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Five symmetrically substituted 2-aryl-3-benzyl-1,3-thiazolidin-4-ones: supramolecular structures in zero, one and two dimensions

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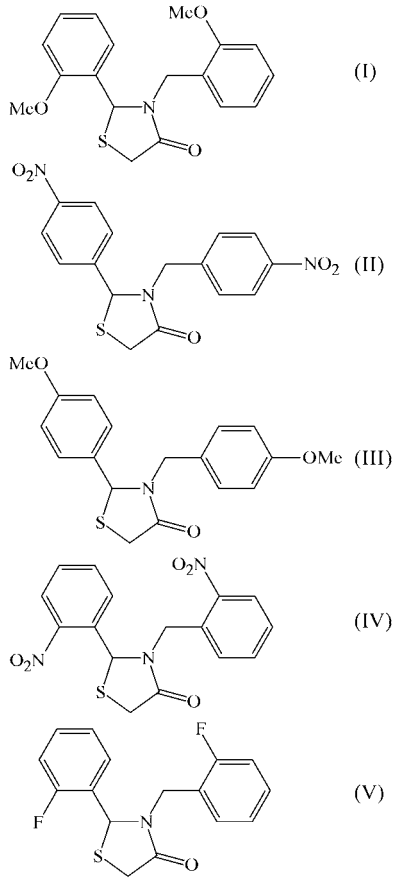
There are no direction-specific interactions between the molecules of 3-(2-methoxybenzyl)-2-(2-methoxyphenyl)-1,3-thiazolidin-4-one, $C_{18}H_{19}NO_3S$, (I); the molecules of 3-(4-nitrobenzyl)-2-(4-nitrophenyl)-1,3-thiazolidin-4-one, $C_{16}H_{13}N_3O_5S$, (II), are linked by four independent C—H···O hydrogen bonds into complex chains of fused rings. In 3-(4-methoxybenzyl)-2-(4-methoxyphenyl)-1,3-thiazolidin-4-one, (III), isomeric with (I), the molecules are linked into sheets by a combination of C—H···O and C—H···π(arene) hydrogen bonds, while in 3-(2-nitrobenzyl)-2-(2-nitrophenyl)-1,3-thiazolidin-4-one, (IV), isomeric with (II), the sheets are built from three independent C—H···O hydrogen bonds and one C—H···π(arene) hydrogen bond, and reinforced by an aromatic π–π stacking interaction. In 3-(2-fluorobenzyl)-2-(2-fluorophenyl)-1,3-thiazolidin-4-one, $C_{16}H_{13}F_2NOS$, (V), where the 2-aryl ring exhibits orientational disorder, the molecules are linked into sheets by a combination of C—H···O and C—H···π(arene) hydrogen bonds, and the sheets are linked in pairs, forming bilayers, by an aromatic π–π stacking interaction.

Comment

We report here the molecular and supramolecular structures of five substituted 2-aryl-3-benzyl-1,3-thiazolidin-4-ones, (I)–(V), all obtained from the reactions of the corresponding aryl aldehydes with L-valine [(*S*)-2-amino-3-methylpropionic acid] and mercaptoacetic acid in the presence of diisopropylethylamine (Cunico *et al.*, 2006). The method of synthesis (Cunico *et al.*, 2006) represents a one-stage simplification of a previously published two-stage procedure (Holmes *et al.*, 1995); in this earlier investigation, it was found that, under the forcing reaction conditions required, the use of enantiomerically pure chiral amines consistently led to products with no enantioselectivity at C2. A preliminary report has appeared on compound (V), establishing proof of the constitution of this unexpected reaction product, but that report gave no stereochemical information nor any discussion of the supramolecular aggregation (Cunico *et al.*, 2006).



In each of compounds (I)–(V) (Figs. 1–5), atom C2 is a stereogenic centre; all the compounds, as prepared, are racemic despite the use of enantiomerically pure L-valine as the source of the ring N atom. Each compound crystallizes in a centrosymmetric space group and for each the reference molecule was selected as one having the *S* configuration at C2.

While the amidic portion of the heterocyclic ring is effectively planar in each compound, overall these rings are all non-planar. In each of compounds (I)–(IV), the heterocyclic ring adopts an envelope conformation, folded across the line C2···C5, while in compound (V), the ring adopts the half-chair conformation, twisted about the S1—C5 bond. The bond distances and angles present no unusual values. The primary interest in the structures is the dramatic influence exerted upon the supramolecular aggregation by the nature and location of the single substituent in the aryl rings; the structural variation involves both the types of direction-specific intermolecular interaction present in the supramolecular structures and the effects of these interactions upon the dimensionality of these structures. We discuss the structures in order of increasing complexity, from the isolated molecules in compound (I) up to the bilayers in compound (V).

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There are no direction-specific intermolecular interactions in the crystal structure of compound (I), which thus consists of effectively isolated molecules.

In the structure of compound (II), there are four independent C—H···O hydrogen bonds (Table 1), which link the molecules into complex chains. Atoms C2 and C37 in the molecule at (x, y, z) act as hydrogen-bond donors, respectively, to nitro atoms O241 and O342 in the molecules at $(x, -1 + y, z)$ and $(x, 1 + y, z)$, so generating by translation a chain of edge-fused $R_2^2(20)$ rings (Bernstein *et al.*, 1995) running parallel to the [010] direction. This chain is weakly reinforced by a further, rather long and possibly adventitious, interaction between C32 at (x, y, z) and O342 at $(x, 1 + y, z)$. Pairs of these chains are linked by the final hydrogen bond in which atom C22 in the molecule at (x, y, z) acts as a donor to ring atom O4 in the molecule at $(1 - x, 1 - y, 1 - z)$, so forming a complex chain of rings (Fig. 6). Two such chains, related to one another by the translational symmetry operations, run along the lines $(\frac{1}{2}, y, 0)$ and $(\frac{1}{2}, y, \frac{1}{2})$, but there are no direction-specific interactions between adjacent chains.

The formation of the hydrogen-bonded sheets in compound (III) is very simple, utilizing only two hydrogen bonds, one of C—H···O and one of C—H···π(arene) type (Table 2). Atoms C5 and C37 in the molecule at (x, y, z) act as hydrogen-bond donors, respectively, to methoxy atom O34 in the molecule at $(-1 + x, -1 + y, z)$ and to the C31–C36 ring in the molecule at $(-1 + x, y, z)$. These interactions thus generate by translation a sheet lying parallel to (001) (Fig. 7). Four such sheets pass through each unit cell, in the domains $0.04 < z < 0.31$, $0.19 < z < 0.46$, $0.54 < z < 0.81$ and $0.69 < z < 0.96$, but there are no direction-specific intermolecular interactions between the sheets, nor is there any interweaving of the pairs of sheets within the domains $0 < z < 0.5$ and $0.5 < z < 1.0$.

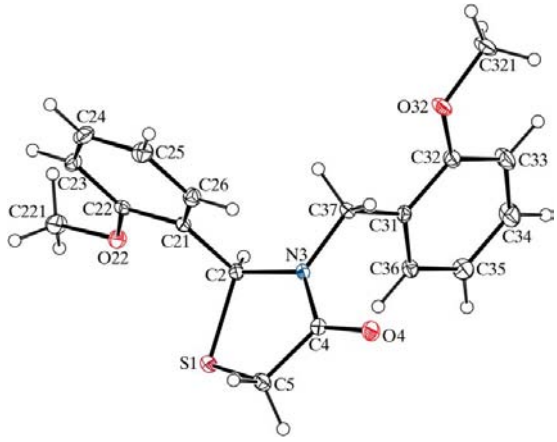

Figure 1
A molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

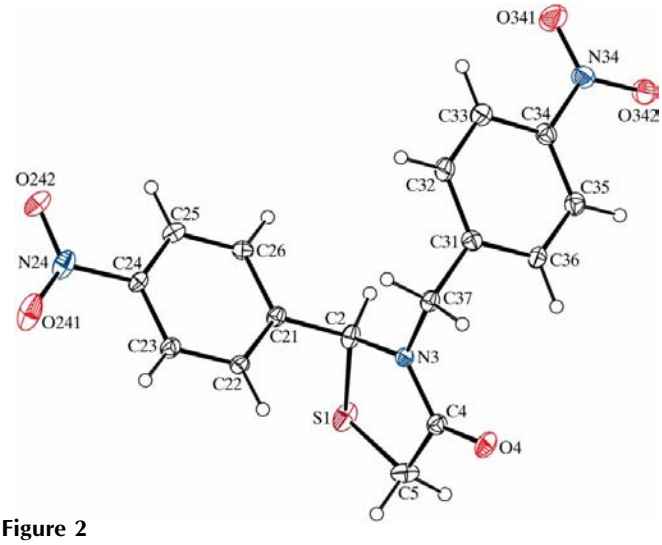

Figure 2
A molecule of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

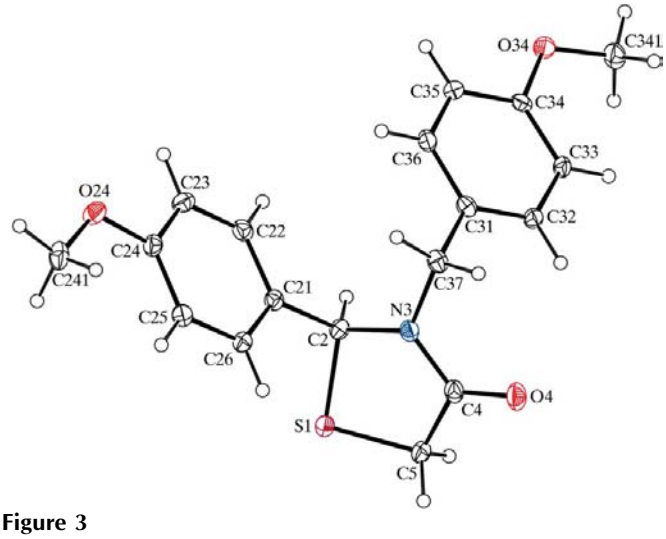

Figure 3
A molecule of (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

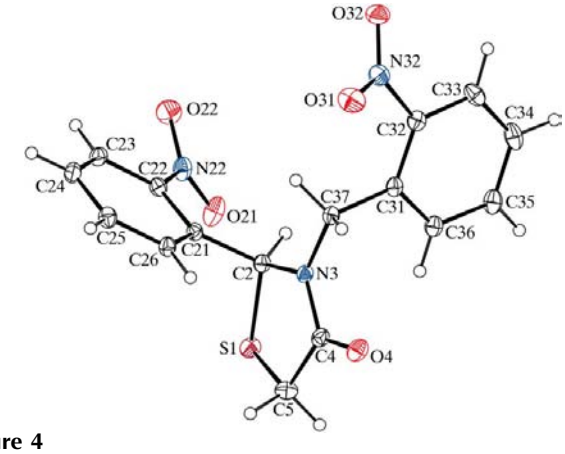

Figure 4
A molecule of (IV), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

The sheet structure of compound (IV) is much more complex than that in compound (III) and it is most readily analysed in terms of two substructures, built, respectively, from two C—H···O hydrogen bonds, and from one C—H···O and one C—H···π(arene) hydrogen bond (Table 3). In the first of these substructures, atoms C24 and C26 in the molecule at (x, y, z) act as hydrogen-bond donors, respectively, to atoms O4 at (x, $1+y$, z) and O31 at ($1-x$, y, $\frac{3}{2}-z$). Propagation of these two interactions by translation and rotation then produces a chain of edge-fused rings running parallel to the [010] direction and generated by the twofold rotation axis along ($\frac{1}{2}$, y, $\frac{3}{4}$), and containing alternating $R_2^2(20)$ and $R_4^4(32)$ rings (Fig. 8).

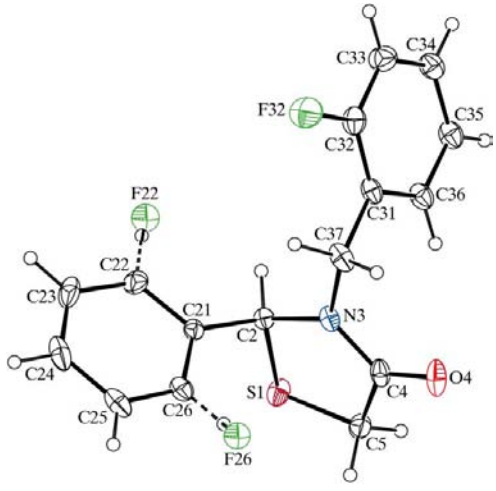

Figure 5
A molecule of (V), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Atom sites F22 and F26, and the associated H-atom sites, have partial occupancies (see *Comment*).

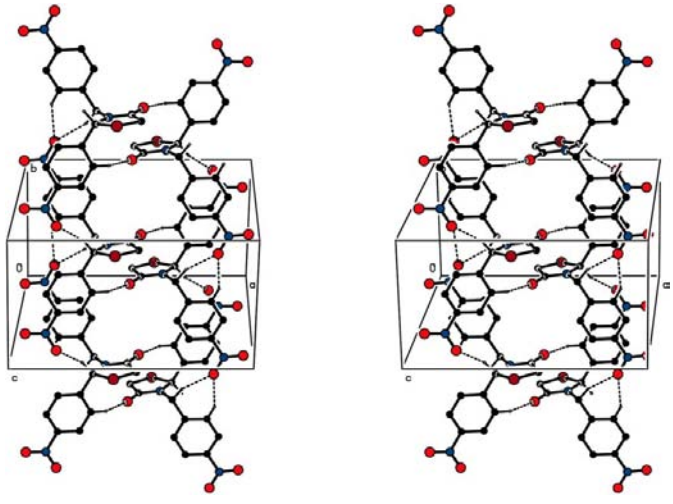

Figure 6
A stereoview of part of the crystal structure of compound (II), showing the formation of a chain of fused rings built from C—H···O hydrogen bonds and running along [010]. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

In the second substructure, atom C35 in the molecule at (x, y, z) acts as a hydrogen-bond donor to atom O4 in the molecule at (x, $-y$, $-\frac{1}{2}+z$), so forming a $C(8)$ chain running parallel to the [001] direction and generated by the c-glide plane at $y=0$ (Fig. 9). At the same time, atom C25 at (x, y, z) acts as a donor to the C31–C36 ring in the molecule at (x, $1-y$, $\frac{1}{2}+z$), so forming another chain along [001], this time generated by the c-glide plane at $y=\frac{1}{2}$. The combination of these two chains along [001] generates a sheet parallel to (100) (Fig. 9). Hence, the combination of this rather simple two-dimensional substructure with the chain of fused rings (Fig. 8) generates a sheet structure of considerable complexity.

The molecule of compound (V) exhibits disorder in the orientation of the C21–C26 ring, where two orientations are related by a 180° rotation about the C2—C21 bond, so that the F atom is disordered over two sites, denoted F22 and F26 (Fig. 5), with occupancies 0.647 (4) and 0.353 (4), respectively. There are only two hydrogen bonds in the structure of compound (V) (Table 4) and these link the molecules into sheets, pairs of which are further linked into bilayers by a single aromatic π–π stacking interaction; the formation of the

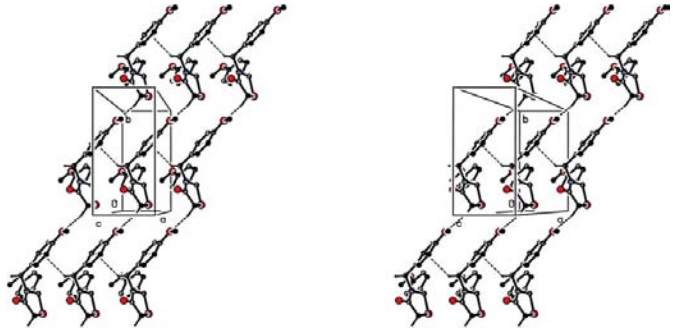

Figure 7
A stereoview of part of the crystal structure of compound (III), showing the formation of a sheet parallel to (001) built from C—H···O and C—H···π(arene) hydrogen bonds. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

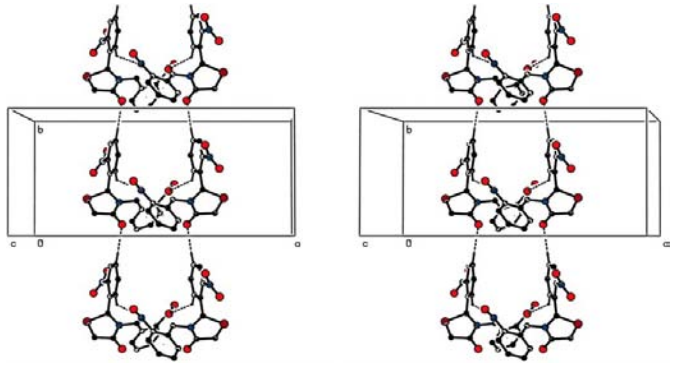

Figure 8
A stereoview of part of the crystal structure of compound (IV), showing the formation of a chain of edge-fused $R_2^2(20)$ and $R_4^4(32)$ rings built from C—H···O hydrogen bonds and running along [010]. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

bilayers is not influenced by the disorder. Atom C24 in the molecule at (x, y, z) acts as a hydrogen-bond donor to atom O4 in the molecule at $(x, 1 + y, z)$, so generating by translation a $C(9)$ chain running parallel to the [010] direction. At the same time atom C35 in the molecule at (x, y, z) acts as a donor to the C21–C26 ring in the molecule at $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$, so forming a chain along [001] generated by the *c*-glide plane at $y = 0.25$; the combination of the chains along [010] and [001] generates a sheet parallel to (100) (Fig. 10). Two sheets of this type, generated by the *c*-glide planes at $y = \frac{1}{4}$ and $y = \frac{3}{4}$, and related to one another by inversion, pass through each unit cell and these pairs are linked into bilayers by a centrosymmetric π–π stacking interaction. The C31–C36 rings in the molecules at (x, y, z) and $(1 - x, -y, 1 - z)$ are strictly parallel with an interplanar spacing of 3.695 (2) Å; the ring-centroid separation is 3.862 (2) Å, corresponding to a ring-centroid offset of 1.125 (2) Å (Fig. 11).

The supramolecular structures described here show some marked variations consequent upon changes only in the location or identity of a single substituent common to the two aryl rings. This variation is particularly striking in the pairs of isomeric compounds (I) and (II), containing methyl substituents, and (I) and (IV), containing nitro substituents. Whereas compound (I), containing 2-methoxy substituents, adopts a structure exhibiting no direction-specific intermolecular interactions, the isomeric compound (III), containing 4-methoxy substituents, aggregates into a two-dimensional structure. On the other hand, compound (IV), containing 2-nitro substituents, has a two-dimensional structure, while the isomeric compound (II), containing 4-nitro substituents, has a supramolecular structure that is only one-dimensional.

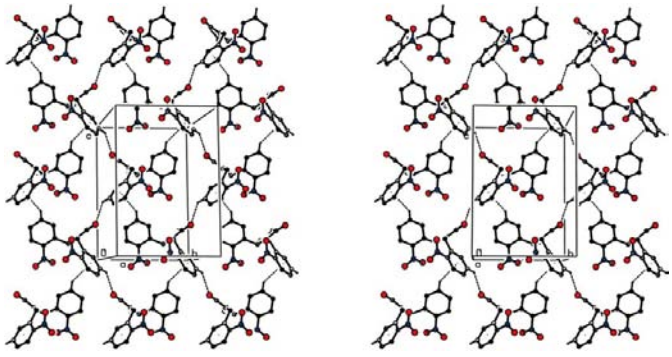

Figure 9
A stereoview of part of the crystal structure of compound (IV), showing the formation of a sheet parallel to (100) built from C—H···O and C—H···π(arene) hydrogen bonds. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

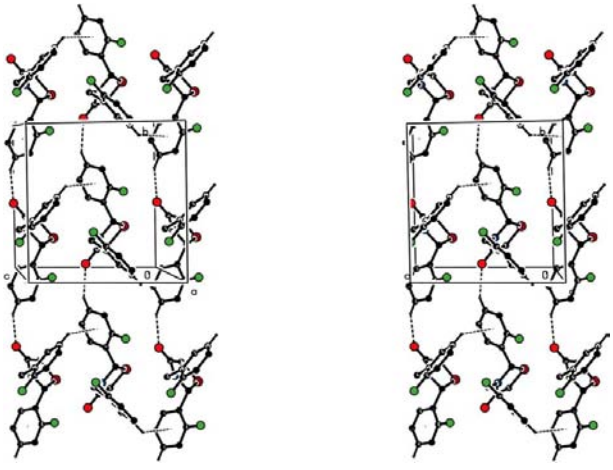

Figure 10
A stereoview of part of the crystal structure of compound (V), showing the formation of a sheet parallel to (100) built from C—H···O and C—H···π(arene) hydrogen bonds. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

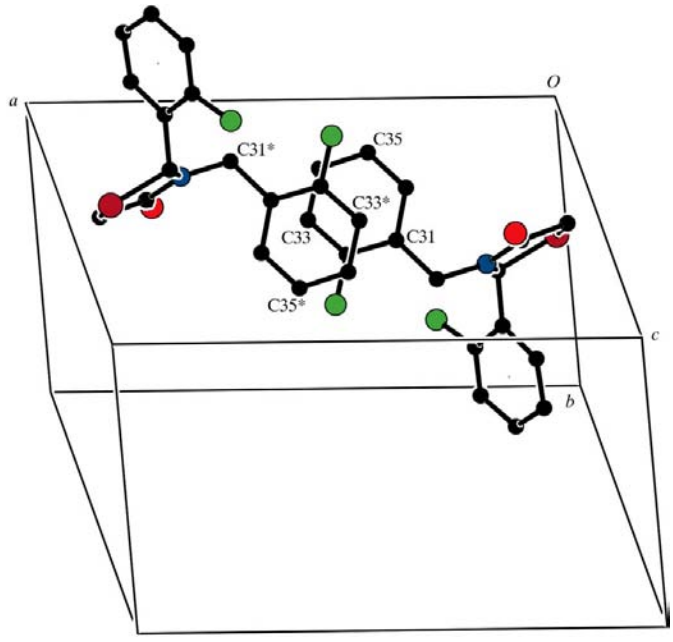

Figure 11
Part of the crystal structure of compound (V), showing the π–π stacking interaction that links pairs of sheets. For the sake of clarity, all H atoms have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1 - x, -y, 1 - z)$.

Experimental

Samples of compounds (I)–(V) were prepared according to a published procedure (Cunico *et al.*, 2006); crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of solutions in ethanol.

Compound (I)

Crystal data

$C_{18}H_{19}NO_3S$
$M_r = 329.40$
Monoclinic, $P2_1/n$
$a = 8.3321$ (2) Å
$b = 18.3668$ (4) Å
$c = 10.8568$ (2) Å
$\beta = 94.450$ (2)°
$V = 1656.45$ (6) Å^3
$Z = 4$
$D_x = 1.321$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.21$ mm^{-1}
$T = 120$ (2) K
Block, colourless
0.35 × 0.35 × 0.20 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.940, T_{max} = 0.959
17857 measured reflections
3643 independent reflections
3259 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.086
S = 1.04
3643 reflections
210 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0269P)^2 + 1.0914P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Compound (II)

Crystal data

$C_{16}H_{13}N_3O_5S$
M_r = 359.35
Monoclinic, $P2_1/c$
a = 14.2230 (7) Å
b = 7.9862 (4) Å
c = 14.1156 (7) Å
β = 93.908 (3)°
V = 1599.63 (14) Å^3
Z = 4
D_x = 1.492 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 120 (2) K
Plate, colourless
0.18 × 0.16 × 0.02 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.966, T_{max} = 0.995
3641 measured reflections
3641 independent reflections
2630 reflections with $I > 2\sigma(I)$
R_{int} = 0.0
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.122
$wR(F^2)$ = 0.438
S = 1.10
3641 reflections
227 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.35P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.01 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O241^{i}	1.00	2.37	3.263 (8)	148
C22—H22···O4^{ii}	0.95	2.45	3.210 (7)	137
C32—H32···O342^{iii}	0.95	2.52	3.355 (7)	147
C37—H37B···O342^{iii}	0.99	2.39	3.327 (7)	158

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

Compound (III)

Crystal data

$C_{18}H_{19}NO_3S$
M_r = 329.42
Monoclinic, $P2_1/c$
a = 4.6687 (4) Å
b = 9.6210 (8) Å
c = 35.478 (3) Å
β = 95.335 (3)°
V = 1586.7 (2) Å^3
Z = 4
D_x = 1.379 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 120 (2) K
Plate, colourless
0.32 × 0.15 × 0.07 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.942, T_{max} = 0.985
7975 measured reflections
3026 independent reflections
2470 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.239
S = 1.10
3026 reflections
210 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1489P)^2 + 1.6071P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

$Cg1$ is the centroid of the C31–C36 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C5—H52···O34^{i}	0.99	2.43	3.360 (5)	156
C37—H37A···$Cg1^{ii}$	0.99	2.81	3.525 (4)	129

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

Compound (IV)

Crystal data

$C_{16}H_{13}N_3O_5S$
M_r = 359.35
Monoclinic, $C2/c$
a = 22.3000 (4) Å
b = 9.9352 (3) Å
c = 14.6945 (4) Å
β = 96.435 (2)°
V = 3235.13 (14) Å^3
Z = 8
D_x = 1.476 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 120 (2) K
Block, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.929, T_{max} = 0.955
34307 measured reflections
3713 independent reflections
2956 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.096
S = 1.06
3713 reflections
226 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2 + 2.1203P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

$Cg1$ is the centroid of the C31–C36 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C24—H24···O4^{i}	0.95	2.38	3.2724 (18)	156
C26—H26···O31^{ii}	0.95	2.45	3.1985 (19)	135
C35—H35···O4^{iii}	0.95	2.46	3.1163 (18)	126
C25—H25···$Cg1^{iv}$	0.95	2.96	3.7624 (17)	143

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

Compound (V)

Crystal data

$C_{16}H_{13}F_2NOS$
M_r = 305.33
Monoclinic, $P2_1/c$
a = 13.9981 (6) Å
b = 10.1236 (3) Å
c = 10.1491 (4) Å
β = 106.333 (2)°
V = 1380.20 (9) Å^3
Z = 4
D_x = 1.469 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 120 (2) K
Plate, colourless
0.36 × 0.30 × 0.04 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.944, T_{max} = 0.990
15108 measured reflections
3123 independent reflections
2575 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.145
S = 1.05
3123 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 1.6351P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.93 e Å^{-3}

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

*Cg*2 is the centroid of the C21–C26 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C24—H24···O4^{i}	0.95	2.40	3.274 (3)	153
C35—H35···*Cg*2^{ii}	0.95	2.86	3.569 (3)	133

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

For compound (I), the space group $P2_1/n$ was uniquely assigned from the systematic absences; the space group $P2_1/c$ was similarly assigned for each of compounds (II), (III) and (V). For compound (IV), the systematic absences permitted *Cc* and *C*2/*c* as possible space groups; *C*2/*c* was selected and then confirmed by the structure analysis. All H atoms were located in difference maps and then treated as riding atoms, with C—H distances of 0.95 (aromatic), 0.98 (CH_3), 0.99 (CH_2) or 1.00 Å (aliphatic CH), and with U_{iso}(H) = kU_{eq}(C), where k = 1.5 for the methyl groups in (III) and k = 1.2 for all other H atoms. The structure of compound (II) indicated twinning. The TwinRotMAt procedure in *PLATON* (Spek, 2003) was used to generate a HKLF5 file containing 3641 reflections, with an estimated BASF value of 0.41; the final BASF value was 0.299 (7). Accordingly, the final R factors are high because of the lack of any averaging of equivalent reflections. It was apparent from an early stage that one of the F atoms in compound (V) was disordered over two sites, denoted F22 and F26; the final refined occupancies were 0.647 (4) and 0.353 (4).

For all compounds, data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG3061). Services for accessing these data are described at the back of the journal.

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