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Two regioisomers of condensed thioheterocyclic triazine synthesized from 6-phenyl-3-thioxo-2,3,4,5-tetrahydro-1,2,4-triazin-5-one

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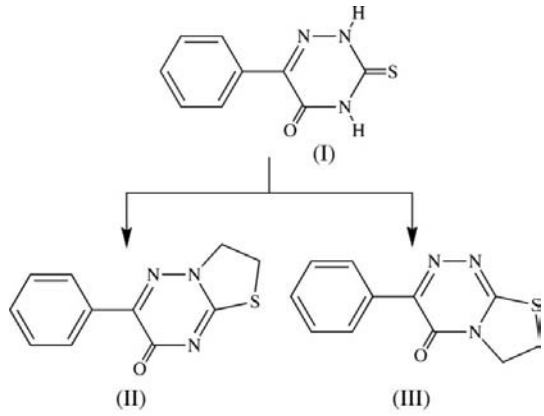
In the crystal structure of 6-phenyl-3-thioxo-2,3,4,5-tetrahydro-1,2,4-triazin-5-one, $C_9H_7N_3OS$, (I), the 1,2,4-triazine moieties are connected by face-to-face contacts through two kinds of double hydrogen bonds (N—H···O and N—H···S), which form planar ribbons along the *a* axis. The ribbons are crosslinked through C—H···π interactions between the phenyl rings. The molecular structures of two regioisomeric compounds, namely 6-phenyl-2,3-dihydro-7*H*-1,3-thiazolo[3,2-*b*]-[1,2,4]triazin-7-one, $C_{11}H_9N_3OS$, (II), and 3-phenyl-6,7-dihydro-4*H*-1,3-thiazolo[2,3-*c*][1,2,4]triazin-4-one, $C_{11}H_9N_3OS$, (III), which were prepared by the condensation reaction of (I) with 1,2-dibromoethane, have been characterized by X-ray crystallography and spectroscopic studies. The crystal structures of (II) and (III) both show two crystallographically independent molecules. While the two compounds are isomers, the unit-cell parameters and crystal packing are quite different and (II) has a chiral crystal structure.

Comment

6-Phenyl-3-thioxo-2,3,4,5-tetrahydro-1,2,4-triazin-5-one, (I), was prepared as an intermediate in the synthesis of thioheterocyclic derivatives (Arndt *et al.*, 1984; Nyitrai *et al.*, 1967), which are used in the creation of biologically active reagents (Odds & Abbott, 1984; Boschelli *et al.*, 1993). Because the base part of thioxotriazinone, *i.e.* 5-hydroxy-3-mercapto-1,2,4-triazine, in (I) is widely considered to be an active constituent of biomolecules (Fotouhi *et al.*, 2008), information about its structure and intermolecular interactions is crucial for chemists and pharmacologists. While many synthetic studies have been reported, few crystal structure analyses of derivatives of thioxotriazinone are known. Only four reports of the methyl derivatives (Ferrari *et al.*, 1995; Voutsas *et al.*, 1978) and its coordination complexes (Ghassemzadeh *et al.*, 2004, 2005) were found during a search of the Cambridge Structural Database (CSD, Version 5.30 of November 2008; Allen, 2002). Thus, we hope that this paper concerning the results of structural studies of (I) and the reaction products 6-phenyl-2,3-dihydro-7*H*-1,3-thiazolo[3,2-*b*][1,2,4]triazin-7-one, (II), and 3-phenyl-6,7-dihydro-4*H*-1,3-thiazolo[2,3-*c*][1,2,4]triazin-4-one, (III), will provide a good basis for understanding the intermolecular interactions and for designing further reactions.



Generally, two isomeric substances of thiazolotriazinone, *viz*. 7*H*-thiazolo[3,2-*b*][1,2,4]triazin-7-one and 4*H*-thiazolo-[2,3-*c*][1,2,4]triazin-4-one derivatives [analogues of (II) and (III), respectively], are obtained by the reaction of the respective dihalogenoalkanes and thioxotriazinones [analogues of (I)], which can be substituted by various alkyl, cycloalkyl, aryl, heteroaryl and benzyl groups. However, the isomers have been mainly identified from the synthetic procedures and by spectroscopic studies. Only one crystallographic study has been carried out, on benzyl-substituted 7*H*-thiazolo[3,2-*b*][1,2,4]triazin-7-one (Miyamoto *et al.*, 1991). Thus, we considered that a full characterization of the two isomers was essential for understanding the difference in the molecular structures and the spectroscopic results. Therefore, the crystal structures of the starting compound, (I), and both of the phenyl-substituted isomers, (II) and (III), are now discussed, along with the results of the condensation reaction.

In the crystal structure of (I), the phenyl and heterocyclic rings are not coplanar and the dihedral angle between the planes of the two rings defined by atoms N1/N2/C3/N4/C5/C6 and C11–C16 is 31.38 (4)° (Fig. 1). The bond lengths in the heterocyclic ring are given in Table 1. The N1—N2, N2—C3,

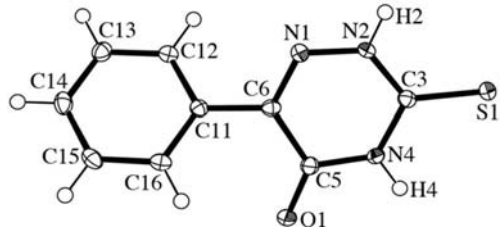


Figure 1
The molecular structure of (I) at 100 K, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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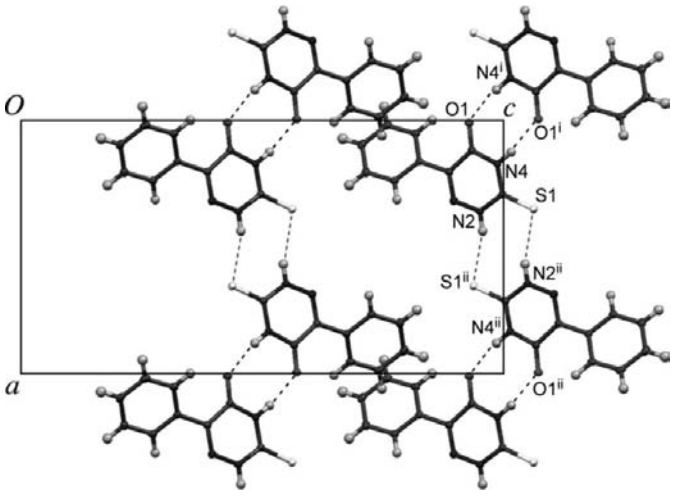

Figure 2
A view of part of the crystal structure of (I), showing the one-dimensional hydrogen-bonded ribbons generated by pairs of N—H···O and N—H···S hydrogen bonds. Symmetry codes are as in Table 2.

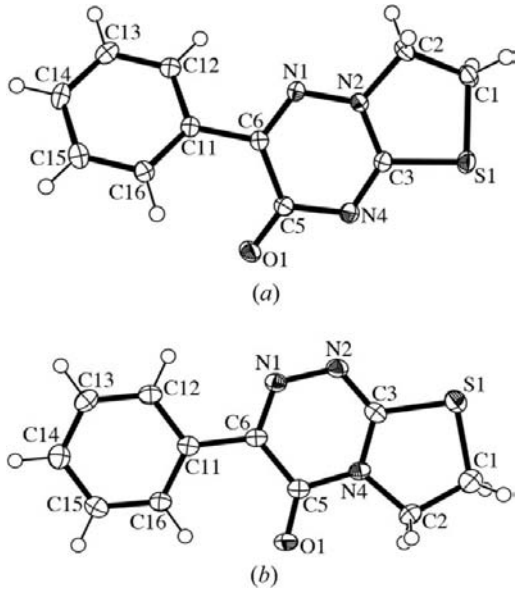

Figure 3
One of the two symmetry-independent molecules in each of (*a*) (II) and (*b*) (III) at 100 K, showing the atom-labelling schemes. Displacement ellipsoids are drawn at the 50% probability level.

C3—N4 and N4—C5 distances correspond with those predicted for the conjugated bond lengths between the single and double bonds, and the bond distances C6—C5 and C6—N1 are those for localized single and double bonds, respectively. The heterocyclic ring is planar and the r.m.s. deviation of the six ring atoms from the mean ring plane is 0.006 Å. In the crystal packing (Fig. 2), there are pairs of N—H···O hydrogen bonds that link amino atom N4 in the reference molecule at (*x*, *y*, *z*), *via* atom H4, across a centre of inversion to carbonyl atom O1 of the molecule at ($-x$, $-y+1$, $-z+2$) and *vice versa*, while pairs of N—H···S hydrogen bonds link amino atom N2, *via* atom H2, across a centre of inversion on the other side of the reference molecule to thiocarbonyl atom S1 of the molecule at ($-x+1$, $-y+1$, $-z+2$) and back again (Table 2). Accordingly, these interactions link the molecules of (I) into one-dimensional ribbons which propagate parallel to the [100] direction. The ribbons are crosslinked by two different intermolecular C—H···π(arene) interactions between the phenyl groups of adjacent molecules: C12···$Cg1^{iii}$ = 3.4527 (15) Å, H12···$Cg1^{iii}$ = 2.76 Å, C12—H12···$Cg1^{iii}$ = 130°, C15···$Cg1^{iv}$ = 3.5085 (16) Å, H15···$Cg1^{iv}$ = 2.77 Å, C15—H15···$Cg1^{iv}$ = 135°, where *Cg*1 is the centroid of the phenyl ring [symmetry codes: (iii) $-x+\frac{1}{2}, y-\frac{1}{2}, z$; (iv) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$].

The same hydrogen-bonded ribbons are also found in the methyl-substituted derivative (Ferrari *et al.*, 1995). The overlay (Macrae *et al.*, 2006) of the atoms of the thioheterocyclic rings of (I) and the methyl derivative shows the same structural characteristics (the r.m.s. deviation of the atoms is 0.03 Å), which indicates that the conformations of the thioheterocyclic rings are almost independent of the substitution of the phenyl and methyl groups.

The reaction of (I) and 1,2-dibromoethane gave the thioheterocyclic compounds (II) and (III) in respective yields of 52 and 19%. Both compounds were isolated by column chromatography and crystallized from 2-propanol. The greater yield of (II) compared with that of (III) is consistent with the previous report (Arndt *et al.*, 1984), indicating that the acidity of the H atom at N2—H2 is higher than that at N4—H4 in starting compound (I) because of the expanding π-conjugation of N2. Accordingly, the N2—C3 bond length in (I) [1.3487 (18) Å] is slightly but significantly shorter than that of C3—N4 [1.3670 (17) Å].

The crystal structures of (II) and (III) each have two crystallographically independent molecules in the asymmetric unit. Here, only one of the symmetry-independent molecules of each structure, *viz.* (II*a*) and (III*a*), is shown in Fig. 3. Selected bond lengths in the heterocyclic rings are summarized in Table 1. The molecular structure of (II*a*) in Fig. 3(*a*) shows that the lateral ethylene chain links atoms N2 and S1 in a pseudo-eclipsed conformation; the torsion angle S1—C1—C2—N2 is −28.20 (17)° and S2—C21—C22—N22 in (II*b*) is 26.76 (16)°. C3—N4 and C6—N1 in the heterocyclic ring are double bonds and C5—C6 is a single bond. The phenyl and six-membered heterocyclic rings are not coplanar and the dihedral angle between them is 26.17 (8)° in (II*a*) and 44.25 (5)° in (II*b*). The molecular structure of (III*a*) in Fig. 3(*b*) shows that the lateral ethylene chain links atoms N4 and S1 in an eclipsed conformation; the torsion angle S1—C1—C2—N4 is 11.09 (19)° and S2—C21—C22—N24 in (III*b*) is −9.3 (2)°. N2—C3 and C6—N1 in the heterocyclic ring are double bonds and C5—C6 is a single bond. The phenyl and six-membered heterocyclic rings are almost coplanar, the dihedral angle between these rings being 7.38 (10)° in (III*a*) and 7.81 (11)° in (III*b*). The molecules of (III) are overall much more planar than those of (II), being influenced by the crystal packing as discussed below. The five-membered rings, including the S atom and Csp^3 atoms, in (III) are flatter than those in (II); the r.m.s. deviation of the ring atoms from their mean planes is

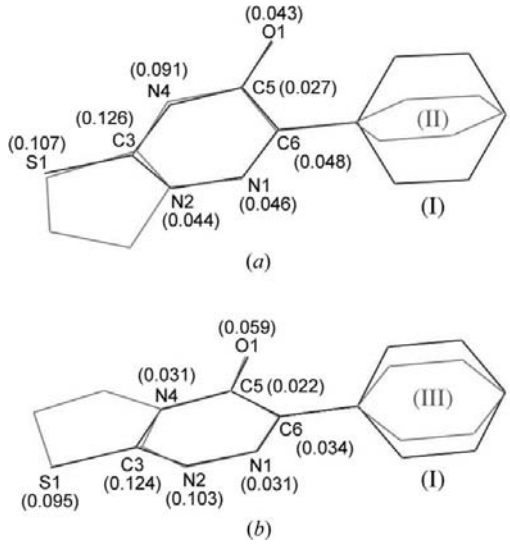

Figure 4
Conformational differences between the molecules in the different structures estimated by superposition of the six-membered heterocyclic ring atoms plus atoms O1 and S1; (*a*) the average r.m.s. value is 0.07 Å between (I) (black) and (II*a*) (grey); (*b*) the average r.m.s. is 0.07 Å between (I) (black) and (III*a*) (grey).

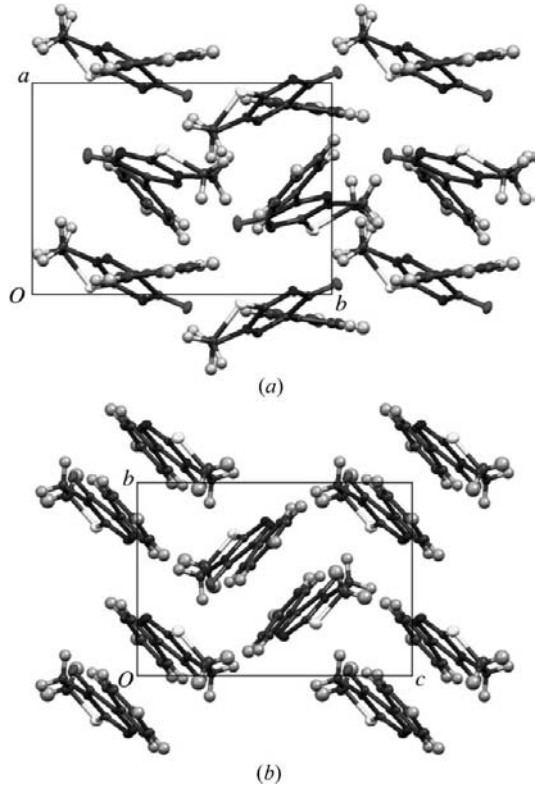

Figure 5
(*a*) The crystal packing of (II), viewed along the *c* axis, and (*b*) that of (III) viewed along the *a* axis.

0.1272 Å in (II*a*), 0.1256 Å in (II*b*), 0.0532 Å in (III*a*) and 0.0450 Å in (III*b*). The changes in the conformations of the heterocyclic six-membered ring in (I) upon the condensation reaction to give (II) and (III) have been analysed by superimposing the molecules and finding the best fit to the six-membered heterocyclic ring atoms plus atoms O1 and S1. Because the average r.m.s. deviations of these atoms in the overlay between (I)/(II*a*) [or (III*a*)] is smaller than that of (I)/(II*b*) [or (III*b*)], the conformational difference after the reaction was estimated by using (II*a*) and (III*a*) in Fig. 4. While the average r.m.s. deviations for (I)/(II) and (I)/(III) show great similarity, 0.07 Å, the differences in the positions of atoms N4, C3 and S1 in (II) and atoms N2, C3 and S1 in (III) are larger than those for the other atoms.

Characterization of derivatives (II) and (III) was also performed using IR and NMR spectroscopies, and mass spectrometry (MS). In the IR spectra, the distinctive C═O band is observed at 1665 cm^{-1} for (II) and 1669 cm^{-1} for (III). The differences in the C═O, N4—C5 and C5—C6 bond lengths for compounds (II) and (III) indicate that the double bond of (II) is more localized than that of (III). Because the π-conjugation is less expanded in (II) than in (III), the lower energy of the C═O stretching for (II) is consistent. Here, IR shifts between the phenyl-substituted derivatives were smaller than for the methyl- and ethyl-substituted derivatives (see *Experimental*), probably because the π-systems compensate for the localization of the C═O double bonds. In the detailed analysis of the mass spectra fragments, the peak at m/z = 128 was observed without the 'phenyl/C6—N1 unit (m/z = 103)' for (II) and the peak at m/z = 203 was observed without the 'ethylene unit (m/z = 28)' for (III). These facts are compatible with the crystal structures. Because the C6—N1 bond length of (II) is shorter than that of (III) and the localized π-conjugation cannot expand to the N2 atoms, the fragment of m/z = 103 was observed for (II). These differences were also observed for the methyl- and ethyl-substituted derivatives. Additionally, the heteronuclear multiple bond correlation (HMBC) of the two-dimensional NMR spectrum of (III) showed a correlation peak between C5 and H2, while no correlation was observed between C5 and the ethylene H atoms for (II).

The crystal packings of (II) and (III) are quite different, as shown in Figs. 5(*a*) and 5(*b*), respectively. In the crystal of (II), the planes of the condensed heterocyclic rings in (II*a*) and (II*b*) involve a significant offset of the centroids of the rings to give columnar arrangements of alternating (II*a*) and (II*b*) molecules along the *a* axis. The five-membered rings and the phenyl rings have face-to-face contact, but this does not appear to involve π–π stacking. The columns are linked through C1—H1*B*···O1^{i}, C13—H13···O1^{ii} and C21—H21*B*···N24^{iii} hydrogen-bond interactions (symmetry codes are as in Table 3). In the crystal packing of (III), however, the molecules associate pairwise between (III*a*)/(III*a*) or (III*b*)/(III*b*). These pairs of molecules clearly show intermolecular π–π stacking between the electron-rich phenyl ring and the electron-poor six-membered ring of centrosymmetrically related molecules. The shortest intermolecular atom-to-atom distances are 3.320 (2) (C3···C15^{ii}), 3.332 (2) (C5···C11^{ii}), 3.342 (2) (C25···C31^{iii}) and 3.362 (2) Å (C26···C26^{iii}), while the centroid–centroid distances between the rings containing atoms C3 and C15^{ii} and between the rings containing atoms

C25 and C31^{iii} are 3.7000 (11) and 3.6689 (12) Å, respectively, with corresponding angles between the ring planes of 5.78 (8) and 7.33 (9)° [symmetry codes: (ii) $-x+1, -y+1, -z+1$; (iii) $-x, -y+1, -z$]. The pairs are further linked through weak C2—H2B···N1^{i} and C22—H22A···N21^{i} hydrogen bonds [symmetry code: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$] (Table 4).

In conclusion, this study clearly characterizes two isomers of the phenyl-substituted thioheterocyclic triazine by means of crystal structure analyses and spectroscopic studies, and highlights the π-conjugation within the triazine ring of (II) and (III).

Experimental

Compound (I) was prepared by procedures similar to those reported previously by Arndt *et al.* (1984). Typically, an aqueous solution (400 ml) of methyl benzoylformate (150 mmol) and thiosemicarbazide (150 mmol) was stirred at 343 K for 1.5 h. After cooling, 30% aqueous NaOH (*ca* 40 ml) was added slowly to the solution to adjust the pH to 11 and the mixture was then stirred at 353 K for 4 h. By the addition of 6 *M* HCl to pH = 1, a white powder of (I) was obtained (62% yield). The products of (I) (50 mmol) and dibromoethylene (50 mmol) were added to an EtOH solution (30 ml) of Na (50 mmol), then the mixture was refluxed for 1 h. Na_2CO_3 (25 mmol) was added to the solution and the mixture refluxed for 10 h. After removing the solid by filtration, the solvent was evaporated to give a white powder of compounds (II) and (III). The products were purified by column chromatography (silica gel, AcOEt) to give pure products of (II) in 52% yield and (III) in 19% yield. Single crystals of (I), (II) and (III) suitable for X-ray crystallography were obtained by cooling hot solutions of 2-propanol.

IR, MS and NMR spectra were measured using Shimadzu FT–IR-8400S (KBr disc), Shimadzu GCMS QP2010Plus and Varian NMR (Mercury-300 and UNITY-400) spectrometers, respectively. IR data on C=O stretching bands and MS fragments are as follows. For (II), IR: 1665 cm^{-1}; MS: 231, 128, 60 *m*/*z*; 6-methyl-2,3-dihydro-7*H*-thiazolo[3,2-*b*][1,2,4]triazin-7-one (methyl derivative), IR: 1637 cm^{-1}; MS: 169, 128, 60 *m*/*z*; 6-ethyl-2,3-dihydro-7*H*-thiazolo[3,2-*b*][1,2,4]triazin-7-one (ethyl derivative), IR: 1649 cm^{-1}; MS: 183, 128, 60 *m*/*z*. For (III), IR: 1669 cm^{-1}; MS: 231, 203, 103 *m*/*z*; 3-methyl-6,7-dihydro-4*H*-thiazolo[2,3-*c*][1,2,4]triazin-4-one (methyl derivative), IR: 1684 cm^{-1}; MS: 169, 141, 56 *m*/*z*; 3-ethyl-6,7-dihydro-4*H*-thiazolo-[2,3-*c*][1,2,4]triazin-4-one (ethyl derivative), IR: 1672 cm^{-1}; MS: 183, 154, 56 *m*/*z*.

Compound (I)

Crystal data

$C_9H_7N_3OS$
M_r = 205.24
Orthorhombic, *Pbca*
a = 11.1891 (11) Å
b = 7.4401 (7) Å
c = 21.359 (2) Å
V = 1778.1 (3) Å^3
Z = 8
Mo *K*α radiation
μ = 0.33 mm^{-1}
T = 100 K
0.30 × 0.20 × 0.06 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: empirical (using intensity measurements) (*SADABS*; Sheldrick, 1996)
T_{min} = 0.908, T_{max} = 0.981
9167 measured reflections
2023 independent reflections
1746 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.082
S = 1.07
2023 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Selected bond distances (Å) for (I), (II) and (III).

	(I)	(II*a*)	(II*b*)	(III*a*)	(III*b*)
N1—N2	1.3603 (16)	1.352 (2)	1.3556 (19)	1.374 (2)	1.375 (2)
N2—C3	1.3487 (18)	1.347 (2)	1.353 (2)	1.298 (2)	1.297 (2)
C3—N4	1.3670 (17)	1.303 (3)	1.302 (2)	1.354 (2)	1.354 (2)
N4—C5	1.3789 (17)	1.390 (2)	1.385 (2)	1.385 (2)	1.382 (2)
C6—C5	1.4823 (19)	1.493 (3)	1.494 (2)	1.477 (2)	1.474 (2)
C6—N1	1.3059 (17)	1.303 (2)	1.300 (2)	1.311 (2)	1.311 (2)
C3—S1	1.6673 (14)	1.7436 (17)	1.7467 (17)	1.7462 (18)	1.7452 (18)
C5—O1	1.2236 (17)	1.219 (2)	1.224 (2)	1.221 (2)	1.224 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···O1^{i}	0.88	1.96	2.8307 (15)	169
N2—H2···S1^{ii}	0.88	2.46	3.3166 (13)	163

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

Compound (II)

Crystal data

$C_{11}H_9N_3OS$
M_r = 231.27
Monoclinic, $P2_1$
a = 8.1257 (4) Å
b = 11.3239 (5) Å
c = 11.3860 (5) Å
β = 99.3630 (10)°
V = 1033.72 (8) Å^3
Z = 4
Mo *K*α radiation
μ = 0.29 mm^{-1}
T = 100 K
0.45 × 0.40 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: empirical (using intensity measurements) (*SADABS*; Sheldrick, 1996)
T_{min} = 0.880, T_{max} = 0.944
5756 measured reflections
2992 independent reflections
2939 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.061
S = 1.04
2992 reflections
290 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack & Bernardinelli (2000), 577 Friedel pairs
Flack parameter: 0.02 (7)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1B···O1^{i}	0.99	2.50	3.183 (2)	126
C13—H13···O1^{ii}	0.95	2.56	3.150 (2)	120
C21—H21B···N24^{iii}	0.99	2.45	3.351 (2)	151

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

Table 4
Hydrogen-bond geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2B···N1^{i}	0.99	2.40	3.338 (2)	158
C22—H22A···N21^{i}	0.99	2.40	3.325 (2)	155

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

Compound (III)

Crystal data

$C_{11}H_9N_3OS$
M_r = 231.27
Monoclinic, $P2_1/c$
a = 21.627 (3) Å
b = 8.1222 (10) Å
c = 11.8888 (15) Å
β = 104.638 (2)°
V = 2020.6 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 100 K
0.30 × 0.30 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: empirical (using intensity measurements) (*SADABS*; Sheldrick, 1996)
T_{min} = 0.916, T_{max} = 0.971
10860 measured reflections
4517 independent reflections
3632 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.101
S = 1.03
4517 reflections
289 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

All H atoms were placed in geometrically idealized positions and refined as riding, with aromatic C—H = 0.95 Å, aliphatic C—H = 0.99 Å and N—H = 0.88 Å, and with U_{iso}(H) = 1.2U_{eq}(parent atom).

For all compounds, data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN3135). Services for accessing these data are described at the back of the journal.

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