Table 1. Selected geometric parameters (Å, °)

Br4—C4	1.897 (2)	O14—C14	1.372 (4)
S6—C6	1.674 (2)	O14—C16	1.412 (4)
O11—C11	1.368 (4)	N2—C6	1.375 (3)
O11—C15	1.438 (4)	N3—C6	1.318 (3)
C11—O11—C15	117.2 (3)	N3—C6—S6	122.9 (2)
C14—O14—C16	118.7 (3)	N2—C6—S6	119.60 (19)
N3—C6—N2	117.5 (2)		
N3—C7—C8—C9	65.7 (3)	C16—O14—C14—C13	15.4 (4)
C15—O11—C11—C12	0.1 (5)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···S6[i]	0.81 (2)	2.62 (3)	3.403 (2)	165 (2)
N3—H3A···N1	0.84 (2)	1.98 (3)	2.671 (3)	138 (2)

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

H atoms were placed at ideal positions and refined as riding atoms with relative isotropic displacement parameters except for H2A and H3A (Fig. 1), which were located in the electron-density difference map and refined isotropically.

Data collection: *SMART* (Bruker, 1998a). Cell refinement: *SAINT* (Bruker, 1998a). Data reduction: *SHELXTL* (Bruker, 1998b). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bernstein, J., Etter, M. C. & MacDonald, J. C. (1990). *J. Chem. Soc. Perkin Trans.* 2, pp. 695–698.

Bruker (1998a). *SMART* and *SAINT. Area Detector Control and Integration Software.* Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (1998b). *SHELXTL/NT Reference Manual.* Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.

Etter, M. C. (1991). *J. Phys. Chem.* **95**, 4601–4610.

Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B46, 256–262.

Johnson, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Mao, C., Sudbeck, E. A., Venkatachalam, T. K. & Uckun, F. M. (1999). *Bioorg. Med. Chem. Lett.* **9**, 1593–1598.

Mao, C., Vig, R., Venkatachalam, T. K., Sudbeck, E. A. & Uckun, F. M. (1998). *Bioorg. Med. Chem. Lett.* **8**, 2213–2218.

Sheldrick, G. M. (1990). *Acta Cryst.* A46, 467–473.

Sheldrick, G. M. (1996). *SADABS. Program for Empirical Absorption Correction of Area Detector Data.* University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Sudbeck, E. A., Mao, C., Vig, R., Venkatachalam, T. K., Tuel-Ahlgren, L. & Uckun, F. M. (1998). *Antimicrob. Agents Chemother.* **42**, 3225–3233.

Vig, R., Mao, C., Venkatachalam, T. K., Tuel-Ahlgren, L., Sudbeck, E. A. & Uckun, F. M. (1998a). *Bioorg. Med. Chem. Lett.* **8**, 1461–1466.

Vig, R., Mao, C., Venkatachalam, T. K., Tuel-Ahlgren, L., Sudbeck, E. A. & Uckun, F. M. (1998b). *Bioorg. Med. Chem.* **6**, 1789–1797.

rac-(3R,5S)-5-(1-Cyclopropyl-1-methylethyl)-3,5-diphenyl-2,3,4,5-tetrahydrofuran-2-one, a triclinic structure with local monoclinic pseudosymmetry

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Abstract

The crystal packing in the title compound, $C_{22}H_{24}O_2$, shows layers with pseudo-monoclinic symmetry. The two independent molecules are related by local $2_1/b$ pseudosymmetry. Adjacent layers are displaced by a distance of about 1.17 Å in the a direction, resulting in an overall triclinic symmetry of the structure. The packing of the layers shows intermolecular C—H···π(arene) interactions.

Comment

The structure of the title compound, (I), contains two crystallographically independent molecules. Both mol-

(I)
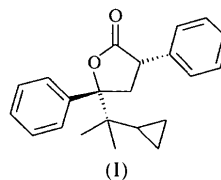
ecules have very similar conformations. Corresponding torsion angles of the molecules differ by no more than 6°. The two independent molecules (Fig. 1) are related by a non-crystallographic glide plane perpendicular to the a axis, with a translation of $b/2$. The equation of the plane expressed in crystal coordinates is approximately: $8.430x + 0.347y + 1.236z = 2.764$. The angle between this plane and the b axis is 1.4°. Thus, the pseudo-glide plane is almost continuous in the crystallographic b direction. In the c direction, however, the pseudo-glide plane only acts in one unit cell and shows a shift of about 1.17 Å along a between adjacent cells (Fig. 2). The pseudo-glide symmetry can be combined with the crystallographic inversion center at $(0,0,\frac{1}{2})$ to give an additional pseudo-twofold screw axis parallel

to **a**, displaced from the origin by $\pm b/4$ and $c/2$. These pseudosymmetry elements can be identified easily using the program *BUNYIP* (Hester & Hall, 1996) or by a combined coordinate midpoint and translation analysis as described by Kálmán & Argay (1998). Thus, the structure of (I) contains pseudo-monoclinic layers parallel to the *ab* plane. The symmetry of the layer corresponds to structural class $P_{l(xy)}2_1/b$, $Z =$ 4 (1), using the nomenclature of Zorky (1996). Adjacent layers are displaced by a distance of 1.17 Å along the *a* axis. This displacement results in a triclinic symmetry of the three-dimensional crystal structure. Bonding within the layer consists of intermolecular electrostatic interactions between keto-O atoms and phenyl-H atoms of symmetry-related molecules, with O···H distances in the range 2.39 (3)–2.48 (3) Å.

Intermolecular interactions between the H atoms of the cyclopropyl groups and the π systems of phenyl groups, with H···Cg distances in the range 2.86–3.03 Å, complete the bonding within the layers (Cg represents a ring centroid). The packing of neighboring layers shows one significant intermolecular interaction, *i.e.* a
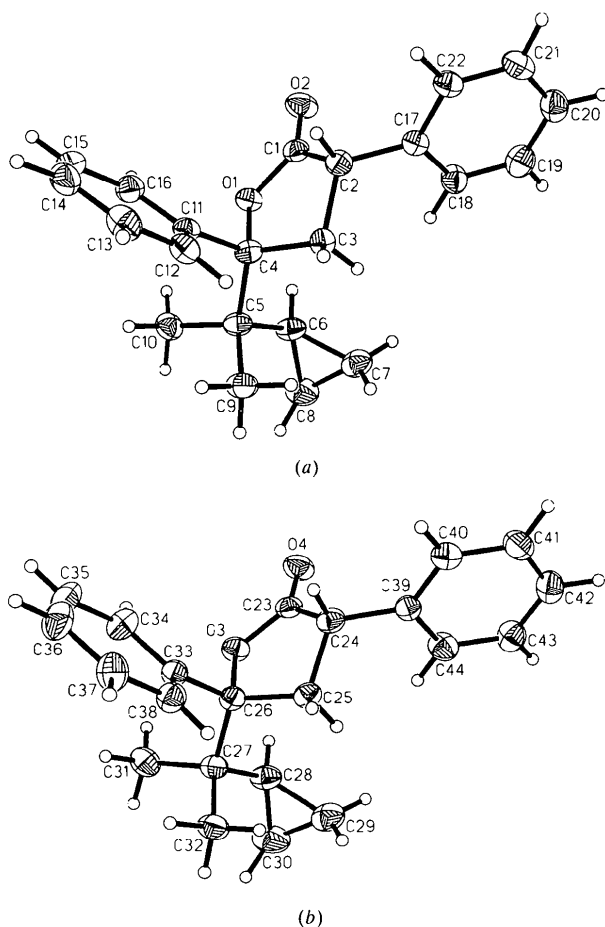
C36—H36···Cg1 contact (Cg1 is the centroid of the C11–C16 phenyl ring with symmetry $-x$, $-y$, $2-z$), with an H···Cg distance of 2.66 Å and a C—H···Cg angle of 166°. The pseudo-related C14—H14 bond is not involved in such an interaction. Intermolecular C—H···π(arene) interactions have been shown to play a significant role in crystal packing (Brady *et al.*, 1998; Gallagher *et al.*, 1998). A monoclinic packing of adjacent layers would disrupt this intermolecular C—H···Cg1 interaction. Repulsive interlayer H···H contacts with distances as short as 2.20 Å would occur instead. Very similar triclinic structures with local monoclinic layers have been reported (Bats, Öhlinger & Mulzer, 1999; Bats, Gördes & Schmalz, 1999). It shows the tendency of these compounds to crystallize in a $2_1/c$ type lattice, which is the most common packing arrangement of crystal structures. A monoclinic packing is prevented in one direction by some unfavorable intermolecular interactions. As a consequence, these compounds crystallize in pseudo-$2_1/c$-type structures. Other examples of crystal structures with local pseudosymmetries have been reported by Zorky (1996).

The five-membered ring has an envelope conformation, with the C3 (for molecule 1) or the C25 atom (for molecule 2) 0.49 Å outside the plane through the other four ring atoms.

The C4—C5 and C26—C27 bonds of 1.569 (3) and 1.571 (3) Å, respectively, are rather long due to the bulky substituents at C4, C5, C26 and C27. A number of intramolecular O···H distances approach the van der Waals contact distance of 2.4 Å; O1···H6 2.36 (2), O1···H16 2.39 (2), O3···H28 2.38 (2) and O3···H34 2.38 (3) Å.

(a)

(b)

Fig. 1. The structures of the two independent molecules of (I), (a) molecule 1 and (b) molecule 2, shown with 50% probability ellipsoids.
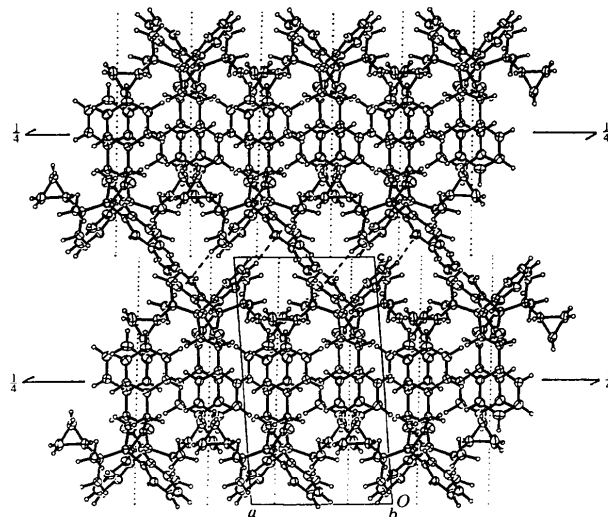

Fig. 2. The crystal packing of (I) shown down the *b* axis. The intermolecular C—H···π(arene) interactions are indicated by broken lines. The positions of the pseudo-glide planes and pseudo-twofold screw axes are also shown.

$C_{22}H_{24}O_2$

Experimental

The title compound was obtained as a product of a dyotropic ring-enlargement reaction of the isomeric β-lactone, with the ester O and the trans-diaxial-positioned H atom migrating (Mulzer et al., 1997). The substance was isolated as the major reaction product and was separated from a minor isomer by high-performance liquid chromatography. The compound was recrystallized from a mixture of ethyl acetate and diethyl ether.

Crystal data

$C_{22}H_{24}O_2$	Mo $K\alpha$ radiation
$M_r = 320.41$	$\lambda = 0.71073$ Å
Triclinic	Cell parameters from 165
$P\bar{1}$	reflections
$a = 8.4311\,(12)$ Å	$\theta = 3–23°$
$b = 14.505\,(2)$ Å	$\mu = 0.073$ mm^{-1}
$c = 15.609\,(3)$ Å	$T = 158\,(2)$ K
$\alpha = 73.317\,(10)°$	Plate
$\beta = 85.702\,(18)°$	$0.44 \times 0.20 \times 0.08$ mm
$\gamma = 89.375\,(10)°$	Colorless
$V = 1823.2\,(5)$ Å^3	
$Z = 4$	
$D_x = 1.167$ Mg m^{-3}	
D_m not measured	

Data collection

Siemens SMART diffractometer	$R_{int} = 0.074$
ω scans	$\theta_{max} = 28.5°$
Absorption correction: none	$h = -11 \rightarrow 11$
23 262 measured reflections	$k = -19 \rightarrow 19$
8657 independent reflections	$l = -19 \rightarrow 20$
5776 reflections with	282 standard reflections
$I > 2\sigma(I)$	frequency: 540 min
	intensity decay: none

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = 0.002$
$R[F^2 > 2\sigma(F^2)] = 0.097$	$\Delta\rho_{max} = 0.300$ e Å^{-3}
$wR(F^2) = 0.147$	$\Delta\rho_{min} = -0.275$ e Å^{-3}
$S = 1.708$	Extinction correction:
8657 reflections	SHELXL97
626 parameters	Extinction coefficient:
All H-atom parameters	0.0059 (10)
refined	Scattering factors from
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$	International Tables for
where $P = (F_o^2 + 2F_c^2)/3$	Crystallography (Vol. C)

All H atoms were refined and C—H distances are in the range 0.94 (3)–1.04 (3) Å.

Data collection: SMART (Siemens, 1995). Cell refinement: SMART. Data reduction: SAINT (Siemens, 1995). Program(s) used to solve structure: SHELXS96 (Sheldrick, 1996a). Program(s) used to refine structure: SHELXL97 (Sheldrick, 1997). Molecular graphics: XP in SHELXTL (Sheldrick, 1996b). Software used to prepare material for publication: CIF in SHELXL97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1338). Services for accessing these data are described at the back of the journal.

References

Bats, J. W., Gördes, D. & Schmalz, H.-G. (1999). Acta Cryst. C55, 1325–1328.
Bats, J. W., Öhlinger, S. H. & Mulzer, J. (1999). Acta Cryst. C55, 1158–1160.
Brady, F., Gallagher, J. F. & Kenny, P. T. M. (1998). Acta Cryst. C54, 1523–1525.
Gallagher, J. F., Briody, J. M. & Cantwell, B. P. (1998). Acta Cryst. C54, 1331–1335.
Hester, J. R. & Hall, S. R. (1996). J. Appl. Cryst. 29, 474–478.
Kálmán, A. & Argay, Gy. (1998). Acta Cryst. B54, 877–888.
Mulzer, J., Hoyer, K. & Müller-Fahrnow, A. (1997). Angew. Chem. Int. Ed. Engl. 36, 1476–1478.
Sheldrick, G. M. (1996a). SHELXS97. Program for the Solution of Crystal Structures. University of Göttingen, Germany.
Sheldrick, G. M. (1996b). SHELXTL. Structure Determination Software Programs. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). SHELXL97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany.
Siemens (1995). SMART and SAINT. Area Detector Control and Integration Software. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Zorky, P. M. (1996). J. Mol. Struct. 374, 9–28.

Pseudosymmetry with Z' = 4 in 1,3-propanesultone at 100 K

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Abstract

The asymmetric unit of the low-melting title compound, $C_3H_6O_3S$, contains four molecules at low temperature. The conformations of two of the five-membered rings are envelopes, one has the half-chair conformation, and the fourth is intermediate between an envelope and a half-chair. S=O distances range from 1.424 (2) to 1.436 (2) Å, S—O distances from 1.582 (2) to 1.587 (2) Å, and S—C distances from 1.760 (3) to 1.776 (3) Å. The crystal structure contains approximate centers, 2_1 screw axes, and translations. Above 180 K, the compound exists as a polymorph in which these symmetry operations are exact, and Z' = 1.

Comment

The title compound, (I), is a common reagent used to introduce sulfonate groups into hydrophobic mol-