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Dimethyl 3,4,5,5-tetraphenyl-1,3-thiazolidine-2,2-dicarboxylate and 3,3-dichloro-2,2,4,4,3′-pentamethyl-*r*-2′,*t*-4′-diphenylcyclobutane-1-spiro-5′-1,3-thiazolidine

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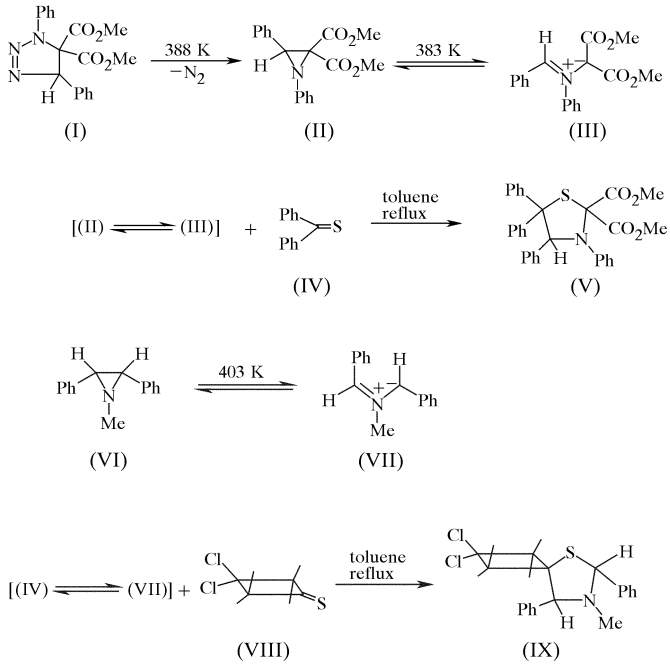
The first of the title compounds, $C_{31}H_{27}NO_4S$, (V), crystallizes in the monoclinic space group $P2_1/c$ with two independent molecules in the asymmetric unit, while the second, $C_{23}H_{27}Cl_2NS$, (IX), crystallizes in the orthorhombic space group *Pbca* with one molecule in the asymmetric unit. In both crystal structures, the 1,3-thiazolidine ring adopts a half-chair conformation. The crystal structures are stabilized by weak C—H···O and C—H···Cl hydrogen bonds in (V) and (IX), respectively.

Comment

1,3-Thiazolidines are known to exhibit biological activity (Hwu *et al.*, 1999; Pellegrini *et al.*, 1999) and have also been explored as valuable starting materials for the preparation of more complex structures (Bringmann *et al.*, 2000; Jin & Kim, 2002). The title compounds, (V) and (IX), were obtained by the [3+2]-dipolar cycloaddition of an azomethine ylide, generated *in situ* by thermal ring-opening of the appropriate aziridine in the presence of thiobenzophenone, (IV), or 3,3-dichloro-2,2,4,4-tetramethylcyclobutanethione, (VIII) (see scheme), according to the general protocol of Mlostoń & Skrzypek (1990).

Sometimes, desulfurization of 1,3-thiazolidines with Raney nickel results unexpectedly in ring contraction and the formation of the corresponding azetidine derivative (Mlostoń, Urbaniak & Heimgartner, 2002; Mlostoń, Urbaniak *et al.*, 2002; Urbaniak *et al.*, 2004). For this reason, our studies of this group of compounds concern not only the elucidation of their structures, conformations and configurations, but also the determination of the influence of the location of the ester groups on the course of the desulfurization reaction. Detailed studies of 1,3-thiazolidine derivatives show that the regioselectivity of the [3+2]-cycloaddition leading to the formation of the five-membered heterocyclic ring is dependent on the type of thioketone used (Domagała, Linden *et al.*, 2003).



(I) (II) (III) (IV) (V) (VI) (VII) (VIII) (IX)

Another aim of the present work was an analysis of the hydrogen bonding, one of the most important interactions influencing the arrangement of molecules in molecular organic crystals (Desiraju, 1989; Jeffrey & Saenger, 1994; Desiraju & Steiner, 1999). Studies of hydrogen-bond interactions have shown that the H-atom-donating and -accepting abilities of molecules determine the architecture of crystals. In recent years, the role of C—H···*X* hydrogen bonds in crystal engineering has been extensively studied. Among C—H···*X* interactions, the C—H···O type is most often investigated because it occurs most frequently in crystals. For the crystal structures reported here, not only C—H···O interactions occur but also C—H···S and C—H···N ones, which are not well known because of their rare occurrence in crystals (Taylor & Kennard, 1982; Desiraju, 1995; Desiraju & Steiner, 1999). The contacts mentioned above have been investigated previously, both theoretically and experimentally (Domagała, Grabowski *et al.*, 2003, 2004). C—H···π contacts are another type of interaction investigated here for the crystal structures of (V) and (IX). Such interactions often have an influence on the crystal packing (Ciunik *et al.*, 1998; Ciunik & Jarosz, 1998).

The asymmetric unit of compound (V) contains two independent molecules (denoted *A* and *B*), which have the same five-membered heterocyclic ring conformation and selected to have opposite absolute configurations. The chiral centre is at atom C4. In Fig. 1, the phenyl substituent is attached in the *S* configuration in molecule *A*, whereas in molecule *B* this configuration is *R*. Compound (IX) (Fig. 2) crystallizes with one molecule in the asymmetric unit. There are two centres of opposite chirality in this molecule, at atoms C2 and C4.

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The bond lengths and angles in the title structures (Tables 1 and 3) are generally in good agreement with expected values (Allen *et al.*, 1987). However, in (V), there is a slight elongation of the C4*A*—C5*A* and C4*B*—C5*B* bonds in comparison with the expected values (both *ca* 1.56 Å), and the C—S bonds are asymmetric, similar to what was observed in our previously reported structures. C—C bond lengths in the range 1.56–1.61 Å have been observed frequently in spirocyclic thiazolidine, thiazole, oxathiolane and dithiolane ring systems (Linden *et al.*, 1998; Domagała, Linden *et al.*, 2003; Domagała, Grabowski *et al.*, 2004). Moreover, the C2*A*—N3*A*—C4*A* and C2*B*—N3*B*—C4*B* bond angles are larger then the corresponding angle in compound (IX). Steric hindrance is probably responsible for the observed geometry of the thiazolidine ring. In both compounds, the 1,3-thiazolidine rings adopt half-chair conformations.

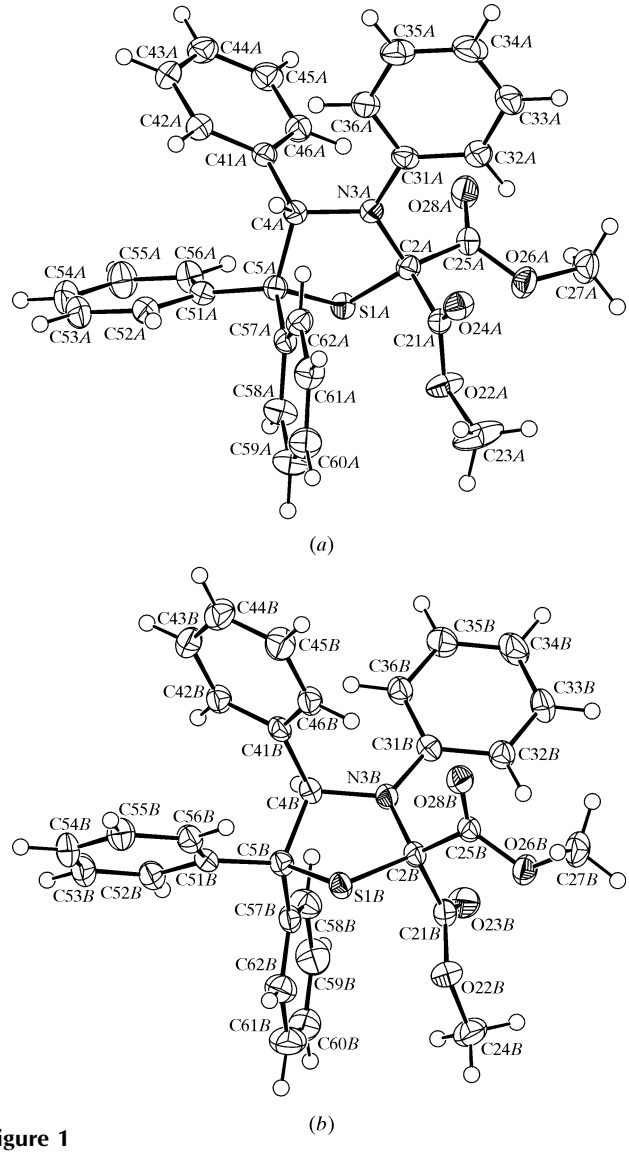

Figure 1
A view of the two symmetry-independent 1,3-thiazolidine molecules of (V), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by small spheres of arbitrary radii.

The puckering parameters (Cremer & Pople, 1975) for (V), for the atom sequence C5/S1/C2/N3/C4, are q_2 = 0.5439 (14) Å and φ_2 = −161.4 (1)°, and q_2 = 0.5355 (15) Å and φ_2 = 18.4 (2)° for molecules *A* and *B*, respectively, with pseudo-twofold axes passing through atom N3 and the mid-point of the C5—S1 bond [asymmetry parameters (Nardelli, 1983) Δ = 0.0135 (6) for molecule *A* and 0.0127 (6) for molecule *B*]. For compound (IX), the puckering parameters for the same sequence of atoms are q_2 = 0.5000 (16) Å and φ_2 = −17.2 (2)°. The pseudo-twofold axis passes through atom S1 and the mid-point of the N3—C4 bond [Δ = 0.0050 (7)]. In both compounds, the substituents attached at N3 are nearly in the plane of the N3/C2/S1/C5/C4 ring, whereas those at C4 occupy pseudo-axial positions. In compound (V), the planes of the phenyl rings at C4, as well as of those at C2 and C4 in (IX), lie almost perpendicular to the mean planes of the 1,3-thiazolidine rings, with interplanar angles of 85.9 (1) and 88.6 (1)° for molecules *A* and *B*, respectively, in (V), and 79.3 (1) and 89.1 (1)°, respectively, in (IX). In contrast, the planes of the phenyl substituents at N3*A* and N3*B* of (V) lie almost parallel to the planes of the respective 1,3-thiazolidine rings, with interplanar angles of 16.1 (1) and 19.6 (1)°, respectively. In compound (IX), the chloro-substituted cyclobutane ring is nearly perpendicular to the central 1,3-thiazolidine ring; the angle between these planes is 85.59 (5)°. This four-membered ring is more puckered than that in a previously reported structure (Domagała, Małecka *et al.*, 2004), where there was a carboxyl group instead of Cl atoms. The puckering parameter for the C54/C56/C5/C51 sequence of atoms is q_2 = 0.209 (2) Å and the pseudo-mirror plane passes through atoms C54 and C5 [Δ = 0.0002 (11)]. The general rule for predicting the conformation of cyclobutane compounds in the solid state (Margulis, 1969; Adman & Margulis, 1969) would suggest that such ring puckering is connected with the asymmetric arrangement of the substituents. The internal bond lengths and angles are consistent with those previously reported (Shirrell & Williams,

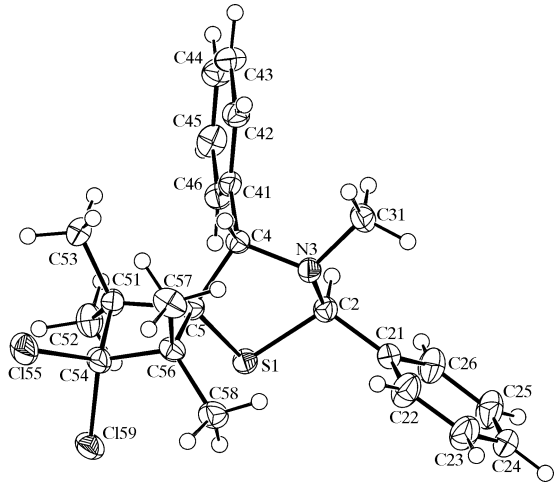

Figure 2
A view of the molecule of (IX), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by small spheres of arbitrary radii.

1973, 1974). It is worth mentioning that the C54—C51 [1.564 (2) Å] and C54—C56 [1.551 (2) Å] bond lengths are significantly shorter than those on the spiro side of the ring [C5—C51 = 1.585 (2) Å and C5—C56 = 1.592 (2) Å], and consequently the C51—C54—C56 angle is the largest [92.3 (1)°] of the C—C—C angles. Presumably, this is a result of the interaction between the cyclobutane and 1,3-thiazolidine rings.

Because of the attached substituents on the 1,3-thiazolidine ring, the title compounds have slightly different abilities to form hydrogen bonds. The criterion of H···*A* distances shorter than the corresponding sum of the van der Waals radii was applied as the definition of hydrogen bonds. Geometric parameters for possible hydrogen bonds in (V) and (IX) are given in Tables 2 and 4, respectively. In both compounds, the molecular structures are stabilized by intramolecular C—H···S and C—H···N interactions. These interactions were the subject of our previous experimental and theoretical investigations on the group of 1,3-thiazolidine derivatives (Domagała, Grabowski *et al.*, 2003, 2004).

The use of the Bader theory (Bader, 1990) has shown that only C—H···S interactions fulfil not only the geometric but also the topological criteria for the existence of hydrogen bonds. The most reasonable explanation for the lack of the topological confirmation of C—H···N interactions could be too small a C—H—N angle, in spite of an appropriate H-atom-acceptor distance. Nevertheless, it is important to take into consideration such interactions and their influence on molecular structures (Desiraju & Steiner, 1999).

The presence of carboxymethyl groups in (V) gives another type of intramolecular contact stabilizing the molecular structure, namely C—H···O interactions. Among all intramolecular C—H···O interactions, two clearly stand out, these being the C46—H46···O28 interactions in molecules *A* and *B*. The length of this H···O contact is distinctly shorter, by about 0.2 Å, than the remaining intramolecular contacts, and, more importantly, the C—H···O angle is clearly near linearity, at 166°. As is known, C—H···O interactions are mostly electrostatic in nature, so a geometry close to linear favours the existence of these hydrogen bonds. In compound (IX), atom C58 is involved in two intramolecular interactions, *viz*. C58—H58···Cl59 and C58—H58···S1.

While intramolecular hydrogen bonds play a crucial role in stabilizing molecular structures, the most important interactions responsible for the architecture of crystals are intermolecular interactions. There are many C—H···O contacts for compound (V). Among these, the C27*A*—H27*A*···O24^{i} contact differs in geometry from the others in that the H···O distance is the shortest, at 2.37 Å, and the angle is the smallest, at 108° [symmetry code: (i) $-x$, $y-\frac{1}{2}$, $\frac{3}{2}-z$]. It is worth mentioning that atom O28*B* takes part in three hydrogen bonds: as an acceptor, it is involved in two intermolecular hydrogen bonds, connecting two symmetrically independent molecules *A* and *B* [C42*A*—H42*A*···O28*B* and C44*A*—H44*A*···O28*B*^{ii}; symmetry code: (ii) $1-x$, $y-\frac{1}{2}$, $\frac{3}{2}-z$], and in one intramolecular hydrogen bond (C46*B*—H46*B*···O28*B*). What is more, the crystal packing is also stabilized by C—H···π interactions, C23*B*—H234···*Cg*1^{iv} [*Cg*1 denotes the centroid of the phenyl ring attached at N3*A*; symmetry code: (iv) x, $\frac{1}{2}-y$, $z-\frac{1}{2}$] (Ciunik *et al.*, 1998; Ciunik & Jarosz, 1998; Desiraju & Steiner, 1999). In compound (IX), just as in compound (V), a stabilizing C23—H23···*Cg*2^{ii} interaction is observed (*Cg*2 denotes the centroid of the phenyl ring attached at C4). However, the most significant intermolecular connections in (IX) are C—H···Cl interactions, which hold the molecules together to form dimers (Fig. 3) of graph-set motif $R_2^2(10)$ (Bernstein *et al.*, 1995).

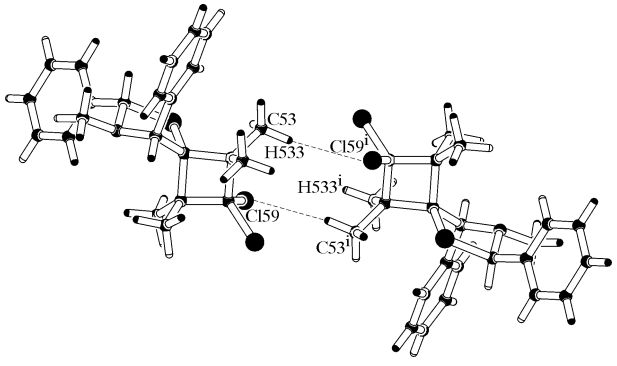

Figure 3
A view of the dimer formed by the intermolecular C—H···Cl interactions (dashed lines) in the structure of (IX) [symmetry code: (i) $-x$, $-y$, $-z$].

Experimental

Compounds (V) and (IX) were obtained by [3+2]-dipolar cycloaddition of the corresponding azomethine ylide, generated *in situ* by thermal ring-opening of dimethyl 1,3-diphenylaziridine-2,2-dicarboxylate with thiobenzophenone for (V) or of 1,3-diphenyl-2-methylaziridine with 3,3-dichloro-2,2,4,4-tetramethylcyclobutanethione for (IX) according to the general procedures of Mlostoń & Skrzypek (1990) and Urbaniak *et al.* (2004). Both compounds were characterized by elemental analysis, and IR and NMR spectroscopy. Crystals suitable for X-ray crystallography were obtained by slow evaporation from methanol and dichloromethane solutions of the compounds at room temperature [m.p. 426–428 K for (V) and 467–469 K for (IX)].

Compound (V)

Crystal data

$C_{31}H_{27}NO_4S$
M_r = 509.6
Monoclinic, $P2_1/c$
a = 19.4758 (9) Å
b = 12.2344 (6) Å
c = 22.1493 (10) Å
β = 100.380 (5)°
V = 5191.2 (4) Å^3
Z = 8
D_x = 1.304 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8000 reflections
θ = 2.4–25.9°
μ = 0.16 mm^{-1}
T = 173 (2) K
Cut block, colourless
0.4 × 0.3 × 0.3 mm

Data collection

Stoe IPDS diffractometer
Rotation scans
35 574 measured reflections
9960 independent reflections
7484 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 25.9°
$h = -23 \rightarrow 23$
$k = -14 \rightarrow 14$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R(F) = 0.037$
$wR(F^2) = 0.092$
$S = 0.91$
9960 reflections
671 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (V).

S1*A*—C2*A*	1.827 (2)	S1*B*—C2*B*	1.834 (1)
S1*A*—C5*A*	1.852 (2)	S1*B*—C5*B*	1.846 (2)
N3*A*—C2*A*	1.456 (2)	N3*B*—C2*B*	1.454 (2)
N3*A*—C4*A*	1.468 (2)	N3*B*—C4*B*	1.472 (2)
C4*A*—C5*A*	1.556 (2)	C4*B*—C5*B*	1.562 (2)
C2*A*—S1*A*—C5*A*	89.1 (1)	C2*B*—S1*B*—C5*B*	89.8 (1)
C2*A*—N3*A*—C4*A*	116.3 (1)	C2*B*—N3*B*—C4*B*	117.0 (1)
N3*A*—C2*A*—S1*A*	104.6 (1)	N3*B*—C2*B*—S1*B*	104.0 (1)
N3*A*—C4*A*—C5*A*	105.8 (1)	N3*B*—C4*B*—C5*B*	105.6 (1)
C4*A*—C5*A*—S1*A*	101.1 (1)	C4*B*—C5*B*—S1*B*	101.3 (1)
C2*A*—N3*A*—C31*A*—C36*A*	179.6 (1)	C2*B*—N3*B*—C31*B*—C36*B*	−175.8 (1)
N3*A*—C4*A*—C41*A*—C46*A*	32.5 (2)	N3*B*—C4*B*—C41*B*—C46*B*	−30.0 (2)

Table 2
Hydrogen-bonding geometry (Å, °) for (V).

*Cg*1 is the centroid of the phenyl ring attached at N3*A*.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C32*A*—H32*A*···O24*A*	0.95	2.62	2.993 (2)	104
C46*A*—H46*A*···O28*A*	0.95	2.49	3.413 (2)	166
C46*A*—H46*A*···N3*A*	0.95	2.68	2.958 (2)	98
C56*A*—H56*A*···S1*A*	0.95	2.66	3.060 (2)	106
C32*B*—H32*B*···O26*B*	0.95	2.57	3.392 (2)	145
C32*B*—H32*B*···O24*B*	0.95	2.60	3.001 (2)	106
C46*B*—H46*B*···O28*B*	0.95	2.40	3.328 (2)	166
C46*B*—H46*B*···N3*B*	0.95	2.71	2.980 (2)	97
C56*B*—H56*B*···S1*B*	0.95	2.66	3.060 (2)	106
C42*A*—H42*A*···O28*B*	0.95	2.65	3.366 (2)	133
C27*A*—H271···O24*A*^{i}	0.98	2.37	2.832 (2)	108
C44*A*—H44*A*···O28*B*^{ii}	0.95	2.53	3.351 (2)	145
C43*B*—H43*B*···O28*A*^{iii}	0.95	2.68	3.358 (2)	128
C23*B*—H234···*Cg*1^{iv}	0.98	2.79	3.743 (2)	165

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

Compound (IX)

Crystal data

$C_{23}H_{27}Cl_2NS$
$M_r = 420.42$
Orthorhombic, *Pbca*
$a = 11.3371$ (8) Å
$b = 12.7769$ (9) Å
$c = 29.1481$ (18) Å
$V = 4222.2$ (5) Å^3
$Z = 8$
$D_x = 1.323$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8000 reflections
$\theta = 2.4$–25.9°
$\mu = 0.42$ mm^{-1}
$T = 173$ (2) K
Prism, colourless
0.5 × 0.5 × 0.4 mm

Data collection

Stoe IPDS diffractometer
Rotation scans
32 423 measured reflections
4063 independent reflections
3125 reflections with $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 25.9°$
$h = -13 \rightarrow 13$
$k = -15 \rightarrow 15$
$l = -35 \rightarrow 35$

Refinement

Refinement on F^2
$R(F) = 0.033$
$wR(F^2) = 0.080$
$S = 0.92$
4063 reflections
249 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Table 3
Selected geometric parameters (Å, °) for (IX).

S1—C5	1.842 (2)	N3—C4	1.478 (2)
S1—C2	1.845 (2)	C4—C5	1.543 (2)
N3—C2	1.462 (2)		
C5—S1—C2	93.6 (1)	N3—C4—C5	103.1 (1)
C2—N3—C4	107.8 (1)	C4—C5—S1	103.2 (1)
N3—C2—S1	103.0 (1)		
S1—C2—C21—C26	98.8 (2)	N3—C4—C41—C46	85.2 (2)

Table 4
Hydrogen-bonding geometry (Å, °) for (IX).

*Cg*2 is the centroid of the phenyl ring attached at C4.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C22—H22···N3	0.95	2.67	2.941 (3)	97
C46—H46···S1	0.95	2.70	3.362 (2)	127
C53—H531···S1	0.98	2.68	3.138 (2)	109
C58—H582···S1	0.98	2.66	3.105 (2)	108
C58—H582···Cl59	0.98	2.65	2.987 (2)	100
C53—H533···Cl59^{i}	0.98	2.88	3.844 (2)	167
C23—H23···*Cg*2^{ii}	0.95	2.89	3.778 (2)	157

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

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å. For methoxy H atoms, $U_{iso}(H) = 1.5U_{eq}(C)$; for all other H atoms, $U_{iso}(H) = 1.2U_{eq}(C)$.

For both compounds, data collection: *IPDS* (Stoe, 1998); cell refinement: *IPDS*; data reduction: *IPDS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990); software used to prepare material for publication: *PARST* (Nardelli, 1996).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1671). Services for accessing these data are described at the back of the journal.

References

Adman, E. & Margulis, T. N. (1969). *J. Phys. Chem.* **73**, 1480–1484.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–83.
Bader, R. F. W. (1990). *Atoms in Molecules, a Quantum Theory*. New York: Oxford University Press Inc.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bringmann, G., Feineis, D., Saeb, W., Hesselmann, Ch., Peters, E.-M. & Peters, K. (2000). *Z. Naturforsch. Teil B*, **55**, 208–212.
Ciunik, Z., Berski, S., Latajka, Z. & Leszczynski, J. (1998). *J. Mol. Struct.* **442**, 125–134.
Ciunik, Z. & Jarosz, S. (1998). *J. Mol. Struct.* **442**, 115–119.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Desiraju, G. R. (1989). *Crystal Engineering – The Design of Organic Solids.* Amsterdam: Elsevier.
Desiraju, G. R. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 2311–2327.
Desiraju, G. R. & Steiner, T. (1999). *The Weak Hydrogen Bond in Structural Chemistry and Biology*. New York: Oxford University Press Inc.
Domagała, M., Grabowski, S. J., Urbaniak, K. & Mlostoń, G. (2003). *J. Phys. Chem. A*, **107**, 2730–2736.
Domagała, M., Grabowski, S. J., Urbaniak, K. & Mlostoń, G. (2004). *J. Mol. Struct.* **690**, 69–75.
Domagała, M., Linden, A., Olszak, T. A., Mlostoń, G. & Heimgartner, H. (2003). *Acta Cryst.* C**59**, o250–o253.
Domagała, M., Małecka, M., Urbaniak, K., Mlostoń, G. & Grabowski, S. J. (2004). *Struct. Chem.* Submitted.
Hwu, J. R., Hakimelahi, S., Moosavi-Movahedi, A. A. & Tsay, S.-C. (1999). *Chem. Eur. J.* **5**, 2705–2711.
Jeffrey, G. A. & Saenger, W. (1994). *Hydrogen Bonding in Biological Structures*. Berlin: Springer–Verlag.
Jin, M.-J. & Kim, S.-H. (2002). *Bull. Korean Chem. Soc.* **23**, 509–510.
Linden, A., Bojkova, N. & Heimgartner, H. (1998). *Acta Cryst.* C**54**, 373–376.
Margulis, T. N. (1969). *Chem. Commun.* pp. 215–216.
Mlostoń, G. & Skrzypek, Z. (1990). *Bull. Soc. Chim. Belg.* **99**, 167–170.
Mlostoń, G., Urbaniak, K. & Heimgartner, H. (2002). *Helv. Chim. Acta*, **85**, 2056–2064.
Mlostoń, G., Urbaniak, K., Linden, A. & Heimgartner, H. (2002). *Helv. Chim. Acta*, **85**, 2644–2656.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1996). *J. Appl. Cryst.* **29**, 296–300.
Pellegrini, N., Refouvelet, B., Crini, G., Blacque, O., Kubicki, M. M. & Robert, J.-F. (1999). *Chem. Pharm. Bull.* **47**, 950–955.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Shirrell, C. D. & Williams, D. E. (1973). *Acta Cryst*. B**29**, 1648–1653.
Shirrell, C. D. & Williams, D. E. (1974). *Acta Cryst*. B**30**, 1974–1978.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Stoe & Cie (1998). *IPDS*. Version 2.89. Stoe & Cie, Darmstadt, Germany.
Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.
Urbaniak, K., Szymański, R., Romański, J., Mlostoń, G., Domagała, M., Linden, A. & Heimgartner, H. (2004). *Helv. Chim. Acta*, **87**, 496–510.