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2,2-Dimethyl-5-oxo-2,3,4,5-tetrahydrofuran-3-yl benzoate

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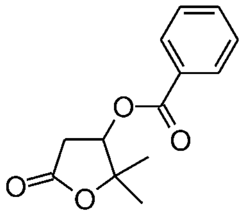
The title compound, $C_{13}H_{14}O_4$, crystallizes as a racemate. The heterocyclic core of the 3*S*-configured molecule adopts a $_2T^3$ conformation, whereas a $^2T_3$ arrangement is found for the 3*R* enantiomer. The majority of substituents in both stereoisomers are located in axial positions.

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.095
Data-to-parameter ratio = 9.8

Comment

Racemic 2,2-dimethyl-5-oxo-tetrahydrofuran-3-yl benzoate, (I), was isolated as a by-product from the vanadium(V)-catalyzed oxidation of 5-methyl-1-phenyl-4-hexen-1-ol with *tert*-butyl hydroperoxide in CH_2Cl_2 (Hartung, Drees *et al.*, 2003). The identity of lactone (I) was established by X-ray crystallography (Fig. 1).



(I)

Compound (I) crystallizes in the monoclinic space group $P2_1/c$ with one molecule in the asymmetric unit. The lactone ring in (I) adopts a twist conformation in the solid state. Atoms C2 and C3 are displaced in opposite directions from the plane defined by atoms O1, C4 and C5 [−0.373 (6) Å for atom C3 and 0.041 (6) Å for atom C2], thus leading to a $_3T^2$ conformation for the *S*-configured molecule, which, for

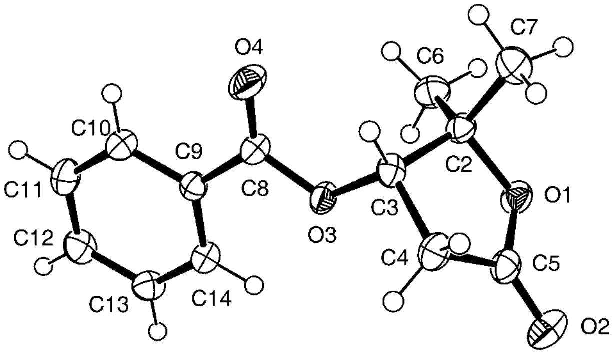


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are plotted at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The 3*S*-configured molecule has been arbitrarily selected for presentation purposes.

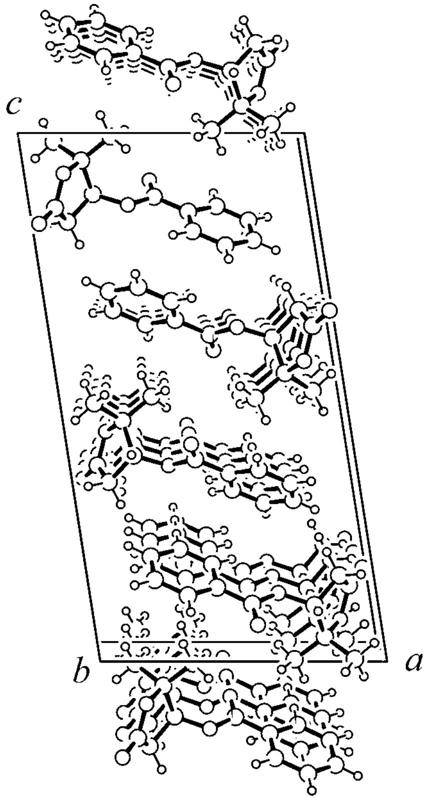


Figure 2
The packing of (I) in the solid state, viewed along [010].

reasons of symmetry, is equivalent to the ${}_2T^3$ arrangement (Romers *et al.*, 1969; Fuchs, 1978). The tetrahydrofuran subunit in 3*R*-(I), on the other hand, displays ${}^3T_2$, *i.e.* ${}^2T_3$, geometry.

The arrangement of the tetrahydrofuran entity in both enantiomers directs the benzoyloxy group and one of the methyl substituents at C2 into axial locations (Hartung, Kneuer *et al.*, 2003); the corresponding torsion angles are O3—C3—C2—O1 = −90.1 (2) and C7—C2—C3—C4 = −89.2 (2)°. The second CH_3 group is positioned equatorially [C6—C2—C3—C4 = 141.4 (2)°].

The ester functionality of the lactone exhibits an (*E*) configuration [O2—C5—O1—C2 = −177.8 (2)°], whereas the (*Z*) geometry is found for the benzoate entity [O4—C8—O3—C3 = 3.9 (3)°]. The C—O bond lengths [C5—O2 1.205 (3), C5—O1 1.340 (2), O1—C2 1.482 (3), C8—O4 1.198 (3), C8—O3 1.356 (3) and O3—C3 1.445 (2) Å] are in agreement with the recommended standard values for the ester functionality (Allen *et al.*, 1987).

Molecules with the same configuration, *i.e.* homomers, are packed on top of one another to form a columnar arrangement of (I) in the solid state, as seen in a view along *b* (Fig. 2).

Experimental

Crystals suitable for X-ray diffraction analysis were obtained by slow addition of petroleum ether to a solution of (I) in diethyl ether.

Crystal data

$C_{13}H_{14}O_4$
$M_r = 234.24$
Monoclinic, $P2_1/c$
$a = 10.811$ (2) Å
$b = 5.4155$ (5) Å
$c = 20.210$ (5) Å
$\beta = 98.871$ (1)°
$V = 1169.1$ (4) Å^3
$Z = 4$
$D_x = 1.331$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 0.10$ mm^{-1}
$T = 173$ (2) K
Block, colourless
0.30 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2926 measured reflections
1512 independent reflections
1179 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.061$
$\theta_{\rm max} = 22.5$°
$h = -11 \rightarrow 11$
$k = -5 \rightarrow 5$
$l = 0 \rightarrow 21$
3 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.095$
$S = 1.03$
1512 reflections
154 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0441P)^2 + 0.1845P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.14$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}

All H atoms were placed in geometrically idealized positions, with C—H distances in the range 0.95–1.00 Å, and were refined using the riding-model approximation, with displacement parameters constrained to 1.2 times $U_{\rm eq}$ of the parent atoms.

Data collection: *CAD*-4 *Diffractometer Control Software* (Enraf–Nonius, 1993); cell refinement: *CAD*-4 *Diffractometer Control Software*; data reduction: *CAD*-4 *Diffractometer Control Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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