewed in a projection on the ac plane, showing layers of stacked dimers (orthogonal to the drawing plane) and their connection through mutual $C-H \cdots O$ bonding of furyl groups. The orthogonal 2_1 axes identify helicate configuration of $C-H \cdots \pi$ bonding motif. [Symmetry codes: (i) $-x + \frac{1}{2}, -y + \frac{3}{2}, -z + \frac{1}{2}$; (ii) $x + \frac{1}{2}, -y, z$; (iii) $x + \frac{1}{2}, -y + 1, z$.]

the adjacent ring centroids is witnessed by the angle between the $O1 \cdots Cg^{vii}$ axis and the ring normal of $11.7(2)^\circ$. This interaction is accompanied by $C-H \cdots \pi$ bonding between the furyl donors and thiazinone acceptors (Fig. 5, Table 2). Similarly to **1**, the columns are linked into layers (parallel to the ab plane) due to $C-H \cdots \pi$ bonds between carbo rings of the benzothiazinone moieties [$C4 \cdots Cg(C1-C6) = 3.688(3) \text{ \AA}$], while two additional $C-H \cdots O$ bonds occur also with the furyl-O acceptor from a second part of the dimer [$C \cdots O = 3.420(3), 3.446(3) \text{ \AA}$; Table 2]. However, the most notable structural function inherent to the furyl rings is the mutual $C-H \cdots O$ bonding, giving inversion dimers with $C \cdots O = 3.359(3) \text{ \AA}$ (Fig. 6). This cyclic pattern itself represents the lowest energy furan dimer calculated for the gas phase (Majerz, 2018). Such interactions provide the connection of the layers into a three-dimensional framework and are particularly essential for structural cohesion as the shortest of the weak hydrogen bonds present with CH donors. One can suppose that a set of supramolecular interactions involving furyl groups may be primarily responsible for the relatively high packing index of 73.4 [vs. 71.5 for **1**], which approaches the upper limit of the 65–75% range expected for organic solids (Dunitz, 1995).

4. Hirshfeld analysis

The supramolecular interactions in the title structures were further assessed by Hirshfeld surface analysis (McKinnon *et al.*, 2007; Hirshfeld, 1977; Spackman *et al.*, 2021) performed with *CrystalExplorer17* (Turner *et al.*, 2017). The two-dimensional fingerprint plots suggest the dominant role of interactions with the H atoms, which account for 71.7 and 65.2% of

Table 3Calculated interaction energies (kJ mol⁻¹).

Interaction energies were calculated employing the CE-B3LYP/6-31G(d,p) functional/basis set combination. The scale factors used to determine E_{tot} : $k_{\text{ele}} = 1.057$, $k_{\text{pol}} = 0.740$, $k_{\text{dis}} = 0.871$, and $k_{\text{rep}} = 0.618$ (Mackenzie *et al.*, 2017). R is the distance between the centroids of the interacting molecules.

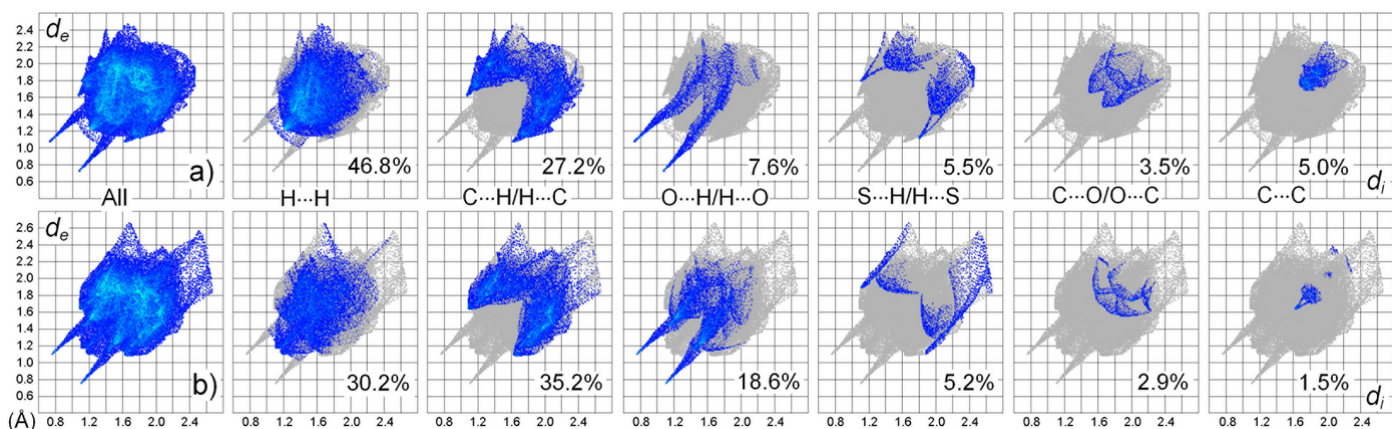
Path	Symmetry code	Type ^a	R (Å)	E_{ele}	E_{pol}	E_{dis}	E_{rep}	E_{tot}
Compound 1								
$A \cdots B$	$-x + 1, -y, -z + 1$	double N—H $\cdots$ O and C—H $\cdots$ O	8.68	-98.2	-24.6	-21.1	108.7	-73.3
$A \cdots C$	$-x + 1, -y + 1, -z + 1$	carbonyl stacking	6.33	-3.8	-3.5	-25.1	10.0	-22.3
$B' \cdots C$	$x, y - 1, z$	π - π , C—H $\cdots$ π , dispersion	5.19	-8.6	-2.4	-62.0	41.3	-39.4
$B \cdots E$	$-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$	C—H $\cdots$ S, dispersion	6.86	-3.5	-0.6	-21.3	15.5	-13.1
$C \cdots D$	$-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$	C—H $\cdots$ π , dispersion	10.31	-4.0	-0.8	-17.2	14.6	-10.8
$C \cdots E$	$-x + \frac{3}{2}, y + \frac{5}{2}, -z + \frac{1}{2}$	dispersion	10.05	-2.9	-0.4	-13.3	7.7	-10.2
Compound 2								
$A \cdots B$	$-x + \frac{1}{2}, -y + \frac{3}{2}, -z + \frac{1}{2}$	double N—H $\cdots$ O and C—H $\cdots$ O	7.90	-89.9	-22.3	-19.8	88.9	-73.9
$A \cdots C$	$-x, -y, -z$	double C—H $\cdots$ O	11.27	-8.4	-1.1	-9.2	7.9	-12.7
$A \cdots D$	$-x + \frac{1}{2}, y, -z$	dispersion	5.26	-2.1	-1.3	-31.2	20.8	-17.5
$A \cdots E$	$-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$	C—H $\cdots$ π , dispersion	9.60	-3.1	-0.9	-18.0	14.8	-10.5
$A \cdots F$	$-x + \frac{1}{2}, -y + \frac{1}{2}, -z + \frac{1}{2}$	C—H $\cdots$ π , dispersion	5.43	-6.6	-1.9	-41.0	30.4	-25.3
$B \cdots E$	$x - \frac{1}{2}, -y + 1, z$	C—H $\cdots$ O	9.93	-4.5	-1.4	-11.9	7.8	-11.3
$B \cdots F$	$x, y + 1, z$	stacking	5.44	-3.1	-3.7	-28.9	12.3	-23.6
$C \cdots D$	$x - \frac{1}{2}, -y, z$	C—H $\cdots$ O, dispersion	9.76	-4.3	-0.7	-13.5	7.9	-12.0

Note: (a) For details of the interaction modes, see Figs. 8 and 9.

the contacts in **1** and **2**, respectively. At the same time, there are essential differences due to the replacement of tolyl for furyl groups. Thus, the fractions of C $\cdots$ H/H $\cdots$ C and O $\cdots$ H/H $\cdots$ O are expanded from 27.2 and 7.6% in **1** to 35.2 and 18.6% in **2**, primarily at the expense of H $\cdots$ H contacts (Fig. 7). Although this is in line with a larger number of the available O-atom acceptors in the latter case, the ability of the furyl group to maintain multiple weak C—H $\cdots$ O interactions is also important. The contributions of S $\cdots$ H/H $\cdots$ S are nearly the same for both compounds and are relatively minor. However, in the case of **1**, one can identify a pair of short spikes pointing to the lower left with $d_e + d_i = 2.95$ Å. These features are similar in nature to the short spikes for C $\cdots$ H/H $\cdots$ C contacts and they likely indicate very weak C—H $\cdots$ S hydrogen bonding. For **2**, the S $\cdots$ H/H $\cdots$ S plot represents rather a collection of points at large d_e and d_i distances and moreover, a scarcely populated extended area above $d_e + d_i = 4.0$ Å suggests the existence of small voids around the S atoms. This observation is supported by the volumes of the Dirichlet–

Voronoi domains associated with the S1 atoms, which are 48.05 Å³ for **1** and 61.41 Å³ for **2** and therefore the S1 environment in the latter case is less crowded. Finally, an overlap between nearly parallel molecules, due to the slipped π - π ethene/tolyl stacking, is clearly indicated by the C $\cdots$ C plots for **1** (5.0%), in the form of the blue area centred at *ca.* $d_e = d_i = 1.80$ Å and with a shortest contact of 3.50 Å (Fig. 7). This feature is only minor in the case of **2**, with a small fraction of slightly shorter C $\cdots$ C contacts (1.5%) associated with carbonyl/thiazine stacking.

The intermolecular interaction energies were calculated using the CE B3LYP/6 31G(d,p) energy model in *Crystal-Explorer17* (Turner *et al.*, 2017). With a cut-off of $|E_{\text{tot}}| > 10$ kJ mol⁻¹, five symmetry-independent paths were considered for the closest environment of the molecules in **1** (Table 3) and the far dominant energy of $E_{\text{tot}} = -73.3$ kJ mol⁻¹ corresponds to the reciprocal N—H $\cdots$ O and C—H $\cdots$ O interactions within the basic dimer (path $A \cdots B$, Fig. 8). This pairing is governed essentially by the electrostatic

**Figure 7**

Two-dimensional fingerprint plots for **1** (a) and **2** (b) for all contacts and delineated into the principal contributions of individual contacts. Other contributions, which account for more than 1.0%, are N $\cdots$ H/H $\cdots$ N (2.0 and 1.8% for **1** and **2**, respectively) and S $\cdots$ C/C $\cdots$ S (1.8 and 2.8%).

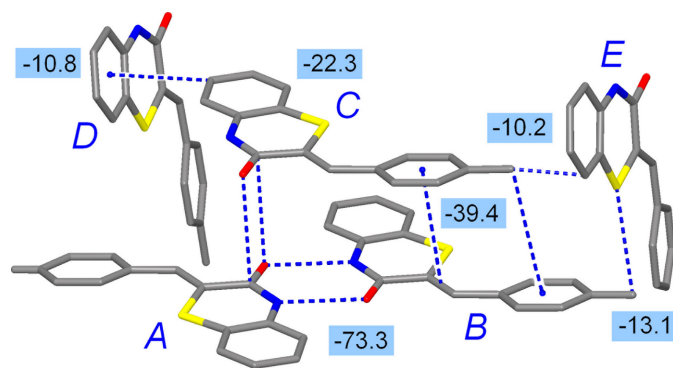


Figure 8
The principal pairwise intermolecular interactions for **1**, identified with a cut-off limit of 10 kJ mol⁻¹. The interaction energies are given in kJ mol⁻¹.

component ($E_{\text{ele}} = -98.2$ kJ mol⁻¹) and is very close in energy to interactions in 2-pyridone dimers [-68.2 kJ mol⁻¹; Inuzuka & Fujimoto, 1982]. Other structure-defining interactions originate in London dispersion, while the most prominent ones are also restricted to the columns of stacked dimers. First, the $B \cdots C$ pair combines tolyl $C-H \cdots \pi$ bonds and $\pi-\pi$ stacking within a very large interaction area. The appreciable resulting energy of -39.4 kJ mol⁻¹ is a reflection of a significant dispersion contributor [$E_{\text{dis}} = -62.0$ kJ mol⁻¹]. Second, mutual antiparallel stacks of the carbonyl groups (path $A \cdots C$) are also very favorable with $E_{\text{tot}} = -22.3$ kJ mol⁻¹, which exactly coincides with the value for model 2-propanone dimers (Allen *et al.*, 1998).

The landscape of interaction energies for **2** is apparently more rich, with nine unique paths above $|E_{\text{tot}}| > 10$ kJ mol⁻¹ (Fig. 9). In fact, beyond the primary interaction in the form of electrostatically dominated strong hydrogen bonding (pair $A \cdots B$, $E_{\text{tot}} = -73.9$ kJ mol⁻¹), most intermolecular paths converge in the interaction energies falling into the -10 to -25 kJ mol⁻¹ range (Table 3). The most prominent interactions within this group are mutual carbonyl- π stacking of path $A \cdots F$ and dispersion and $C-H \cdots \pi$ driven path $B \cdots F$ [$E_{\text{tot}} = -25.3$ and -23.6 kJ mol⁻¹, respectively]. Both of them are also found within the column of stacked dimers. At the same time, the growing importance of bonding between the subconnectivities of lower dimensionality is best illustrated by the energetics of the furyl dimers established between the layers. The interaction within the path $A \cdots C$ has comparable electrostatic and dispersion contributors and it results in $E_{\text{tot}} = -12.7$ kJ mol⁻¹, which is superior to most pairwise interactions between the layers in the structure of **1**. The latter value reproduces an energy of -13.0 kJ mol⁻¹, found for the doubly $C-H \cdots O$ -bonded furan dimer in the gas phase (Majerz, 2018). One can suppose that the specific behavior of furyl groups in **2**, either as a donor or acceptor of weak $C-H \cdots O$ bonding (as may be compared with tolyl groups in **1**) contributes not only to the larger fraction of $O \cdots H/H \cdots O$ contacts, but also enhances the interaction energies. Even in spite of the possible presence of small crystal voids around the S1 atoms in **2**, the furyl derivative develops a perceptibly higher packing index.

5. Database survey

A search of the Cambridge Structural Database (CSD, updated to July 2025; Groom *et al.*, 2016) for 1,4-benzothiazin-3-one derivatives bearing a substituted methyldene fragment at the C²-atom and with no substitution at benzo-ring C atoms revealed 18 hits. The group of closest 4-*H* analogs is represented by (Z)-2-(1-bromoethylidene) (CSD refcode BOLDOV; Bates *et al.*, 1982) and (Z)-2-(nitromethylene) compounds (GETNOJ; Berestovskaya *et al.*, 2006) and two polymorphs of structurally related thiazine-indigo (SAJMOH and SAJMOH01; Buchsbaum *et al.*, 2011), whereas the larger family of 4-*R* derivatives features the incorporation of 2-benzylidene and derived fragments, including one example of a 4-methylbenzylidene compound related to the structure of **1** (RURBEO; Sebbar *et al.*, 2020a). All these compounds follow the trend established above: the 1,4-thiazin-3-one core is essentially flat in the case of 4-*H* species, but even 4-methyl substitution (VUXWES; Ellouz *et al.*, 2015) causes loss of planarity. An appreciable bend of the heteroring may be assessed with values of dihedral angles between the S¹C⁹C¹⁰

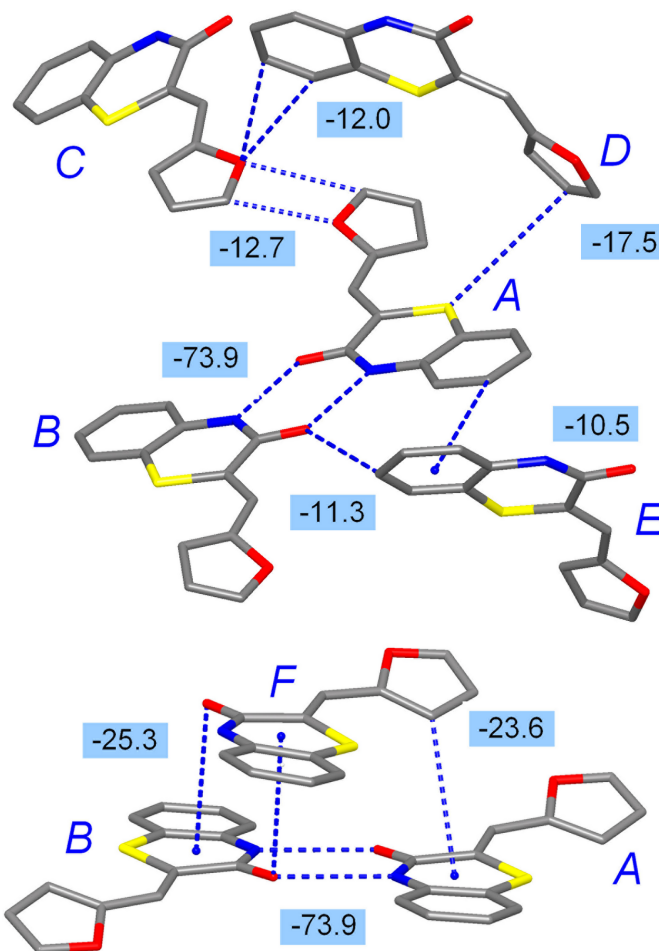


Figure 9
The principal pathways of intermolecular interactions for **2**, identified with a cut-off limit of 10 kJ mol⁻¹, which involve different kinds of stacking and hydrogen bonding. The interaction energies are given in kJ mol⁻¹.

Table 4
Experimental details.

	1	2
Crystal data		
Chemical formula	C ₁₆ H ₁₃ NOS	C ₁₃ H ₉ NO ₂ S
<i>M_r</i>	267.33	243.27
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>n</i>	Monoclinic, <i>I</i> 2/ <i>a</i>
Temperature (K)	150	150
<i>a</i> , <i>b</i> , <i>c</i> (Å)	14.418 (3), 5.1901 (9), 17.425 (3)	18.9170 (5), 5.4280 (2), 20.9874 (7)
β (°)	103.541 (2)	98.702 (1)
<i>V</i> (Å ³)	1267.7 (4)	2130.21 (12)
<i>Z</i>	4	8
Radiation type	Mo <i>K</i> α	Cu <i>K</i> α
μ (mm ^{−1})	0.25	2.60
Crystal size (mm)	0.31 × 0.21 × 0.03	0.13 × 0.05 × 0.02
Data collection		
Diffractometer	Bruker SMART APEX CCD	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T</i> _{min} , <i>T</i> _{max}	0.83, 0.99	0.84, 0.95
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	11287, 3106, 2192	11794, 1826, 1506
<i>R</i> _{int}	0.048	0.069
(sin θ/λ) _{max} (Å ^{−1})	0.667	0.589
Refinement		
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.051, 0.131, 1.04	0.038, 0.090, 1.10
No. of reflections	3106	1826
No. of parameters	224	190
H-atom treatment	All H-atom parameters refined	All H-atom parameters refined
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ^{−3})	0.53, −0.37	0.21, −0.29

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXS97* (Sheldrick, 2008), *SHELXL2019/3* (Sheldrick, 2015), *DIAMOND* (Brandenburg, 1999) and *WinGX* (Farrugia, 2012).

and N⁴C³OC² planes, which are nearly uniform for all 4-*R* compounds within the range 19.2–24.3°. They are systematically much larger than the parameters for 4-*H* species: 5.70 (BOLDOV); 5.82 (GETNOJ); 1.71 (SAJMOH) and 1.51° (SAJMOH01). From a supramolecular perspective, the comparable examples are restricted to the category of N-species and every such 1,4-benzothiazin-3-one sustains dimeric motifs of reciprocal N—H···O interactions, which are similar to those in the title structures.

6. Synthesis and crystallization

To 300 mg (2.84 mmol) of 3,4-dihydro-2*H*-1,4-benzothiazin-3-one and 5.68 mmol of either 4-methylbenzaldehyde (for the synthesis of **1**) or furan-2-carbaldehyde (for the synthesis of **2**) dissolved in 10 ml of anhydrous DMF, 383.4 mg (7.1 mmol) of sodium methoxide were added. The mixture was refluxed for 18 h while being stirred vigorously with a magnetic stirrer. After cooling, the precipitate was filtered out and the filtrate was concentrated under reduced pressure. The resulting crude residue was purified by column chromatography on silica gel using ethyl acetate/hexane (10:90, *v/v*) as eluent. Slow evaporation of the collected fractions afforded the pure products: (*Z*)-2-(4-methylbenzylidene)-2*H*-benzo[*b*][1,4]thiazin-3(4*H*)-one (**1**), obtained as colorless plate-like crystals in 85% yield or 2-(furfurylidene)-3,4-dihydro-2*H*-1,4-benzothiazin-3-one (**2**), obtained as colorless column-like crystals in 79% yield. ¹H NMR (300 MHz, DMSO-*d*₆), δ , ppm for **1**: 2.40 (s, 3H, CH₃), 7.02–7.63 (*m*, 8H, Ar–H), 7.80 (s, 1H, ethene

CH), 11.00 (*s*, 1H, NH). ¹H NMR (300 MHz, DMSO-*d*₆), δ , ppm For **2**: 7.63–6.77 (*m*, 7H, Ar–H), 7.98 (*s*, 1H, ethene CH), 11.03 (*s*, 1H, NH).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. All hydrogen atoms were located and then freely refined with isotropic displacement parameters, which results in N—H = 0.87 (3) and 0.89 (3); C—H = 0.90 (3)–0.99 (3) and C—H (CH₃) = 0.94 (4)–0.98 (3) Å.

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References

- Allen, F. H., Baalham, C. A., Lommerse, J. P. M. & Raithby, P. R. (1998). *Acta Cryst.* **B54**, 320–329.
- Arman, H. D., Poplaukhin, P. & Tiekinck, E. R. T. (2009). *Acta Cryst.* **E65**, o3187.

- Armenise, D., Muraglia, M., Florio, M. A., Laurentis, N. D., Rosato, A., Carrieri, A., Corbo, F. & Franchini, C. (2012). *Mol. Pharmacol.* **50**, 1178–1188.
- Bates, R. B., Duguay, L. M., Klenck, R. E., Kriek, G. R., Tempesta, M. S. & Brewer, A. D. (1982). *J. Heterocycl. Chem.* **19**, 927–928.
- Berestovskaya, V. M., Tafeenko, V. A., Makarenko, S. V., Sadikov, K. D. & Chernyshev, I. V. (2006). *Zh. Obshch. Khim.* **76**, 137–142.
- Brandenburg, K. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Bruker (2016). *APEX3* and *SAINT*. Bruker AXS, Madison, Wisconsin, USA.
- Buchsbau, C., Paulus, E. F. & Schmidt, M. U. (2011). *Z. Kristallogr.* **226**, 822–831.
- Chen, X., Kobiro, K., Asahara, H., Kakiuchi, K., Sugimoto, R., Saigo, K. & Nishiwaki, N. (2013). *Tetrahedron* **69**, 4624–4630.
- Dunitz, J. D. (1995). *Helv. Chim. Acta* 106–111.
- Ellouz, M., Sebbar, N. K., Boulhaoua, M., Essassi, E. M. & Mague, J. T. (2017a). *IUCr Data* **2**, x170646.
- Ellouz, M., Sebbar, N. K., Essassi, E. M., Saadi, M. & El Ammari, L. (2015). *Acta Cryst. E* **71**, o862–o863.
- Ellouz, M., Sebbar, N. K., Fichtali, I., Ouzidan, Y., Mennane, Z., Charof, R., Mague, J. T., Urrutigoity, M. & Essassi, E. M. (2018). *Chem. Cent. J.* **12**, 123.
- Ellouz, M., Sebbar, N. K., Ouzidan, Y., Essassi, E. M. & Mague, J. T. (2017b). *IUCr Data* **2**, x170097.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Gowda, J., Khader, A. M. A., Kalluraya, B., Shree, P. & Shabaraya, A. R. (2011). *Eur. J. Med. Chem.* **46**, 4100–4106.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst. B* **72**, 171–179.
- Gupta, R. R., Kumar, R. & Gautam, R. K. (1985). *J. Fluor. Chem.* **28**, 381–385.
- Gupta, V. & Gupta, R. R. (1991). *J. Prakt. Chem.* **333**, 153–156.
- Haji Ali, S., Osmaniye, D., Sağlık, B. N., Levent, S., Özkay, Y. & Kaplancıklı, Z. A. (2022). *Molecules* **27**, 2121.
- Hirshfeld, H. L. (1977). *Theor. Chim. Acta* **44**, 129–138.
- Hni, B., Sebbar, N. K., Hökelek, T., El Ghayati, L., Bouzian, Y., Mague, J. T. & Essassi, E. M. (2019b). *Acta Cryst. E* **75**, 593–599.
- Hni, B., Sebbar, N. K., Hökelek, T., Ouzidan, Y., Moussaif, A., Mague, J. T. & Essassi, E. M. (2019a). *Acta Cryst. E* **75**, 372–377.
- Inuzuka, K. & Fujimoto, A. (1982). *Bull. Chem. Soc. Jpn* **55**, 2537–2540.
- Irrou, E., Ait Elmachkouri, Y., Mazzah, A., Hökelek, T., Haoudi, A., Mague, J. T., Taha, M. L. & Sebbar, N. K. (2023). *Acta Cryst. E* **79**, 1037–1043.
- Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
- Mackenzie, C. F., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *IUCrJ* **4**, 575–587.
- Mague, J. T. & Ouzidan, Y. (2024). *CSD Communication* (CCDC 2396918). CCDC, Cambridge, England. <https://doi.org/10.5517/ccdc.csd.cc2lg5y6>
- Majerz, I. (2018). *J. Comput. Aided Mol. Des.* **32**, 1247–1258.
- McKinnon, J. J., Jayatilaka, D. & Spackman, M. A. (2007). *Chem. Commun.* pp. 3814–3816.
- McMahon, J. A., Bis, J. A., Vishweshwar, P., Shattock, T. R., McLaughlin, O. L. & Zaworotko, M. J. (2005). *Z. Kristallogr.* **220**, 340–350.
- Ramasubbu, N., Parthasarathy, R. & Tsoucaris, G. (1988). *Acta Cryst. C* **44**, 2016–2018.
- Sebbar, G., Hni, B., Hökelek, T., Mague, J. T., Sebbar, N. K., Belkadi, B. & Essassi, E. M. (2020b). *Acta Cryst. E* **76**, 889–895.
- Sebbar, G., Mohamed, E., Hökelek, T., Mague, J. T., Sebbar, N. K., Essassi, E. M. & Belkadi, B. (2020a). *Acta Cryst. E* **76**, 629–636.
- Sebbar, N. K., Ellouz, M., Essassi, E. M., Ouzidan, Y. & Mague, J. T. (2015). *Acta Cryst. E* **71**, o999.
- Sebbar, N. K., Ellouz, M., Essassi, E. M., Saadi, M. & El Ammari, L. (2016b). *IUCr Data* **1**, x161012.
- Sebbar, N. K., Mekhzoum, M. E. M., Essassi, E. M., Zerzouf, A., Talbaoui, A., Bakri, Y., Saadi, M. & Ammari, L. E. (2016a). *Res. Chem. Intermed.* **42**, 6845–6862.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst. C* **71**, 3–8.
- Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.
- Tawada, H., Sugiyama, Y., Ikeda, H., Yamamoto, Y. & Meguro, K. (1990). *Chem. Pharm. Bull.* **38**, 1238–1245.
- Trapani, G., Reho, A., Morlacchi, F., Latrofa, A., Marchini, P., Venturi, F. & Cantalamessa, F. (1985). *Farmaco Ed. Sci.* **40**, 369–376.
- Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *Crystal Explorer 17*. The University of Western Australia.
- Warren, B. K. & Knaus, E. E. (1987). *Eur. J. Med. Chem.* **22**, 411–415.
- Zia-ur-Rehman, M., Choudary, J. A., Elsegood, M. R. J., Siddiqui, H. L. & Khan, K. M. (2009). *Eur. J. Med. Chem.* **44**, 1311–1316.

supporting information

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Crystal structures, Hirshfeld surface analysis and interaction energies of (Z)-2-(4-methylbenzylidene)- and (Z)-2-(furfurylidene)-2*H*-benzo[*b*][1,4]thiazin-3(4*H*)-one

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Computing details

(Z)-2-(4-Methylbenzylidene)-2*H*-benzo[*b*][1,4]thiazin-3(4*H*)-one (1)

Crystal data

C₁₆H₁₃NOS

$M_r = 267.33$

Monoclinic, $P2_1/n$

$a = 14.418$ (3) Å

$b = 5.1901$ (9) Å

$c = 17.425$ (3) Å

$\beta = 103.541$ (2)°

$V = 1267.7$ (4) Å³

$Z = 4$

$F(000) = 560$

$D_x = 1.401$ Mg m⁻³

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

Cell parameters from 2851 reflections

$\theta = 2.4$ – 27.0°

$\mu = 0.25$ mm⁻¹

$T = 150$ K

Plate, colourless

$0.31 \times 0.21 \times 0.03$ mm

Data collection

Bruker SMART APEX CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.3333 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Krause *et al.*, 2015)

$T_{\min} = 0.83$, $T_{\max} = 0.99$

11287 measured reflections

3106 independent reflections

2192 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.048$

$\theta_{\max} = 28.3^\circ$, $\theta_{\min} = 1.7^\circ$

$h = -19 \rightarrow 19$

$k = -6 \rightarrow 6$

$l = -23 \rightarrow 22$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.051$

$wR(F^2) = 0.131$

$S = 1.04$

3106 reflections

224 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 0.425P]$

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

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

$\Delta\rho_{\max} = 0.53$ e Å⁻³

$\Delta\rho_{\min} = -0.37$ e Å⁻³

Special details

Experimental. The diffraction data were collected in three sets of 363 frames (0.5° width in ω) at $\varphi = 0, 120$ and 240° . A scan time of 80 sec/frame was used.

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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.55852 (4)	0.55290 (11)	0.30333 (3)	0.03071 (18)
O1	0.58296 (11)	0.2623 (3)	0.51760 (8)	0.0307 (4)
N1	0.48480 (13)	0.1456 (4)	0.40334 (10)	0.0268 (4)
C1	0.46507 (14)	0.3340 (4)	0.27280 (12)	0.0236 (4)
C2	0.41636 (16)	0.3409 (4)	0.19356 (12)	0.0276 (5)
C3	0.34365 (16)	0.1682 (4)	0.16488 (13)	0.0310 (5)
C4	0.31880 (16)	−0.0129 (4)	0.21486 (14)	0.0308 (5)
C5	0.36606 (15)	−0.0184 (4)	0.29363 (13)	0.0284 (5)
C6	0.43919 (14)	0.1572 (4)	0.32306 (11)	0.0233 (4)
C7	0.55369 (15)	0.2991 (4)	0.44557 (12)	0.0241 (4)
C8	0.59591 (15)	0.5073 (4)	0.40564 (12)	0.0242 (4)
C9	0.66788 (15)	0.6442 (4)	0.45071 (12)	0.0253 (4)
C10	0.72877 (14)	0.8460 (4)	0.43109 (12)	0.0246 (4)
C11	0.71411 (16)	0.9745 (4)	0.35848 (13)	0.0277 (5)
C12	0.77792 (16)	1.1582 (4)	0.34453 (13)	0.0298 (5)
C13	0.85867 (16)	1.2238 (4)	0.40155 (12)	0.0290 (5)
C14	0.87274 (17)	1.1000 (5)	0.47383 (13)	0.0336 (5)
C15	0.80970 (16)	0.9165 (5)	0.48863 (13)	0.0314 (5)
C16	0.9272 (2)	1.4253 (5)	0.38564 (15)	0.0366 (6)
H1N	0.466 (2)	0.019 (5)	0.4291 (17)	0.056 (9)*
H2	0.4369 (16)	0.468 (4)	0.1593 (14)	0.030 (6)*
H3	0.3106 (17)	0.170 (4)	0.1096 (14)	0.036 (6)*
H4	0.2677 (15)	−0.136 (4)	0.1952 (12)	0.023 (5)*
H5	0.3464 (15)	−0.137 (4)	0.3269 (13)	0.026 (6)*
H9	0.6836 (16)	0.600 (4)	0.5040 (14)	0.028 (6)*
H11	0.6590 (17)	0.940 (4)	0.3189 (14)	0.034 (6)*
H12	0.7653 (16)	1.240 (4)	0.2961 (14)	0.032 (6)*
H14	0.9262 (19)	1.134 (5)	0.5113 (15)	0.043 (7)*
H15	0.8185 (17)	0.837 (5)	0.5372 (15)	0.037 (7)*
H16A	0.989 (3)	1.390 (7)	0.417 (2)	0.097 (13)*
H16B	0.909 (3)	1.603 (8)	0.389 (2)	0.102 (13)*
H16C	0.937 (2)	1.410 (6)	0.3321 (19)	0.066 (9)*

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0319 (3)	0.0350 (3)	0.0211 (3)	−0.0111 (2)	−0.0021 (2)	0.0073 (2)
O1	0.0343 (9)	0.0342 (8)	0.0217 (7)	−0.0073 (7)	0.0025 (6)	0.0067 (6)
N1	0.0283 (9)	0.0288 (10)	0.0216 (9)	−0.0042 (8)	0.0022 (7)	0.0072 (7)
C1	0.0225 (10)	0.0229 (10)	0.0240 (10)	−0.0004 (8)	0.0027 (8)	0.0010 (8)
C2	0.0301 (11)	0.0287 (11)	0.0224 (10)	0.0000 (9)	0.0031 (9)	0.0021 (8)
C3	0.0316 (12)	0.0340 (12)	0.0241 (11)	−0.0007 (10)	0.0002 (9)	−0.0029 (9)
C4	0.0274 (12)	0.0280 (12)	0.0343 (12)	−0.0031 (9)	0.0019 (10)	−0.0025 (9)
C5	0.0261 (11)	0.0272 (11)	0.0312 (11)	−0.0017 (9)	0.0052 (9)	0.0034 (9)
C6	0.0217 (10)	0.0253 (10)	0.0220 (10)	0.0025 (8)	0.0034 (8)	0.0024 (8)
C7	0.0244 (10)	0.0241 (10)	0.0235 (10)	−0.0004 (8)	0.0048 (8)	0.0037 (8)
C8	0.0247 (10)	0.0255 (11)	0.0219 (10)	0.0019 (8)	0.0046 (8)	0.0042 (8)
C9	0.0273 (11)	0.0271 (11)	0.0206 (10)	0.0001 (9)	0.0041 (8)	0.0030 (8)
C10	0.0249 (10)	0.0260 (11)	0.0232 (10)	−0.0022 (9)	0.0063 (8)	−0.0027 (8)
C11	0.0252 (11)	0.0318 (12)	0.0240 (10)	−0.0045 (9)	0.0019 (9)	−0.0010 (9)
C12	0.0353 (12)	0.0298 (11)	0.0246 (11)	−0.0038 (10)	0.0074 (9)	0.0009 (9)
C13	0.0331 (12)	0.0275 (11)	0.0288 (11)	−0.0053 (9)	0.0121 (10)	−0.0082 (9)
C14	0.0332 (13)	0.0411 (13)	0.0237 (11)	−0.0112 (10)	0.0011 (10)	−0.0072 (9)
C15	0.0362 (13)	0.0374 (13)	0.0188 (10)	−0.0068 (10)	0.0032 (9)	−0.0009 (9)
C16	0.0410 (15)	0.0359 (14)	0.0360 (13)	−0.0163 (12)	0.0150 (12)	−0.0100 (11)

Geometric parameters ($\text{\AA}$, $^\circ$)

S1—C1	1.747 (2)	C8—C9	1.348 (3)
S1—C8	1.754 (2)	C9—C10	1.458 (3)
O1—C7	1.242 (2)	C9—H9	0.93 (2)
N1—C7	1.350 (3)	C10—C15	1.397 (3)
N1—C6	1.401 (2)	C10—C11	1.402 (3)
N1—H1N	0.87 (3)	C11—C12	1.385 (3)
C1—C6	1.379 (3)	C11—H11	0.94 (2)
C1—C2	1.395 (3)	C12—C13	1.384 (3)
C2—C3	1.381 (3)	C12—H12	0.92 (2)
C2—H2	0.98 (2)	C13—C14	1.386 (3)
C3—C4	1.384 (3)	C13—C16	1.508 (3)
C3—H3	0.97 (2)	C14—C15	1.382 (3)
C4—C5	1.382 (3)	C14—H14	0.90 (3)
C4—H4	0.98 (2)	C15—H15	0.92 (2)
C5—C6	1.397 (3)	C16—H16A	0.94 (4)
C5—H5	0.93 (2)	C16—H16B	0.96 (4)
C7—C8	1.490 (3)	C16—H16C	0.98 (3)
C1—S1—C8	104.68 (10)	C8—C9—C10	131.7 (2)
C7—N1—C6	129.24 (19)	C8—C9—H9	115.3 (14)
C7—N1—H1N	116.1 (19)	C10—C9—H9	112.9 (14)
C6—N1—H1N	114.7 (19)	C15—C10—C11	116.56 (19)
C6—C1—C2	119.82 (19)	C15—C10—C9	117.86 (19)

C6—C1—S1	122.91 (15)	C11—C10—C9	125.57 (19)
C2—C1—S1	117.27 (16)	C12—C11—C10	121.3 (2)
C3—C2—C1	120.3 (2)	C12—C11—H11	119.0 (14)
C3—C2—H2	121.9 (13)	C10—C11—H11	119.7 (14)
C1—C2—H2	117.7 (13)	C13—C12—C11	121.6 (2)
C2—C3—C4	120.0 (2)	C13—C12—H12	119.7 (15)
C2—C3—H3	120.5 (14)	C11—C12—H12	118.7 (15)
C4—C3—H3	119.5 (14)	C12—C13—C14	117.3 (2)
C5—C4—C3	119.9 (2)	C12—C13—C16	121.0 (2)
C5—C4—H4	119.4 (12)	C14—C13—C16	121.7 (2)
C3—C4—H4	120.7 (12)	C15—C14—C13	121.8 (2)
C4—C5—C6	120.3 (2)	C15—C14—H14	119.2 (16)
C4—C5—H5	118.7 (13)	C13—C14—H14	118.9 (17)
C6—C5—H5	120.9 (13)	C14—C15—C10	121.4 (2)
C1—C6—C5	119.61 (18)	C14—C15—H15	121.7 (15)
C1—C6—N1	121.97 (19)	C10—C15—H15	116.9 (15)
C5—C6—N1	118.41 (19)	C13—C16—H16A	109 (2)
O1—C7—N1	119.53 (18)	C13—C16—H16B	117 (2)
O1—C7—C8	120.28 (18)	H16A—C16—H16B	112 (3)
N1—C7—C8	120.17 (18)	C13—C16—H16C	111.5 (18)
C9—C8—C7	116.94 (18)	H16A—C16—H16C	102 (3)
C9—C8—S1	122.19 (16)	H16B—C16—H16C	104 (3)
C7—C8—S1	120.71 (15)		
C8—S1—C1—C6	−5.2 (2)	O1—C7—C8—S1	177.44 (16)
C8—S1—C1—C2	175.45 (17)	N1—C7—C8—S1	−1.1 (3)
C6—C1—C2—C3	−1.3 (3)	C1—S1—C8—C9	−179.98 (18)
S1—C1—C2—C3	178.04 (17)	C1—S1—C8—C7	4.81 (19)
C1—C2—C3—C4	0.0 (3)	C7—C8—C9—C10	175.5 (2)
C2—C3—C4—C5	0.8 (3)	S1—C8—C9—C10	0.1 (4)
C3—C4—C5—C6	−0.3 (3)	C8—C9—C10—C15	−167.7 (2)
C2—C1—C6—C5	1.8 (3)	C8—C9—C10—C11	11.2 (4)
S1—C1—C6—C5	−177.52 (16)	C15—C10—C11—C12	1.2 (3)
C2—C1—C6—N1	−178.91 (19)	C9—C10—C11—C12	−177.8 (2)
S1—C1—C6—N1	1.8 (3)	C10—C11—C12—C13	−0.2 (3)
C4—C5—C6—C1	−1.0 (3)	C11—C12—C13—C14	−0.7 (3)
C4—C5—C6—N1	179.7 (2)	C11—C12—C13—C16	−179.6 (2)
C7—N1—C6—C1	3.7 (3)	C12—C13—C14—C15	0.7 (4)
C7—N1—C6—C5	−177.0 (2)	C16—C13—C14—C15	179.6 (2)
C6—N1—C7—O1	177.5 (2)	C13—C14—C15—C10	0.3 (4)
C6—N1—C7—C8	−4.0 (3)	C11—C10—C15—C14	−1.2 (3)
O1—C7—C8—C9	2.0 (3)	C9—C10—C15—C14	177.8 (2)
N1—C7—C8—C9	−176.53 (19)		

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	0.87 (3)	1.95 (3)	2.822 (2)	177 (3)

C4—H4...Cg(C1—C6) ⁱⁱ	0.98 (2)	2.94 (2)	3.687 (3)	134 (2)
C5—H5...O1 ⁱ	0.93 (2)	2.74 (2)	3.440 (3)	132.4 (16)
C16—H16B...Cg(C10—C15) ⁱⁱⁱ	0.96 (4)	2.90 (4)	3.813 (3)	158 (2)
C16—H16C...S1 ^{iv}	0.98 (3)	3.02 (3)	3.865 (3)	146 (2)

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x+1/2, y-1/2, -z+1/2$; (iii) $x, y+1, z$; (iv) $-x+3/2, y+1/2, -z+1/2$.

(Z)-2-(Furan-2-ylmethylidene)-2H-benzo[b][1,4]thiazin-3(4H)-one (2)

Crystal data

C ₁₃ H ₉ NO ₂ S	$F(000) = 1008$
$M_r = 243.27$	$D_x = 1.517 \text{ Mg m}^{-3}$
Monoclinic, $I2/a$	Cu $K\alpha$ radiation, $\lambda = 1.54178 \text{ \AA}$
$a = 18.9170 (5) \text{ \AA}$	Cell parameters from 7352 reflections
$b = 5.4280 (2) \text{ \AA}$	$\theta = 4.3\text{--}65.2^\circ$
$c = 20.9874 (7) \text{ \AA}$	$\mu = 2.60 \text{ mm}^{-1}$
$\beta = 98.702 (1)^\circ$	$T = 150 \text{ K}$
$V = 2130.21 (12) \text{ \AA}^3$	Column, colourless
$Z = 8$	$0.13 \times 0.05 \times 0.02 \text{ mm}$

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer	$T_{\min} = 0.84, T_{\max} = 0.95$
Radiation source: INCOATEC I μ S micro-focus source	11794 measured reflections
Mirror monochromator	1826 independent reflections
Detector resolution: $10.4167 \text{ pixels mm}^{-1}$	1506 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.069$
Absorption correction: multi-scan (SADABS; Krause <i>et al.</i> , 2015)	$\theta_{\max} = 65.2^\circ, \theta_{\min} = 4.3^\circ$
	$h = -22 \rightarrow 22$
	$k = -6 \rightarrow 6$
	$l = -24 \rightarrow 24$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.038$	All H-atom parameters refined
$wR(F^2) = 0.090$	$w = 1/[\sigma^2(F_o^2) + (0.0382P)^2 + 2.4209P]$
$S = 1.10$	where $P = (F_o^2 + 2F_c^2)/3$
1826 reflections	$(\Delta/\sigma)_{\max} < 0.001$
190 parameters	$\Delta\rho_{\max} = 0.21 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.28 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

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 ($\text{\AA}^2$)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.31212 (3)	0.10632 (11)	0.11631 (3)	0.02437 (19)
O1	0.18959 (8)	0.5139 (3)	0.21259 (8)	0.0278 (4)

O2	0.08078 (8)	−0.0479 (3)	0.04622 (8)	0.0290 (4)
N1	0.30581 (10)	0.5594 (4)	0.20528 (9)	0.0207 (4)
C1	0.38122 (12)	0.3098 (4)	0.14519 (10)	0.0192 (5)
C2	0.44861 (12)	0.2685 (4)	0.12735 (11)	0.0221 (5)
C3	0.50469 (12)	0.4255 (5)	0.14732 (11)	0.0232 (5)
C4	0.49456 (13)	0.6284 (5)	0.18555 (11)	0.0249 (5)
C5	0.42823 (12)	0.6707 (4)	0.20361 (11)	0.0219 (5)
C6	0.37169 (12)	0.5111 (4)	0.18414 (10)	0.0192 (5)
C7	0.24287 (12)	0.4442 (4)	0.18943 (10)	0.0203 (5)
C8	0.23628 (11)	0.2355 (4)	0.14215 (10)	0.0188 (5)
C9	0.17072 (13)	0.1519 (4)	0.11978 (11)	0.0224 (5)
C10	0.15189 (12)	−0.0431 (4)	0.07434 (11)	0.0219 (5)
C11	0.18668 (13)	−0.2345 (4)	0.05112 (11)	0.0243 (5)
C12	0.13564 (14)	−0.3639 (5)	0.00687 (12)	0.0291 (6)
C13	0.07351 (14)	−0.2461 (5)	0.00537 (12)	0.0302 (6)
H1N	0.3056 (15)	0.691 (6)	0.2301 (15)	0.046 (9)*
H2	0.4560 (13)	0.129 (5)	0.1012 (12)	0.030 (7)*
H3	0.5534 (15)	0.393 (5)	0.1376 (13)	0.034 (7)*
H4	0.5335 (14)	0.741 (5)	0.1993 (12)	0.029 (7)*
H5	0.4188 (12)	0.807 (5)	0.2282 (12)	0.020 (6)*
H9	0.1318 (13)	0.231 (5)	0.1329 (11)	0.021 (6)*
H11	0.2378 (15)	−0.266 (5)	0.0648 (13)	0.033 (7)*
H12	0.1460 (16)	−0.504 (6)	−0.0150 (15)	0.046 (8)*
H13	0.0271 (15)	−0.272 (5)	−0.0154 (14)	0.040 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0212 (3)	0.0225 (3)	0.0298 (3)	−0.0008 (2)	0.0052 (2)	−0.0102 (2)
O1	0.0239 (9)	0.0300 (10)	0.0315 (9)	−0.0028 (7)	0.0111 (7)	−0.0116 (7)
O2	0.0231 (9)	0.0302 (10)	0.0321 (9)	−0.0055 (7)	−0.0006 (7)	−0.0064 (7)
N1	0.0220 (10)	0.0182 (10)	0.0218 (10)	−0.0014 (8)	0.0033 (8)	−0.0067 (8)
C1	0.0217 (11)	0.0199 (12)	0.0155 (10)	0.0000 (9)	0.0008 (9)	0.0017 (9)
C2	0.0236 (12)	0.0227 (12)	0.0195 (11)	0.0025 (10)	0.0017 (9)	−0.0011 (10)
C3	0.0200 (12)	0.0279 (13)	0.0219 (12)	0.0008 (10)	0.0031 (9)	0.0015 (10)
C4	0.0213 (12)	0.0267 (13)	0.0259 (12)	−0.0034 (11)	0.0009 (10)	0.0003 (10)
C5	0.0235 (12)	0.0210 (12)	0.0206 (11)	0.0009 (10)	0.0015 (10)	−0.0029 (10)
C6	0.0223 (11)	0.0192 (11)	0.0165 (11)	0.0017 (10)	0.0043 (9)	0.0020 (9)
C7	0.0248 (12)	0.0191 (12)	0.0172 (11)	−0.0010 (10)	0.0037 (9)	−0.0007 (9)
C8	0.0226 (11)	0.0172 (12)	0.0173 (11)	0.0000 (9)	0.0050 (9)	−0.0017 (9)
C9	0.0221 (12)	0.0209 (12)	0.0251 (12)	0.0004 (10)	0.0064 (10)	−0.0027 (10)
C10	0.0200 (11)	0.0252 (13)	0.0199 (11)	−0.0057 (10)	0.0014 (9)	0.0008 (10)
C11	0.0260 (12)	0.0235 (13)	0.0231 (12)	−0.0030 (11)	0.0028 (10)	−0.0024 (10)
C12	0.0378 (15)	0.0267 (14)	0.0234 (12)	−0.0078 (12)	0.0061 (11)	−0.0060 (11)
C13	0.0330 (14)	0.0308 (15)	0.0253 (13)	−0.0115 (12)	−0.0006 (11)	−0.0021 (11)

Geometric parameters (Å, °)

S1—C1	1.748 (2)	C4—C5	1.384 (3)
S1—C8	1.755 (2)	C4—H4	0.97 (3)
O1—C7	1.242 (3)	C5—C6	1.389 (3)
O2—C13	1.370 (3)	C5—H5	0.93 (3)
O2—C10	1.385 (3)	C7—C8	1.499 (3)
N1—C7	1.342 (3)	C8—C9	1.337 (3)
N1—C6	1.409 (3)	C9—C10	1.433 (3)
N1—H1N	0.89 (3)	C9—H9	0.93 (2)
C1—C6	1.393 (3)	C10—C11	1.359 (3)
C1—C2	1.400 (3)	C11—C12	1.420 (3)
C2—C3	1.376 (3)	C11—H11	0.98 (3)
C2—H2	0.96 (3)	C12—C13	1.334 (4)
C3—C4	1.393 (3)	C12—H12	0.93 (3)
C3—H3	0.99 (3)	C13—H13	0.93 (3)
C1—S1—C8	104.12 (11)	C1—C6—N1	122.0 (2)
C13—O2—C10	106.29 (19)	O1—C7—N1	120.2 (2)
C7—N1—C6	129.0 (2)	O1—C7—C8	119.9 (2)
C7—N1—H1N	116.2 (19)	N1—C7—C8	119.90 (19)
C6—N1—H1N	114.7 (19)	C9—C8—C7	118.0 (2)
C6—C1—C2	119.1 (2)	C9—C8—S1	120.93 (17)
C6—C1—S1	122.87 (17)	C7—C8—S1	121.11 (16)
C2—C1—S1	118.01 (17)	C8—C9—C10	127.5 (2)
C3—C2—C1	120.7 (2)	C8—C9—H9	118.3 (15)
C3—C2—H2	119.5 (15)	C10—C9—H9	114.1 (15)
C1—C2—H2	119.8 (15)	C11—C10—O2	109.0 (2)
C2—C3—C4	119.9 (2)	C11—C10—C9	135.9 (2)
C2—C3—H3	121.7 (16)	O2—C10—C9	115.2 (2)
C4—C3—H3	118.3 (16)	C10—C11—C12	107.1 (2)
C5—C4—C3	119.9 (2)	C10—C11—H11	122.5 (16)
C5—C4—H4	119.7 (15)	C12—C11—H11	130.4 (16)
C3—C4—H4	120.4 (15)	C13—C12—C11	106.7 (2)
C4—C5—C6	120.4 (2)	C13—C12—H12	129 (2)
C4—C5—H5	122.7 (15)	C11—C12—H12	124 (2)
C6—C5—H5	116.9 (15)	C12—C13—O2	110.9 (2)
C5—C6—C1	120.0 (2)	C12—C13—H13	134.7 (18)
C5—C6—N1	118.0 (2)	O2—C13—H13	114.3 (18)
C8—S1—C1—C6	4.7 (2)	O1—C7—C8—C9	8.7 (3)
C8—S1—C1—C2	−174.43 (17)	N1—C7—C8—C9	−169.4 (2)
C6—C1—C2—C3	−0.7 (3)	O1—C7—C8—S1	−171.80 (17)
S1—C1—C2—C3	178.49 (18)	N1—C7—C8—S1	10.0 (3)
C1—C2—C3—C4	−0.2 (3)	C1—S1—C8—C9	168.76 (19)
C2—C3—C4—C5	0.3 (4)	C1—S1—C8—C7	−10.7 (2)
C3—C4—C5—C6	0.4 (4)	C7—C8—C9—C10	179.5 (2)
C4—C5—C6—C1	−1.2 (3)	S1—C8—C9—C10	0.1 (4)

C4—C5—C6—N1	178.4 (2)	C13—O2—C10—C11	−0.2 (2)
C2—C1—C6—C5	1.4 (3)	C13—O2—C10—C9	−179.4 (2)
S1—C1—C6—C5	−177.75 (17)	C8—C9—C10—C11	18.1 (4)
C2—C1—C6—N1	−178.2 (2)	C8—C9—C10—O2	−162.9 (2)
S1—C1—C6—N1	2.6 (3)	O2—C10—C11—C12	0.0 (3)
C7—N1—C6—C5	174.8 (2)	C9—C10—C11—C12	179.0 (3)
C7—N1—C6—C1	−5.6 (4)	C10—C11—C12—C13	0.1 (3)
C6—N1—C7—O1	−179.4 (2)	C11—C12—C13—O2	−0.2 (3)
C6—N1—C7—C8	−1.2 (3)	C10—O2—C13—C12	0.3 (3)

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	0.89 (3)	2.00 (3)	2.881 (3)	177 (3)
C2—H2 $\cdots$ O2 ⁱⁱ	0.96 (3)	2.81 (2)	3.446 (3)	124.2 (19)
C3—H3 $\cdots$ O1 ⁱⁱⁱ	0.99 (3)	2.85 (3)	3.573 (3)	130.2 (19)
C3—H3 $\cdots$ O2 ⁱⁱ	0.99 (3)	2.78 (3)	3.420 (3)	122.6 (19)
C4—H4 $\cdots$ CgC1—C6) ^{iv}	0.97 (3)	3.07 (3)	3.688 (3)	123 (2)
C5—H5 $\cdots$ O1 ⁱ	0.93 (3)	2.73 (2)	3.490 (3)	138.8 (18)
C11—H11 $\cdots$ Cg(S1/N1/C1/C6—C8) ^v	0.98 (3)	3.06 (3)	3.749 (3)	129 (2)
C13—H13 $\cdots$ O2 ^{vi}	0.93 (3)	2.68 (3)	3.359 (3)	130 (2)

Symmetry codes: (i) $-x+1/2, -y+3/2, -z+1/2$; (ii) $x+1/2, -y, z$; (iii) $x+1/2, -y+1, z$; (iv) $-x+1, y+1/2, -z+1/2$; (v) $x, y-1, z$; (vi) $-x, -y, -z$.