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Planarity of heteroaryldithiocarbazic acid derivatives showing tuberculostatic activity. III. Mono- and diesters of 3-(pyrazin-2-ylcarbonyl)dithiocarbazic acid[1]

Małgorzata Szczesio,[a] Andrzej Olczak,[a] Jolanta Gołka,[a] Katarzyna Gobis,[b] Henryk Foks[b] and Marek L. Główka[a]*

[a]Institute of General and Ecological Chemistry, Technical University of Łódź, Poland, and [b]Department of Organic Chemistry, Medical University of Gdańsk, Poland
Correspondence e-mail: marekglo@p.lodz.pl

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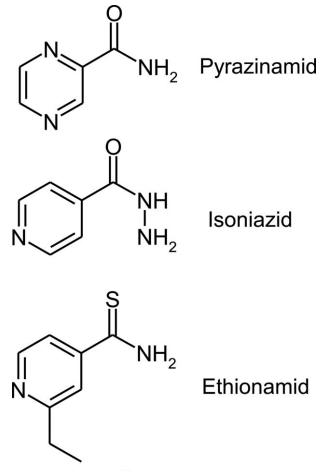
Methyl 2-(pyrazin-2-ylcarbonyl)hydrazinecarbodithioate, $C_7H_8N_4OS_2$, (*E*1), *N*′-[bis(methylsulfanyl)methylidene]pyrazine-2-carbohydrazide, $C_8H_{10}N_4OS_2$, (*F*1), *N*′-[bis(methylsulfanyl)-methylidene]-6-methoxypyrazine-2-carbohydrazide, $C_9H_{12}N_4O_2S_2$, (*F*2), and methyl 1-methyl-2-(pyrazin-2-ylcarbonyl)-hydrazinecarbodithioate, $C_8H_{10}N_4OS_2$, (*G*1), can be considered as derivatives of classical (thio)amide-type tuberculostatics, and all are moderately active against *Mycobacterium tuberculosis*. This study was undertaken in a search for relationships between activity and specific intramolecular interactions, especially conjugations and hydrogen-bond contacts, and the molecular structures were compared with respective amine analogues, also active against the pathogen. Despite the differences between the amine and carbonyl groups with opposite functions in the hydrogen bond, the two types of structure show a surprisingly similar planar geometry, mostly due to the conjugations aided by the bifurcated intramolecular hydrogen-bond contact between the N—H group of the central hydrazide group as donor and a pyrazine N atom and an S atom of the dithio function as acceptors. Planarity was suggested to be crucial for the tuberculostatic activity of these compounds. The *N*-methylated derivative (*G*1) showed a significant twist at the N—N bond [torsion angle = −121.9 (3)°] due to the methyl substitution, which precludes an intramolecular N—H···S contact and the planarity of the whole molecule. Nonetheless, the compound shows moderate tuberculostatic activity.

Comment

For many years, tuberculosis was considered a disease of the past, limited mainly to poor countries which could not afford expensive treatment, and this led to decreased interest in searching for more effective drugs. However, a more recent rise in mortality rates and the spread of the disease in developed countries, attributed to the emergence of multi-drug-resistant strains, have changed this attitude. The search for new lead compounds was continued by several groups, including that of Foks (Foks *et al.*, 2000; Gobis, Foks, Zwolska & Augustynowicz-Kopeć, 2006; Gobis, Foks, Żuralska & Kędzia, 2006), who synthesized several chemical classes of potential tuberculostatic agents, in particular those containing either pyrazin-2-yl-carbonimidoyldithiocarbazic acid esters, heteroaroylcarbonimidoyldithiocarbazate or heteroaroyl-dithiocarbazate systems. The compounds comprise molecular features present in the classical amide-type tuberculostatics, in particular heteroaroyl, amide and thioacid functions, such as in pyrazinamid, isoniazid and ethionamid (see Scheme 1), which act through the formation of a covalent adduct with nicotinamide adenine dinucleotide (Wang *et al.*, 2007).



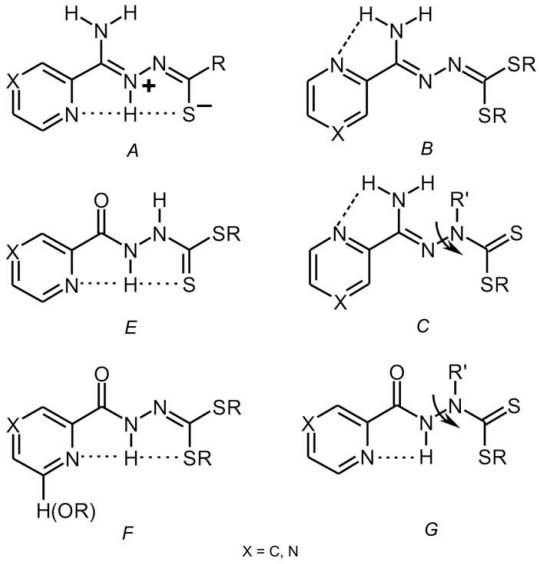
Scheme 1

Our earlier crystallographic studies of selected representatives of 3-[amino(pyrazin-2-yl)methylidene]thiocarbazic acid esters (see Scheme 2, formula *A*) showed that all of them maintained planarity of the whole molecule, except for the terminal aliphatic substituents (Główka *et al.*, 2005; Olczak *et al.*, 2007; Orlewska *et al.*, 2001). The planarity observed in these structures was caused by extensive conjugation aided by a bifurcated intramolecular hydrogen-bond contact between the protonated atom N3 as a donor and two acceptors. One acceptor was a negatively charged S atom and the other was an *ortho*-positioned N atom of a pyrazine or pyridine ring on the other side of the donor (Scheme 2, formula *A*). The analysis of these data resulted in a working hypothesis that planarity of the molecules is a prerequisite for their tuberculostatic activity (Orlewska *et al.*, 2001).

Next, we showed that similar planarity was maintained in compounds lacking an H atom at N3, such as the dithioesters (formula *B* in Scheme 2), due to conjugation, and, except in thioesters (formula *A*), an intramolecular N—H···N contact was maintained between the same *ortho* N atom of the reversed pyridine (or pyrazine) ring as acceptor and the N5 amine group as donor. The overall planarity was lost only in

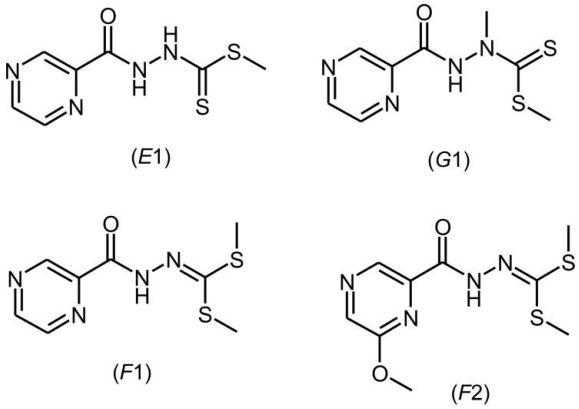
[1] For Part II, see Olczak *et al.* (2011).

the case of N2 substitution in an appropriate monoester (formula *C* in Scheme 2) (Olczak *et al.*, 2011).



Scheme 2

The compounds described here represent both *S*-monothioesters [(*E*1) and (*G*1)] and *S*,*S*′-dithioesters [(*F*1) and (*F*2)] of heteroaroyldithiocarbazic acids (see Scheme 3 and Figs. 1–4), in which an aryl-C(NH$_2$)=N– function present in the former study (Olczak *et al.*, 2011) has been replaced by a 2-pyrazine-C(O)—NH– function. Thus, the compounds may be considered derivatives of the well known tuberculostatic pyrazinamid (Scheme 1). Despite the significant differences between these compounds and their amine analogues *A*, *B* and *C* (Scheme 2), we supposed that, due to the presence of an H atom at N3 in compounds (*E*1), (*F*1) and (*F*2), an analogous intramolecular hydrogen-bond contact will be formed as in the N—H heteroarylcarbamidoyl hydrazinium cation (compound *A* in Scheme 2). As a result, the molecules should be planar and the compounds may show similar antibacterial activity.



Scheme 3

In addition, we included in this study compound (*G*1) (with a methyl substituent at atom N2), which was unable to maintain planarity of the whole molecule due to spatial

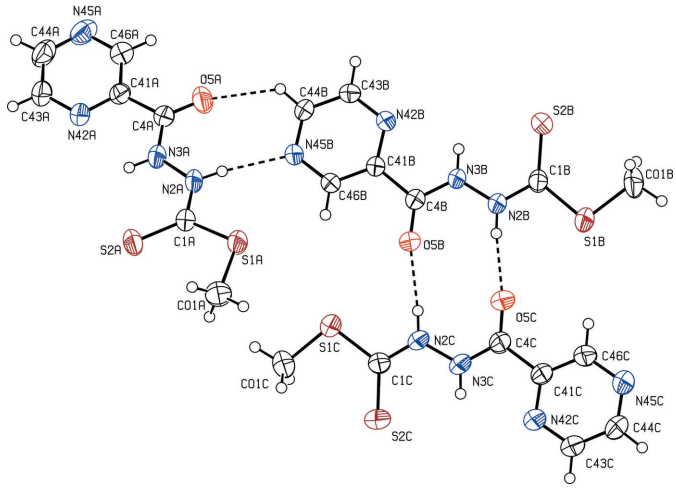


Figure 1
The molecular structure of (*E*1), showing the atom-numbering scheme. Intermolecular hydrogen bonds determining the packing of the molecules in the crystal structure are indicated as dashed lines. Displacement ellipsoids are drawn at the 50% probability level.

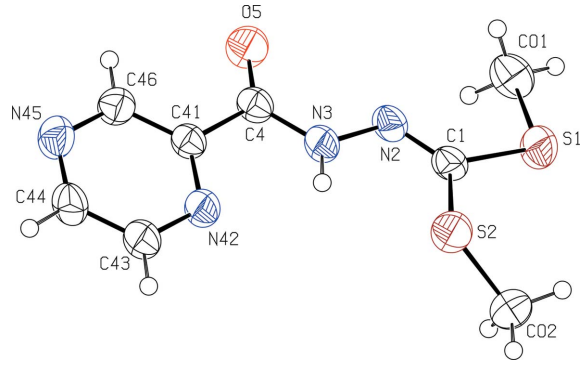


Figure 2
The molecular structure of (*F*1), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

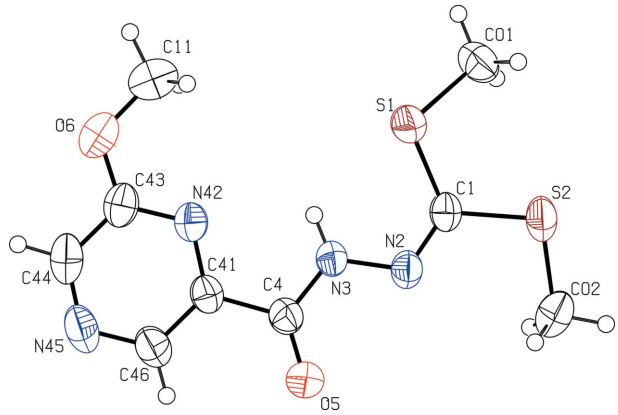


Figure 3
The molecular structure of (*F*2), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

repulsion between this methyl group and the carbonyl group (see Scheme 2). As the compound showed some tuberculostatic activity, it was important to examine changes in its molecular structure. The question concerning planarity could

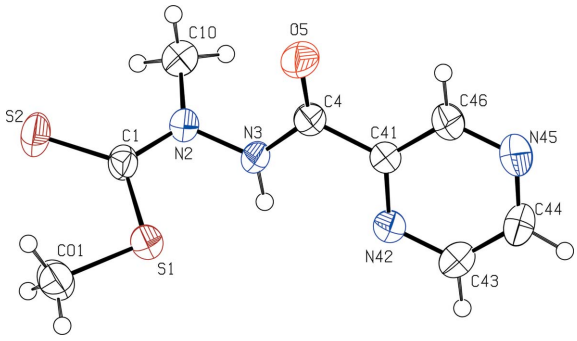


Figure 4
The molecular structure of (*G*1), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

not be answered by simple inspection of the Cambridge Structural Database (CSD, Version 5.32; Allen, 2002), as among about 20 structures comprising esters of heteroaroyldithiocarbazic acid, N2-substituted derivatives are not present. Structures in which the N atom is incorporated into the pyrazine or pyridine ring at the *ortho* position, thus playing an important role of acceptor in an N3—H···N(aryl) intramolecular hydrogen-bond contact, are similarly absent.

As we expected, the molecules of compounds (*E*1), (*F*1) and (*F*2) are planar except for the terminal ester group, while the molecule of (*G*1) shows a twist at the N—N bond (Fig. 4) caused by spatial repulsion introduced by the methyl group at N2. Thus, we suppose that the main factor responsible for planarity is conjugation from the aryl ring to the thio group. Conjugation along the C1═N2—N3—C4—C41 chain in (*F*1) and (*F*2), or along the S═C1—N2—N3—C4—C41 chain in (*E*1), is confirmed by both the values of the respective torsion angles in these fragments, all being close to 180°, and the shortening of the formally single bonds N3—C4 and C4—C41 (Table 5).

Due to the overall planarity of the molecules of (*E*1), (*F*1) and (*F*2), there are two short intramolecular hydrogen-bond contacts, both with the N3—H group as the donor (see Scheme 2 and Tables 1–4). The first contact, N3—H···N(pyrazine), is observed in all compounds studied here having an N3—H group, including (*G*1). The H···N distances and N—H···N angles are in the ranges 2.21–2.28 Å and 107–110°, respectively (Table 6). Similar contacts are observed in N-substituted picolinamides found in the CSD, *i.e.* 2.15–2.32 Å and 100–116°. The other intramolecular contact is that of N3—H···S, with H···S distances between 2.42 and 2.55 Å and N—H···S angles between 108 and 113° (Table 6). The values agree well with those found in similar molecules in the CSD (2.37–2.61 Å and 107–113°). The exception is structure (*G*1), in which substitution at atom N2 twists the molecule (see Scheme 2) and makes an intramolecular N3—H···S contact impossible.

There are some doubts concerning the structural significance of these contacts (Table 6), mostly due to the commonly accepted view that hydrogen bonds with *D*—H···*A* angles below 120° do not contribute substantially to the stabilization energy of the structure (Wood *et al.*, 2009). However, the value

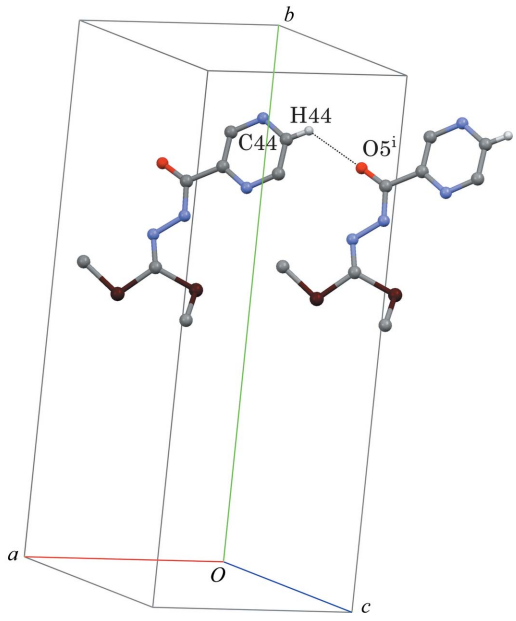


Figure 5
The intermolecular hydrogen-bond contact (dotted line) of (*F*1) determining the packing of the molecules in the crystal structure. [Symmetry code: (i) $x-1, y, z$.]

of 120° refers to intermolecular hydrogen bonds, while in this study we are concerned with intramolecular systems. In the crystallographic literature, arrangements similar to that formed by the N3—H···N contact (*i.e.* five-membered ring motifs) were discussed by Bilton *et al.* (2000) and Galek *et al.* (2010), who found for them a probability of formation of over 70% and an average hydrogen-bond angle of only 109°. Less is known about N—H···S arrangements similar to those present in the structures described in this study. However, it seems from our data (Table 6), and from a couple of examples comprising a five-membered intramolecular N—H···S motif which were found in the CSD, that N3—H···S contacts are geometrically less stressed than N3—H···N ones. Consequently, we believe that both the N3—H···N and N3—H···S close contacts observed in this study may be considered intramolecular resonance-assisted hydrogen bonds of some structural significance. The bonds aid conjugation along the main molecular chain to maintain planarity of the whole molecule, except for the terminal substituents of the ester group.

The molecular packing in (*E*1), (*F*1) and (*F*2) is determined mainly by their planarity (Fig. 1 and Figs. 5–6) and relatively weak intermolecular hydrogen bonds. In the diesters (*F*1) and (*F*2), there is only one weak C—H···O interaction in each structure. In (*F*1), C44—H···O5($x-1, y, z$) hydrogen bonds join the molecules into infinite *C*(7) chains parallel to the [100] direction, while in (*F*2) the molecules form dimers – $R_2^2(10)$ rings according to the graph-set definition of Bernstein *et al.* (1995) – through C46—H···O5($-x+1, y, \frac{3}{2}-z$) hydrogen bonds. In ester (*E*1), which comprises one hydrogen-bond donor group, N2—H, in each of the three independent molecules, the intermolecular interactions are more complicated. Molecules *B* and *C* are connected by N2*B*—H···O5*C* and

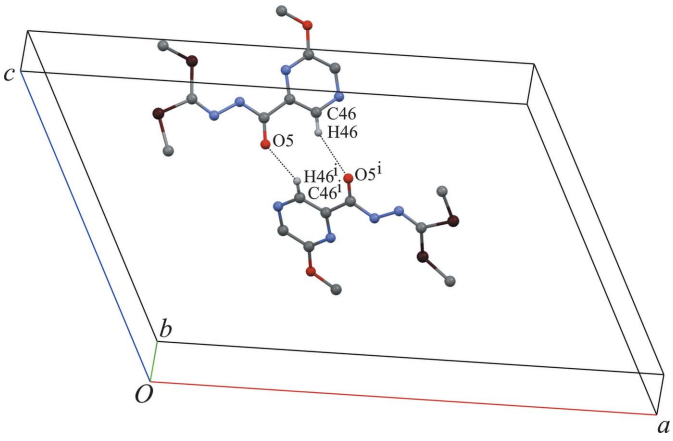


Figure 6
The intermolecular hydrogen-bond contacts (dotted lines) of (*F*2) determining the packing of the molecules in the crystal structure. [Symmetry code: (i) $-x+1, y, -z+\frac{3}{2}$.]

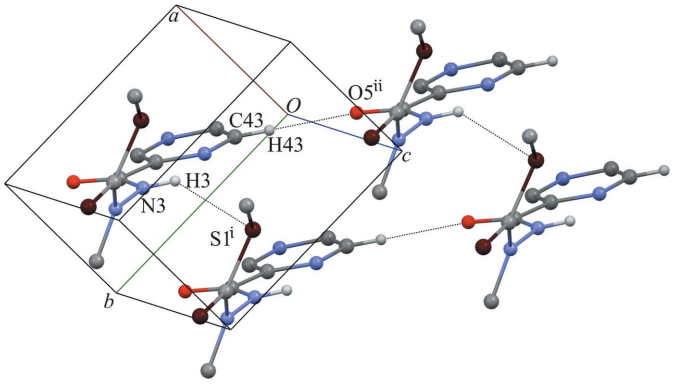


Figure 7
The intermolecular hydrogen-bond contacts (dotted lines) of (*G*1) determining the packing of the molecules in the crystal structure. [Symmetry codes: (i) $x-1, y, z$; (ii) $x-1, y-1, z$.]

N2*C*—H···O5*B* hydrogen bonds (Table 1), joining molecules into approximately coplanar dimers in which $R_2^2(10)$ rings are present. Molecules *A* and *B* are connected into asymmetric dimers (Fig. 1) by C44*B*—H···O5*A* and N2*A*—H···N45*B* hydrogen bonds, forming $R_2^2(8)$ rings. In the *N*′-methylated ester (*G*1), one weak intermolecular C43—H···O5($x-1$, $y-1$, z) hydrogen bond is observed (Table 4). The bonds join the molecules into infinite *C*(6) chains running parallel to the [110] direction. In addition, an intermolecular N3—H3···S1($x-1$, y, z) short contact may be considered a weak hydrogen bond (Fig. 7).

Experimental

The syntheses of the title compounds were described by Foks *et al.* (2000) for (*E*1) and (*F*1), by Gobis, Foks, Zwolska & Augustynowicz-Kopeć (2006) for (*G*1), and by Gobis, Foks, Żuralska & Kędzia (2006) for (*F*2). Single crystals of compounds (*E*1), (*F*1), (*F*2) and (*G*1) suitable for X-ray diffraction were obtained from chloroform–ethanol (1:1 *v*/*v*), dioxane, ethanol and ethanol solutions, respectively, by slow evaporation of the solvents at room temperature.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2*A*—H2*A*···N45*B*	0.86	2.13	2.987 (2)	174
C44*B*—H44*B*···O5*A*	0.93	2.36	3.061 (2)	132
N2*B*—H2*B*···O5*C*	0.86	1.94	2.790 (2)	172
N2*C*—H2*C*···O5*B*	0.86	2.00	2.850 (2)	170

Compound (*E*1)

Crystal data

$C_7H_8N_4OS_2$
$M_r = 228.29$
Triclinic, $P\overline{1}$
$a = 7.2326$ (3) Å
$b = 12.7207$ (4) Å
$c = 17.7516$ (5) Å
$\alpha = 77.877$ (3)°
$\beta = 79.096$ (3)°
$\gamma = 73.968$ (4)°
$V = 1519.67$ (9) Å^3
$Z = 6$
Mo $K\alpha$ radiation
$\mu = 0.50$ mm^{-1}
$T = 295$ K
0.3 × 0.2 × 0.05 mm

Data collection

Kuma KM-4 CCD area-detector diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
$T_{min} = 0.876$, $T_{max} = 1.000$
18215 measured reflections
6187 independent reflections
4162 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.094$
$S = 1.00$
6187 reflections
382 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Compound (*F*1)

Crystal data

$C_8H_{10}N_4OS_2$
$M_r = 242.32$
Monoclinic, $P2_1/c$
$a = 7.8332$ (2) Å
$b = 21.0883$ (4) Å
$c = 7.3920$ (2) Å
$\beta = 116.121$ (4)°
$V = 1096.36$ (5) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.46$ mm^{-1}
$T = 297$ K
0.2 × 0.1 × 0.05 mm

Data collection

Kuma KM-4 CCD area-detector diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
$T_{min} = 0.946$, $T_{max} = 1.000$
12975 measured reflections
2236 independent reflections
1960 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.087$
$S = 1.07$
2236 reflections
139 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Table 2
Hydrogen-bond geometry (Å, °) for (*F*1).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C44—H44···O5^{i}	0.93	2.59	3.219 (2)	125

Symmetry code: (i) $x-1, y, z$.

Table 3
Hydrogen-bond geometry (Å, °) for (*F*2).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C46—H46···O5^{i}	0.93	2.53	3.385 (3)	153

Symmetry code: (i) $-x+1, y, -z+\frac{3}{2}$.

Compound (*F*2)

Crystal data

$C_9H_{12}N_4O_2S_2$
M_r = 272.35
Monoclinic, *C*2/*c*
a = 23.111 (2) Å
b = 7.5812 (7) Å
c = 15.6547 (14) Å
β = 115.430 (2)°
V = 2477.1 (4) Å^3
Z = 8
Mo *K*α radiation
μ = 0.43 mm^{-1}
T = 270 K
0.5 × 0.2 × 0.1 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.516, T_{max} = 1.000
27591 measured reflections
3051 independent reflections
2351 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.126
S = 1.03
3051 reflections
157 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Compound (*G*1)

Crystal data

$C_8H_{10}N_4OS_2$
M_r = 242.32
Monoclinic, $P2_1$
a = 4.0900 (4) Å
b = 6.4482 (6) Å
c = 20.7828 (19) Å
β = 91.151 (2)°
V = 548.00 (9) Å^3
Z = 2
Mo *K*α radiation
μ = 0.47 mm^{-1}
T = 290 K
0.35 × 0.2 × 0.1 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.716, T_{max} = 1.000
12608 measured reflections
2642 independent reflections
2523 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.123
S = 1.10
2642 reflections
138 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), with 1175 Friedel pairs
Flack parameter: 0.18 (11)

Table 4
Hydrogen-bond geometry (Å, °) for (*G*1).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···S1^{i}	0.86	2.85	3.473 (3)	131
C43—H43···O5^{ii}	0.93	2.37	3.297 (4)	179

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

Table 5
Selected bond lengths (Å) and torsion angles (°) for the title compounds.

Structure	N2—N3	N3—C4	C4—C41	C1—N2—N3—C4
(*E*1)	1.377 (2)	1.338 (2)	1.494 (3)	176.6 (2)
(*E*1)	1.372 (2)	1.333 (2)	1.493 (2)	−178.79 (19)
(*E*1)	1.382 (2)	1.322 (3)	1.498 (3)	179.6 (2)
(*F*1)	1.3785 (17)	1.344 (2)	1.502 (2)	177.07 (15)
(*F*2)	1.3839 (19)	1.342 (2)	1.499 (2)	−177.06 (16)
(*G*1)	1.395 (3)	1.341 (4)	1.497 (4)	−121.9 (3)

Table 6
Intramolecular hydrogen-bond contact geometry (Å, °) for the title compounds.

N3—H3···N42	H···N	N···N	N—H···N
(*E*1)	2.25	2.641 (2)	108
(*E*1)	2.25	2.648 (2)	109
(*E*1)	2.27	2.663 (3)	108
(*F*1)	2.21	2.625 (2)	110
(*F*2)	2.25	2.652 (2)	108
(*G*1)	2.28	2.666 (4)	107

N3—H3···S	H···S	N···S	N—H···S
(*E*1)	2.54	2.9228 (18)	108
(*E*1)	2.50	2.9031 (16)	109
(*E*1)	2.55	2.9305 (19)	108
(*F*1)	2.43	2.8775 (14)	113
(*F*2)	2.42	2.8587 (18)	112

H atoms were located in difference Fourier maps and subsequently geometrically optimized and allowed for as riding atoms, with C—H = 0.95 Å for aromatic CH groups, 0.97 Å for secondary CH_2 groups and 0.96 Å for methyl groups, and N—H = 0.86 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N). For (*G*1), the precision of the Flack *x* parameter [Flack (1983); *x* = 0.18 (11)], whether calculated by the 'hole-in-one' method or using TWIN/BASF in *SHELXL97* (Sheldrick, 2008), indicated that the structure was correctly oriented with respect to the polar-axis direction, but was too imprecise to rule out the possibility of partial inversion twinning. However, the value of the Hooft *y* parameter [Hooft *et al.* (2008); *y* = 0.20 (2)] certainly suggests that partial inversion twinning is present.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007) for (*E*1) and (*F*1); *APEX2* (Bruker, 2002) for (*F*2) and (*G*1). Cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007) for (*E*1) and (*F*1); *SAINT-Plus* (Bruker, 2003) for (*F*2) and (*G*1). Data reduction: *CrysAlis RED* for (*E*1) and (*F*1); *SAINT-Plus* for (*F*2) and (*G*1). For all compounds, program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) for (*E*1) and (*G*1); *SHELXTL* (Sheldrick, 2008) for (*F*1) and (*F*2). For all compounds, molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *PLATON*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD3386). Services for accessing these data are described at the back of the journal.

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