Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Planarity of heteroaryldithiocarbazic acid derivatives showing tuberculostatic activity. IV. Diesters of benzoylcarbonohydrazonodithioic acid[1]

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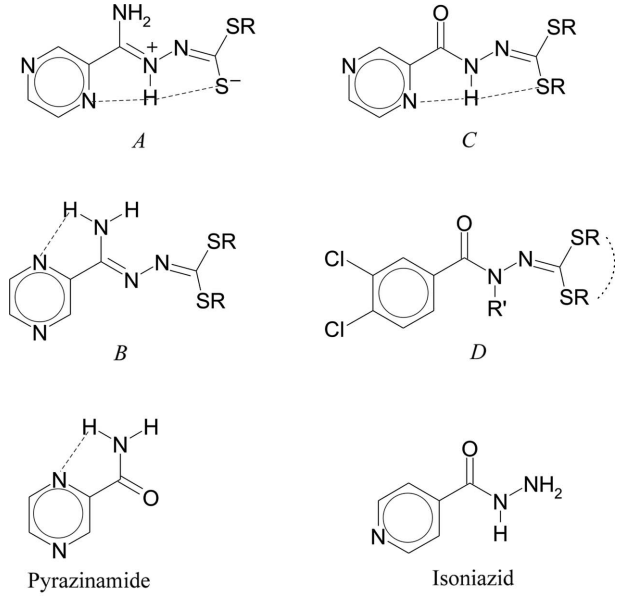
Received 25 November 2011
Accepted 14 January 2012
Online 4 February 2012

Dimethyl (3,4-dichlorobenzoyl)carbonohydrazonodithioate, $C_{10}H_{10}Cl_2N_2OS_2$, (*D*1), dibenzyl (3,4-dichlorobenzoyl)carbonohydrazonodithioate, $C_{22}H_{18}Cl_2N_2OS_2$, (*D*2), dimethyl (3,4-dichlorobenzoyl)-1-methylcarbonohydrazonodithioate, $C_{11}H_{12}Cl_2N_2OS_2$, (*D*3), 3,4-dichloro-*N*′-(1,3-dithiolan-2-ylidene)-*N*-methylbenzohydrazide, $C_{11}H_{10}Cl_2N_2OS_2$, (*D*4), were synthesized as potential tuberculostatics. Compound (*D*1) (with two molecules in the asymmetric unit) was the only one showing tuberculostatic activity of the same range as the common drugs isoniazid and pyrazinamide. The molecular structures of the studied compounds depend on the substitution at the N atom adjacent to the carbonyl group. In the case of the unsubstituted derivatives (*D*1) and (*D*2), their central frames are generally planar with a twist of the 3,4-dichlorophenyl ring by 30–40°. Until now, coplanarity of the aromatic ring with the (methylene)carbonohydrazone fragment has been considered a prerequisite for tuberculostatic activity. The *N*-methylated derivatives (*D*3) and (*D*4) show an additional twist along the N—C(=O) bond by 20–30° due to the spatial repulsion introduced by the methyl substituent.

Comment

Because of the emergence of multi-drug-resistant strains of *Mycobacterium tuberculosis* in developed countries, significant efforts have been put into a search for new lead tuberculostatics. Among others, Foks and coworkers have synthesized hundreds of compounds, many of which showed reasonable activity against tuberculosis (Pancechowska-Ksepko *et al.*, 1993; Milczarska *et al.*, 1998, 1999; Foks *et al.*, 2002; Gobis *et al.*, 2011).

Several crystal structures of selected representatives of these compounds were determined by our group in a search for relationships between activity and molecular structure (Główka *et al.*, 2005; Olczak *et al.*, 2007, 2011; Szczesio *et al.*, 2011). The main working hypothesis tested in these studies was that planarity of a molecule is a prerequisite for tuberculostatic activity (Olczak *et al.*, 2007). The hypothesis was based on an observation from our very first study on the molecular structure of 2-[amino(pyridine-2-yl)methylene]-hydrazinecarbodithioic acid methyl ester in the crystalline state (Główka *et al.*, 2005). This active compound in the crystal state exists in the zwitterionic form (formula *A* in Scheme 1) and it is planar (except for the terminal ester group) due to conjugations and intramolecular hydrogen bonds (Główka *et al.*, 2005; Olczak *et al.*, 2007; Orlewska *et al.*, 2001). Surprisingly, overall planarity was also observed in the crystals of [amino(pyrazin-2-yl)methylene]carbonohydrazonodithioic acid diesters showing tuberculostatic activity (formula *B* in Scheme 1) (Olczak *et al.*, 2011). Owing to the lack of an H atom at N3, the same intramolecular hydrogen-bond contacts as those observed in all type *A* (Scheme 1) structures could not be formed. Instead, another intramolecular hydrogen contact was observed, *i.e.* that between the amine group as a donor and the N atom at the *ortho* position of the pyrazine ring as an acceptor, similar to that observed in all pyrazinamide structures [Cambridge Structural Database (CSD), Version 5.32; Allen, 2002]. In the case of type *B* compounds (Scheme 1), planarity of the central structure frame (as evidenced by the torsion angle at the N—N bond) was secured by the conjugation alone (Olczak *et al.*, 2011).



Scheme 1

Next, the amine function in compounds of the types *A* and *B* has been replaced by a carbonyl group (compounds type *C* in Scheme 1), with some of the resulting structures also showing promising activity (Foks *et al.*, 2004; Sitarz *et al.*,

[1] For Part III, see Szczesio *et al.* (2011).

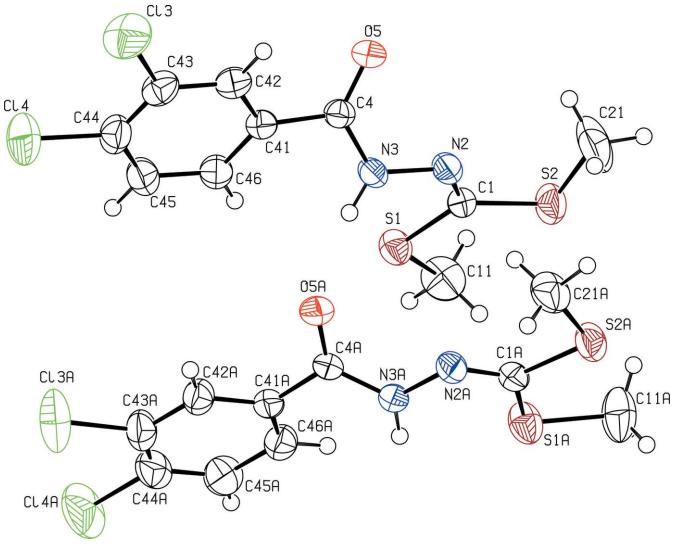


Figure 1
The molecular structure of the two independent molecules of (*D*1), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

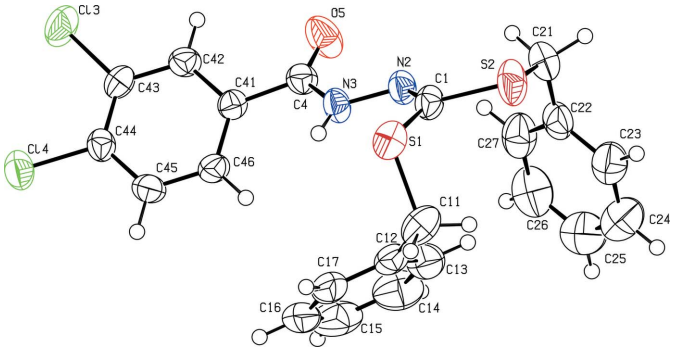


Figure 2
The molecular structure of (*D*2), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

2005). This was in accord with our hypothesis as the compounds could form similar intramolecular hydrogen bonds as in compounds of type *A*. The overall planarity of the molecules was confirmed in our studies on their crystal structures (Szczesio *et al.*, 2011).

Our earlier observation that the conjugation in type *B* compounds was sufficient to secure planarity of the central molecular chain prompted us to synthesize the respective analogues *D* (Scheme 1), in which we have removed the final possibility of keeping the aromatic ring coplanar with the hydrazide fragment due to the intramolecular N5—H···N(ring) hydrogen bond (formula *D* in Scheme 1). We were encouraged by the structural characteristics of the well known agent isoniazid, in which this intramolecular hydrogen bond could also not be formed (Scheme 1). Most of the type *B* compounds show weak tuberculostatic activity [minimum inhibitory concentration (MIC) about 50 mg ml^{-1}] but for some the values are 20 times lower (Foks *et al.*, 1992; Orlewska *et al.*, 1995).

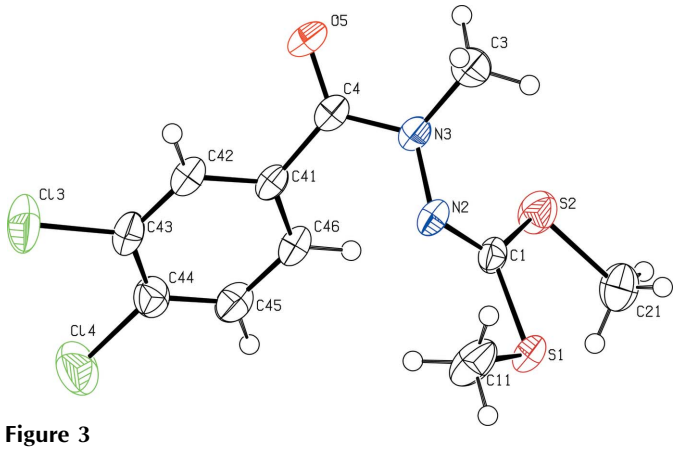
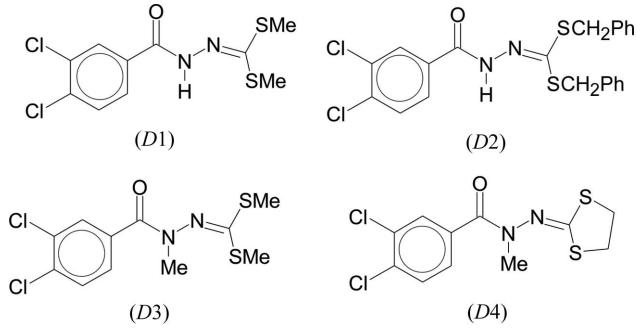


Figure 3
The molecular structure of (*D*3), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

We report here a study on the crystal structures of four type *D* compounds, (*D*1)–(*D*4) (Figs. 1–4), which differ in the ester groups (methyl, benzyl or cyclic) or in the substitution by the methyl group at the N3 atom (see Scheme 2). There are seven diesters of benzoylcarbonohydrazonodithioic acid (CSD, Version 5.32; Allen, 2002). All are unsubstituted at the N3 atom. However, no mention has been made of their tuberculostatic activity in the literature.



Scheme 2

In accord with our expectation, replacement of the aromatic ring containing an N atom in the *ortho* position by a phenyl ring resulted in its significant twist of 30–40° in comparison with planar 2-pyrazine or 2-pyrazine derivatives studied earlier. Similar values are observed in 19 benzamide structures found in the CSD, with an average of 22° (CSD, Version 5.32; Allen, 2002).

The other significant difference in the molecular structures of the *D* type and the formerly studied *C* type (Szczesio *et al.*, 2011) compounds is the conformation around the N2—N3 bond. The absolute values of the respective C1═N2—N3—C4 torsion angles are close to 177° in two planar diesters of (pyrazine-2-carbonyl)carbonohydrazonodithioic acid (type *C*) (Szczesio *et al.*, 2011) as compared with values of 166.39 (15)° in (*D*2), −139.31 (18) and −150.87 (17)° in (*D*1), 131.65 (16)° in (*D*3) and 126.49 (17)° in (*D*4) (Table 1). The lack of an H atom at the N3 atom in (*D*3) and (*D*4) also results in their very

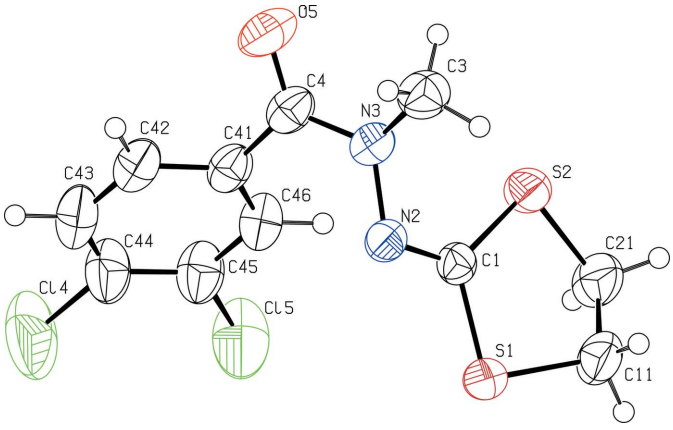


Figure 4
The molecular structure of (*D*4), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

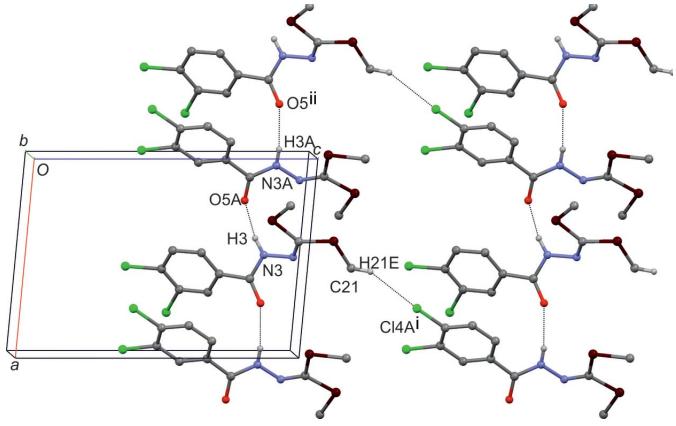


Figure 5
The intermolecular hydrogen bonds of (*D*1) determining the packing of molecules in the crystal. [Symmetry codes: (i) $x+1, y, z+1$; (ii) $x-1, y, z$.]

different conformation around their N3—C4 bonds in comparison with N3-unsubstituted aroylcarbonohydrazonodithioic acid esters (Table 1). The crucial N2—N3—C4—C41 torsion angle is about 180° in (*D*1) and (*D*2) and only 21–31° in (*D*3) and (*D*4), due to spatial repulsion introduced by the methyl group in the latter structures. A similar difference is seen in the related N2—N3—C4═O5 torsion angle, being about 4° in (*D*1) and (*D*2), and about 155° in (*D*3) and (*D*4) (Table 1).

The question arises whether the observed differences in the molecular structures may be related to low tuberculostatic activity of the N3-substituted compounds. The fact is that only N3-unsubstituted derivatives show higher activity, especially (*D*1) (Gobis *et al.*, 2011).

The differences in conformations along the main chain correlate with differences in the respective bond lengths (Table 1) indicating the change in conjugations. The largest difference is found for the N2—N3 bond length, which is 1.435 (2) Å (torsion about 130°) in (*D*3), 1.433 (2) Å (torsion about 125°) in (*D*4), 1.416 (2) and 1.402 (2) Å (torsion about

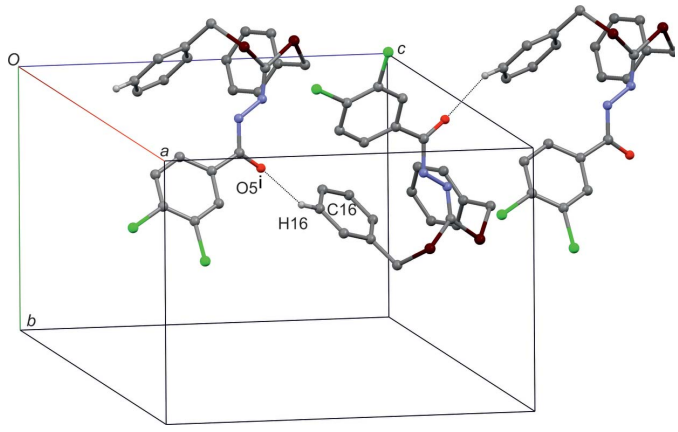


Figure 6
The intermolecular hydrogen bonds of (*D*2) determining the packing of molecules in the crystal. [Symmetry code: (i) $x, -y, z-\frac{1}{2}$.]

−140 and −150°) in (*D*1) and 1.3703 (18) Å (torsion about 166°) in (*D*2) due to increasing (in the listed order) *p*(N3)–*p*(N2═C1) conjugation for the antiperiplanar (torsion about 180°) conformation.

Another interesting issue concerns the intramolecular N3—H···S hydrogen-bond contact possible in (*D*1) and (*D*2). Although the H···S distances of 2.49 and 2.52 Å in (*D*1) and 2.59 Å in (*D*2) are significantly shorter than the sum of their van der Waals radii (2.89 Å), the angles at the H atom are only 104, 99 and 108°, respectively. So, despite the H···S distances indicating hydrogen bonds, there is some doubt in the literature concerning contribution of the contacts to the stabilization energy of the crystals, especially because of the low values of the N3—H···S angles (Wood *et al.*, 2009; Galek *et al.*, 2010; Bilton *et al.*, 2000). Our view on the subject has been presented in Szczesio *et al.* (2011).

In the studied crystals there are several intermolecular hydrogen bonds, some of which are quite strong and important for crystal packing (Figs. 5–7). In (*D*1), the two independent molecules form an infinite $C_2^2(8)$ chain [according to the graph-set definition of Bernstein *et al.* (1995)] parallel to the [100] direction through two intermolecular hydrogen bonds, *viz*. N3—H···O5*A* and N3*A*—H···O5($x-1, y, z$). In addition, in (*D*1), there exists a C21—H···Cl4*A*($x+1, y, z+1$)

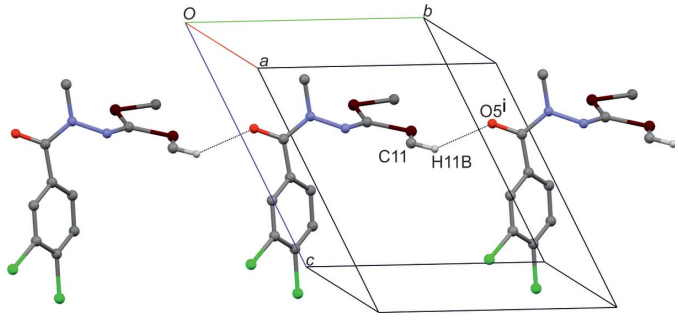


Figure 7
The intermolecular hydrogen bonds of (*D*3) determining the packing of molecules in the crystal. [Symmetry code: (i) $x, y+1, z$.]

contact taking part in the formation of two additional chains, *viz*. $C_2^2(14)$ parallel to the [101] direction with an N3—H···O5*A* hydrogen bond and $C_2^2(16)$ parallel to the [001] direction with an N3*A*—H···O5($x-1, y, z$) hydrogen bond. At the third level of the graph-set theory, the $R_6^6(38)$ ring formed by N3—H···O5*A*, N3*A*—H···O5($x-1, y, z$) and C21—H···Cl4*A*($x+1, y, z+1$) can be identified (Fig. 5). In (*D*2), there is only one weak hydrogen bond, C16—H···O5($x, -y, z-\frac{1}{2}$), forming a $C(11)$ chain parallel to the [001] direction (Fig. 6). In (*D*3), there is also one weak hydrogen bond, C11—H···O5($x, y+1, z$), forming a $C(8)$ chain parallel to the [010] direction (Fig. 7). No significant intermolecular contacts were observed in (*D*4).

Experimental

The synthesis of (*D*1) was described previously by Gobis *et al.* (2011). The other compounds were obtained from 3,4-dichlorobenzohydrazide [for (*D*2)] or 3,4-dichloro-*N*-methylhydrazide [for (*D*3) and (*D*4)] according to the same method. Benzohydrazides were dissolved in a methanol solution of triethylamine and treated with carbon disulfide and the respective halide, *viz*. methyl iodide for (*D*3), benzyl chloride for (*D*2) or ethylene bromide for (*D*4). Single crystals of (*D*1), (*D*2), (*D*3) and (*D*4) suitable for X-ray diffraction were obtained from ethanol solutions by slow evaporation of the solvents at room temperature.

Compound (*D*1)

Crystal data

$C_{10}H_{10}Cl_2N_2OS_2$
$M_r = 309.22$
Monoclinic, $P2_1$
$a = 9.2225$ (4) Å
$b = 11.2741$ (5) Å
$c = 13.0892$ (5) Å
$\beta = 95.367$ (1)°
$V = 1354.99$ (10) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.77$ mm^{-1}
$T = 296$ K
0.3 × 0.3 × 0.1 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.914$, $T_{max} = 1.000$
33334 measured reflections
7766 independent reflections
7138 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.092$
$S = 1.08$
7766 reflections
312 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max} = 0.79$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}
Absolute structure: Flack (1983), 3697 Friedel pairs
Flack parameter: −0.02 (4)

Compound (*D*2)

Crystal data

$C_{22}H_{18}Cl_2N_2OS_2$
$M_r = 461.40$
Monoclinic, $C2/c$
$a = 28.6760$ (11) Å
$b = 10.3705$ (3) Å
$c = 14.7176$ (6) Å
$\beta = 90.900$ (4)°
$V = 4376.2$ (3) Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 0.50$ mm^{-1}
$T = 292$ K
0.55 × 0.4 × 0.3 mm

Data collection

Kuma KM-4 CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)
$T_{min} = 0.942$, $T_{max} = 1.000$
17461 measured reflections
3868 independent reflections
3021 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.084$
$S = 0.94$
3868 reflections
262 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Compound (*D*3)

Crystal data

$C_{11}H_{12}Cl_2N_2OS_2$
$M_r = 323.25$
Triclinic, $P\overline{1}$
$a = 8.9307$ (4) Å
$b = 9.0858$ (7) Å
$c = 10.3873$ (5) Å
$\alpha = 65.039$ (6)°
$\beta = 69.172$ (4)°
$\gamma = 74.892$ (5)°
$V = 708.08$ (7) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.74$ mm^{-1}
$T = 291$ K
0.5 × 0.35 × 0.2 mm

Table 1
Selected bond lengths (Å) and torsion angles (°) for the title compounds.

	C1—N2—N3—C4	N2—N3—C4—C41	N2—N3—C4—O5	N2—N3
(*D*1)	−139.31 (18)	−177.95 (16)	4.0 (3)	1.416 (2)
	−150.87 (17)	−178.19 (15)	3.1 (3)	1.402 (2)
(*D*2)	166.39 (15)	177.63 (13)	−4.1 (2)	1.3703 (18)
(*D*3)	131.65 (16)	−31.2 (2)	152.86 (16)	1.435 (2)
(*D*4)	126.49 (17)	−21.1 (2)	159.99 (18)	1.433 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C21—H21*E*···Cl4*A*^{i}	0.96	2.75	3.565 (3)	144
N3*A*—H3*A*···O5^{ii}	0.86	2.10	2.8434 (18)	144
N3—H3···O5*A*	0.86	2.06	2.834 (2)	149
N3—H3···S1	0.86	2.52	2.7810 (17)	99
N3*A*—H3*A*···S1*A*	0.86	2.49	2.8216 (16)	104

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···S1	0.86	2.59	2.9751 (14)	108
C16—H16···O5^{i}	0.93	2.44	3.294 (3)	153

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

Table 4
Hydrogen-bond geometry (Å, °) for (*D*3).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*B*···O5^{i}	0.96	2.49	3.200 (2)	131

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

Data collection

Kuma KM-4 CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)
T_{min} = 0.447, T_{max} = 1.000
10270 measured reflections
2494 independent reflections
2181 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.097
S = 1.15
2494 reflections
166 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Compound (*D*4)

Crystal data

$C_{11}H_{10}Cl_2N_2OS_2$
M_r = 321.23
Monoclinic, $P2_1/c$
a = 13.3680 (2) Å
b = 8.1817 (1) Å
c = 13.5158 (2) Å
β = 105.415 (1)°
V = 1425.08 (3) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 6.76 mm^{-1}
T = 296 K
0.3 × 0.2 × 0.05 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.423, T_{max} = 0.753
15835 measured reflections
2643 independent reflections
2589 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.100
S = 1.06
2643 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

H atoms were located in difference Fourier maps and subsequently geometrically optimized and allowed for as riding atoms, with C—H = 0.93 Å for aromatic CH groups, 0.97 Å for secondary CH_2 groups and 0.96 Å for methyl groups, N—H = 0.86 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl groups or 1.2U_{eq}(C,N) otherwise.

Data collection: *APEX2* (Bruker, 2005) for (*D*1) and (*D*4); *CrysAlis CCD* (Oxford Diffraction, 2008) for (*D*2) and (*D*3). Cell refinement: *SAINT-Plus* (Bruker, 2008) for (*D*1) and (*D*4); *CrysAlis RED* (Oxford Diffraction, 2008) for (*D*2) and (*D*3). Data reduction: *SAINT-Plus* for (*D*1) and (*D*4); *CrysAlis RED* for (*D*2) and (*D*3). For all compounds, program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON*.

The present study was supported by the Ministry of Science and Higher Education under project No. N204 111735.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: WQ3009). Services for accessing these data are described at the back of the journal.

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