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Different hydrogen-bonded structures in three 2-thienyl-substituted tetrahydro-1,4-epoxy-1-benzazepines

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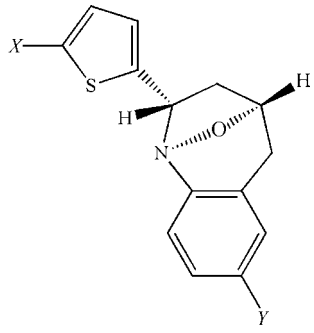
The molecules of (2*RS*,4*SR*)-2-*exo*-(5-bromo-2-thienyl)-7-chloro-2,3,4,5-tetrahydro-1*H*-1,4-epoxy-1-benzazepine, $C_{14}H_{11}BrClNOS$, (I), are linked into cyclic centrosymmetric dimers by C—H···π(thienyl) hydrogen bonds. Each such dimer makes rather short Br···Br contacts with two other dimers. In (2*RS*,4*SR*)-2-*exo*-(5-methyl-2-thienyl)-2,3,4,5-tetrahydro-1*H*-1,4-epoxy-1-benzazepine, $C_{15}H_{15}NOS$, (II), a combination of C—H···O and C—H···π(thienyl) hydrogen bonds links the molecules into chains of rings. A more complex chain of rings is formed in (2*RS*,4*SR*)-7-chloro-2-*exo*-(5-methyl-2-thienyl)-2,3,4,5-tetrahydro-1*H*-1,4-epoxy-1-benzazepine, $C_{15}H_{14}ClNOS$, (III), built from a combination of two independent C—H···O hydrogen bonds, one C—H···π(arene) hydrogen bond and one C—H···π(thienyl) hydrogen bond.

Comment

We report here the structures of three racemic thienyl-substituted tetrahydro-1,4-epoxy-1-benzazepines, namely (2*RS*,4*SR*)-2-*exo*-(5-bromo-2-thienyl)-7-chloro-2,3,4,5-tetrahydro-1*H*-1,4-epoxy-1-benzazepine, (I), (2*RS*,4*SR*)-2-*exo*-(5-methyl-2-thienyl)-2,3,4,5-tetrahydro-1*H*-1,4-epoxy-1-benzazepine, (II), and (2*RS*,4*SR*)-7-chloro-2-*exo*-(5-methyl-2-thienyl)-2,3,4,5-tetrahydro-1*H*-1,4-epoxy-1-benzazepine, (III) (Fig. 1), and we compare these structures with those of two close analogues, namely (2*RS*,4*SR*)-2-*exo*-(5-bromo-2-thienyl)-7-fluoro-2,3,4,5-tetrahydro-1*H*-1,4-epoxy-1-benzazepine, (IV), and (2*R*,4*S*)-2-*exo*-(5-bromo-2-thienyl)-7-trifluoromethoxy-2,3,4,5-tetrahydro-1*H*-1,4-epoxy-1-benzazepine, (V) (see scheme), the structures of which were reported recently (Blanco *et al.*, 2008). The present work is a continuation of a structural study of this class of epoxybenzazepines (Acosta *et al.*, 2008; Blanco *et al.*, 2008; Gómez *et al.*, 2008, 2009), which is itself part of a wider programme aimed at the identification of novel antiparasitic agents (Gómez *et al.*, 2006; Yépez *et al.*, 2006). The synthesis of compounds (I)–(III) followed the previously reported procedure (Acosta *et al.*, 2008), in which an appropriately N-substituted 2-allylaniline is oxidized with hydrogen peroxide in the presence of sodium tungstate to give a nitrone, which then undergoes an internal 1,3-dipolar cycloaddition to generate the tricyclic epoxybenzazepine product in satisfactory yield *via* a single-stage process.



(I) X = Br, Y = Cl
(II) X = Me, Y = H
(III) X = Me, Y = Cl
(IV) X = Br, Y = F
(V) X = Br, Y = OCF_3

While compound (I) differs from (IV) only in the identity of the halogen substituent in the fused aryl ring [Cl in (I) *versus* F in (IV)], these two compounds nonetheless crystallize in different space groups [*Pbca* for (I) and $P2_1/c$ for (IV)]. Similarly, although (II) and (III) differ only in the presence or absence of the Cl substituent in the fused aryl ring, again these two compounds crystallize in, respectively, space groups *Pbca* and $P2_1/c$. Although (I) and (II) crystallize in a common space group, as do (III) and (IV), within each pair the unit-cell dimensions differ substantially. Finally, while compounds (I)–(IV) all crystallize as racemic mixtures in centrosymmetric space groups, the crystals of (V) in space group $P2_1$ contain only one enantiomer. Hence, despite their close similarities, particularly for (I)–(IV), there are no isomorphisms between any pairs of these compounds.

In each of the racemic compounds (I)–(III), the reference molecules were selected to have the *S* configuration at atom C2, as for (IV) and (V) (Blanco *et al.*, 2008), and on this basis each of the reference molecules had configuration *R* at atom C4. The ring-puckering parameters (Cremer & Pople, 1975) in Table 1 show that the heterobicyclic systems in (I)–(V) all have very similar shapes, with the five- and six-membered rings all adopting conformations intermediate between envelope and half-chair forms. However, the orientations of the substituted 2-thienyl rings relative to the adjacent fused-ring system show some sharp variations. The orientation of this ring can conveniently be defined by the N1—C2—C22—S21 torsion angle, which takes the values 31.4 (4), 37.7 (3), −88.3 (2), −60.1 (3) and 36.6 (4)° in (I)–(V), respectively. Thus, the orientation of the 2-thienyl ring is similar in (I), (II)

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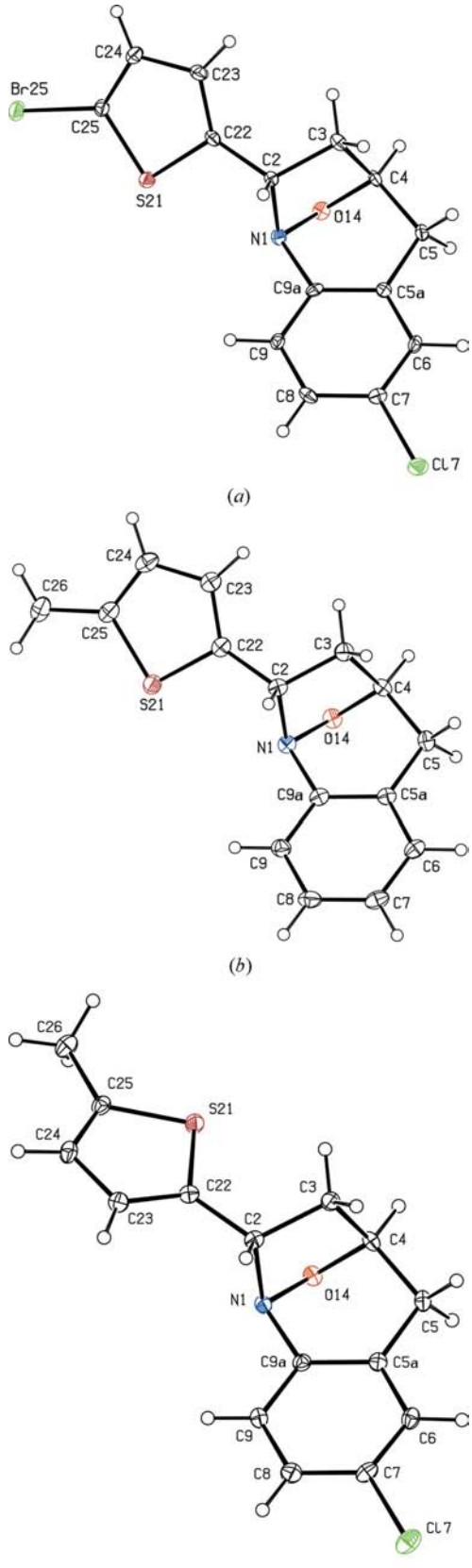

Figure 1
The molecular structures of the (2*S*,4*R*)-enantiomers of (*a*) compound (I), (*b*) compound (II) and (*c*) compound (III), showing the atom-labelling schemes. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

and (V), but in (III) and (IV) the orientation of this ring is almost orthogonal to that found in the first group. It is notable, however, that in none of compounds (I)–(V) does the substituted 2-thienyl ring exhibit any orientational disorder. While such disorder, usually characterized by a 180° rotation about the exocyclic C—C bond, here C22—C2, is not uncommon with unsubstituted 2-thienyl groups, it is possible that the steric requirement of the single substituent at position 5 of the thienyl ring effectively locks this ring into a single preferred orientation.

There are short C—H···Cl contacts in both (I) and (III) (Table 2), but these are not regarded as structurally significant because it has been well established that Cl atoms, when bound to C atoms, are extremely poor acceptors of hydrogen bonds, even from donors such as O or N (Aakeröy *et al.*, 1999; Brammer *et al.*, 2001; Thallapally & Nangia, 2001).

The supramolecular aggregation in (I) is very simple. Pairs of molecules related by inversion are linked by C—H···π(thienyl) hydrogen bonds (Table 2) to form cyclic dimers (Fig. 2). The only direction-specific interaction between adjacent dimers is a rather short Br···Br contact. Atoms Br25 in the molecules at (x, y, z) and ($1-x$, $2-y$, $-z$), which form parts of the cyclic dimers centred at ($\frac{1}{2}$, $\frac{1}{2}$, 0) and ($\frac{1}{2}$, $\frac{3}{2}$, 0), respectively, are separated by 3.5234 (7) Å, and the corresponding C—Br···Br angle is 143.9 (2)°. Studies (Ramasubbu *et al.*, 1986) of the preferred orientation of C—X···X—C contacts, where X represents a halogen other than F, based on data extracted from the Cambridge Structural Database in 1984 (CSD; Allen, 2002), have shown that such contacts fall into two main clusters, with C—X···X angles of *ca* 90 and 180°. The Br···Br contact in (I) thus deviates from this pattern. While the Br···Br distance is certainly less than twice the van der Waals radius estimated on the basis of a spherical atom (1.85 Å; Bondi, 1964), in terms of the polar flattening model (Nyburg & Faerman, 1985) the effective radius to be applied here appropriate for the observed C—Br···Br angle

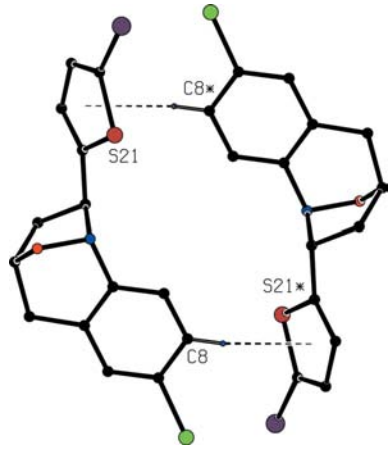

Figure 2
Part of the crystal structure of (I), showing the formation of a cyclic centrosymmetric dimer. For the sake of clarity, the unit-cell outline and H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position ($1-x$, $1-y$, $-z$).

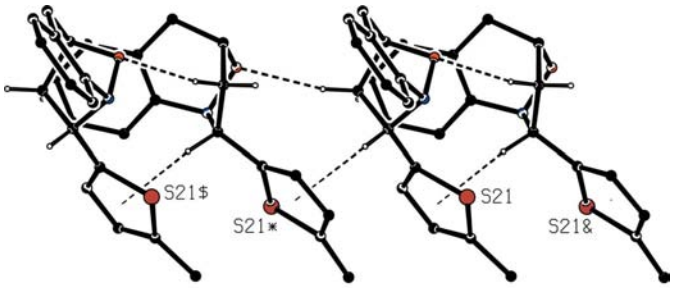

Figure 3
Part of the crystal structure of (II), showing the formation of a chain of rings parallel to [010] built from a combination of C—H···O and C—H···π(thienyl) hydrogen bonds. The [010] direction is vertical and the chain is viewed approximately along [100]. For the sake of clarity, the unit-cell outline and H atoms bonded to C atoms which are not involved in the motifs shown have been omitted. S atoms marked with an asterisk (*), a dollar sign ($) or an ampersand (&) are at the symmetry positions $(\frac{3}{2} - x, \frac{1}{2} + y, z)$, $(x, 1 + y, z)$ and $(\frac{3}{2} - x, -\frac{1}{2} + y, z)$, respectively.

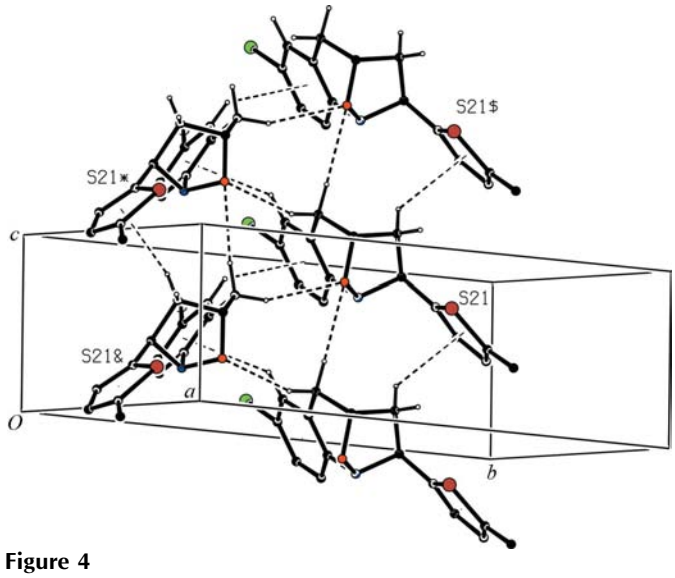

Figure 4
Part of the crystal structure of (III), showing the formation of a chain of rings parallel to [001] built from a combination of four independent hydrogen bonds. For the sake of clarity, H atoms bonded to C atoms which are not involved in the motifs shown have been omitted. S atoms marked with an asterisk (*), a dollar sign ($) or an ampersand (&) are at the symmetry positions $(x, \frac{1}{2} - y, \frac{1}{2} + z)$, $(x, y, 1 + z)$ and $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$, respectively.

lies approximately midway between the major and minor radii, which were estimated using database analysis (Nyburg & Faerman, 1985) for covalently bonded Br as 1.84 and 1.54 Å, respectively. On this basis, the observed Br···Br distance of 3.5234 (7) Å in (I) does not appear to be exceptional and it cannot be taken as evidence of any significant electrostatic attraction (Lommerse *et al.*, 1996; Bui *et al.*, 2009) between the dimers stacked along [010].

The dimer formation *via* hydrogen bonding in (I) may be contrasted with that in the very close analogue (IV) (see scheme). There are no hydrogen bonds of any kind in the crystal structure of (IV), but instead pairs of molecules are linked into centrosymmetric dimers by means of an aromatic π–π stacking interaction involving the fluoro-substituted aryl rings (Blanco *et al.*, 2008).

In the structure of (II), the co-operative action of C—H···O and C—H···π(thienyl) hydrogen bonds, one of each type (Table 2), links the molecules into a chain of rings. Atoms C2 and C3 in the molecule at (x, y, z) act as hydrogen-bond donors to, respectively, the thienyl ring and atom O14, both in the molecule at $(\frac{3}{2} - x, \frac{1}{2} + y, z)$. Hence, molecules related by the *b*-glide plane at $x = \frac{3}{4}$ are linked to form a chain of rings running parallel to the [010] direction (Fig. 3). Four chains of this type pass through each unit cell, in the domains $0.0 < z < \frac{1}{4}$, $\frac{1}{4} < z < \frac{1}{2}$, $\frac{1}{2} < z < \frac{3}{4}$ and $\frac{3}{4} < z < 1.0$, respectively, but there are no direction-specific interactions between the chains.

The chain formation in (III) is more complex than that in (II) because it is based on four independent hydrogen bonds, as opposed to just two in (II) (Table 2). Atoms C5 and C6 in the molecule at (x, y, z) act as hydrogen-bond donors *via* atoms H5*A* and H6 to, respectively, atom O14 and the fused aryl ring C5a/C6–C9/C9a, both in the molecule at $(x, \frac{1}{2} - y, \frac{1}{2} + z)$, so linking molecules related by the *c*-glide plane at $y = \frac{1}{4}$ into a chain running parallel to the [001] direction. At the same time, atoms C3 and C5 at (x, y, z) act as donors *via* H3*B* and H5*B* to, respectively, the thienyl ring and atom O14, both in the molecule at $(x, y, 1 + z)$. The combination of these two pairs of hydrogen bonds thus generates a complex chain of fused rings running parallel to the [001] direction, in which atom C5 acts as a double donor of hydrogen bonds and atom O14 as a double acceptor (Fig. 4).

It is striking that the hydrogen-bonded structure of (III) is only one-dimensional, despite the involvement of four independent hydrogen bonds. By contrast, in the structure of (V) (Blanco *et al.*, 2008), just two C—H···O hydrogen bonds are sufficient to generate a hydrogen-bonded structure in two dimensions.

Experimental

For the preparation of compounds (I)–(III), sodium tungstate dihydrate (5 mol%), followed by 30% aqueous hydrogen peroxide solution (12 mmol), were added to a stirred and cooled (273 K) solution of the appropriately substituted 2-allyl-*N*-(thienylmethyl)-aniline (4 mmol) in methanol (20 ml). The resulting mixtures were then stirred at ambient temperature for periods ranging from 18 to 20 h. Each mixture was filtered and the solvent was removed under reduced pressure. Toluene (30 ml) was added to the solid residues and the resulting solutions were heated under reflux for periods ranging from 6 to 8 h. After cooling each solution to ambient temperature, the solvent was removed under reduced pressure and the crude product was purified by chromatography on silica using heptane–ethyl acetate (compositions ranging from 60:1 to 10:1 *v*/*v*) as eluent. Crystallization from heptane gave colourless crystals of compounds (I)–(III) suitable for single-crystal X-ray diffraction. For compound (I): m.p. 358 K, yield 50%; MS (EI, 70 eV) *m*/*z* (%): 257 (M^+, 59), 240 (59), 227 (12), 124 (100), 105 (50), 104 (78). For compound (II): m.p. 398 K, yield 64%; MS (EI, 70 eV) *m*/*z* (%): 291 (M^+, ^{35}Cl, 27), 274 (21), 261 (5), 151 (8), 138 (40), 124 (100). For compound (III): m.p. 362 K, yield 64%; MS (EI, 70 eV) *m*/*z* (%): 355 (M^+, ^{35}Cl, ^{79}Br, 37), 338 (6), 325 (2), 188 (31), 164 (7), 139 (81), 138 (100).

Compound (I)

Crystal data

$C_{14}H_{11}BrClNOS$
M_r = 356.66
Orthorhombic, *Pbca*
a = 10.7959 (11) Å
b = 14.2116 (16) Å
c = 17.4303 (11) Å
V = 2674.3 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.42 mm^{-1}
T = 120 K
0.31 × 0.16 × 0.04 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.447, T_{max} = 0.875
31926 measured reflections
3068 independent reflections
1866 reflections with $I > 2\sigma(I)$
R_{int} = 0.160

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.076
S = 1.05
3068 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Compound (II)

Crystal data

$C_{15}H_{15}NOS$
M_r = 257.34
Orthorhombic, *Pbca*
a = 8.3789 (12) Å
b = 9.6964 (14) Å
c = 30.952 (4) Å
V = 2514.7 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 120 K
0.42 × 0.10 × 0.05 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.916, T_{max} = 0.988
21293 measured reflections
2874 independent reflections
1730 reflections with $I > 2\sigma(I)$
R_{int} = 0.078

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.121
S = 1.06
2874 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Compound (III)

Crystal data

$C_{15}H_{14}ClNOS$
M_r = 291.78
Monoclinic, $P2_1/c$
a = 15.042 (3) Å
b = 15.739 (3) Å
c = 5.6216 (13) Å
β = 100.120 (16)°
V = 1310.2 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 120 K
0.27 × 0.15 × 0.14 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.913, T_{max} = 0.940
19866 measured reflections
3002 independent reflections
2221 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.094
S = 1.07
3002 reflections
173 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Ring-puckering parameters (Å, °) for compounds (I)–(V).

Puckering parameters for five-membered rings are defined for the atom sequence O14—N1—C2—C3—C4 and those for six-membered rings for the atom sequence O14—N1—C9a—C5a—C5—C4.

Compound	Five-membered ring			Six-membered ring	
	Q_2	φ_2	Q	θ	φ
(I)	0.441 (4)	190.8 (6)	0.623 (4)	54.2 (4)	348.6 (5)
(II)	0.436 (2)	189.6 (3)	0.615 (2)	52.0 (2)	349.1 (3)
(III)	0.438 (2)	191.0 (3)	0.605 (2)	50.3 (2)	346.2 (3)
(IV)†	0.455 (3)	199.7 (4)	0.626 (3)	53.2 (3)	343.2 (4)
(V)†	0.447 (3)	197.2 (5)	0.623 (3)	54.1 (3)	347.0 (4)

† Data taken from Blanco *et al.* (2008).

Table 2
Hydrogen bonds and short intermolecular contacts (Å, °) for compounds (I)–(III).

*Cg*1 is the centroid of the S21/C22–C25 ring and *Cg*2 is the centroid of the C5a/C6–C9/C9a ring.

Compound	D—H···A	D—H	H···A	D···A	D—H···A
(I)	C4—H4···Cl7^{i}	1.00	2.78	3.497 (4)	129
	C8—H8···*Cg*1^{ii}	0.95	2.97	3.573 (5)	132
(II)	C2—H2···*Cg*1^{iii}	1.00	2.59	3.570 (2)	167
	C3—H3*B*···O14^{iii}	0.99	2.55	3.527 (3)	169
(III)	C2—H2···Cl7^{iv}	1.00	2.80	3.742 (4)	156
	C3—H3*B*···*Cg*1^{v}	0.99	2.70	3.372 (3)	125
	C5—H5*A*···O14^{vi}	0.99	2.58	3.518 (3)	158
	C5—H5*B*···O14^{v}	0.99	2.55	3.530 (3)	173
	C6—H6···*Cg*2^{vi}	0.95	2.92	3.689 (3)	139

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

All H atoms were located in difference maps and then treated as riding atoms in geometrically idealized positions, with C—H = 0.95 (aromatic and heteroaromatic), 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, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. For each compound, the configuration at C2 was set to be *S* in the reference molecule, and on that basis the configurations at C4 in the reference molecules are all *R*.

For all three compounds, data collection: *COLLECT* (Nonius, 1999); cell refinement: *DIRAX*/*LSQ* (Duisenberg *et al.*, 2000); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG3212). Services for accessing these data are described at the back of the journal.

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