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Literatur

- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
 Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
 Diefenbach, U. & Engelhardt, U. (1992). *Phosphorus, Sulfur and Silicon*, **65**, 107–109.
 Engelhardt, U., Bünger, T. & Viertel, H. (1984). *J. Crystallogr. Spectrosc. Res.* **14**, 603–615.
 Engelhardt, U., Diefenbach, U. & Damerius, R. (1990). *Z. Naturforsch. Teil B*, **45**, 457–460.
 Engelhardt, U. & Simon, A. (1992). *Acta Cryst.* **C48**, 492–495, 495–498.
 Engelhardt, U. & Simon, A. (1993). *Z. Anorg. Allg. Chem.* **619**, 1177–1182.
 Engelhardt, U. & Viertel, H. (1982). *Acta Cryst.* **B38**, 1972–1975, 3049–3052.
 Hall, S. R. & Stewart, J. M. (1989). Herausgeber. *Xtal2.6 User's Manual*. Univ. Western Australia, Australien und Maryland, VSta.
 Main, P., Lessinger, L., Woolfson, M. M., Germain, G. & Declercq, J.-P. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. York, England, und Louvain, Belgien.
 Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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4-(4-Methoxybenzoyl)-3-(2-methylallyl)-tetrahydropyran-2-one

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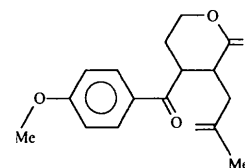
Abstract

The X-ray analysis of the title compound, $C_{17}H_{20}O_4$, allows the assignment of the *trans* (synclinal) relationship between the C(3) and C(4) substituents of the lactone ring. Moreover, it indicates that the heterocyclic ring assumes a boat conformation and

elucidates the conformations of the aroyl and allylic groups.

Comment

The present work forms part of a study of the structure and reactivity of 3,4-disubstituted tetrahydropyranones (Roux *et al.*, 1993; Roux, Wartski, Nierlich, Vigner & Lance, 1994). Since the 1H NMR data did not give unambiguous information about the conformation of the lactone ring and its aroyl and allylic substituents, the structure of the title compound has been obtained from single-crystal X-ray analysis. An ORTEPII (Johnson, 1976) drawing of the molecule is shown in Fig. 1.



(I)

The lactone ring adopts a boat conformation: O(1), C(2), C(4) and C(5) lie in a plane to within ± 0.1 Å, while C(3) and C(6) are at a distance of -0.514 (4) and -0.676 (5) Å, respectively, from this mean plane. The C(7)—C(3) and C(4)—C(11) bonds are synclinally (Klyne & Prelog, 1960) oriented [C(7)—C(3)—C(4)—C(11) = 84.0 (4) $^\circ$]. The relative configuration of C(3) and C(4) is $3R^*,4S^*$. The aroyl group conformation is denoted by the C(5)—C(4)—C(11)—O(3) and C(4)—C(11)—C(12)—C(17) torsion angles of -80.1 (4) and 26.1 (5) $^\circ$, respectively; the dihedral angle between the plane of the phenyl ring and the C(4)—C(11)—C(12) plane bearing the carbonyl group is 26.4 (5) $^\circ$. The orientation of the allylic group is denoted by the C(3)—C(7)—C(8)—C(9) and C(3)—C(7)—C(8)—C(10) torsion angles of 116.0 (5) and -67.0 (5) $^\circ$, respectively.

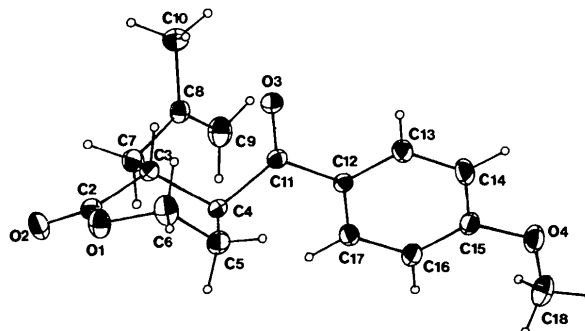


Fig. 1. ORTEPII (Johnson, 1976) drawing of the title compound with the atom-labelling scheme. H atoms are depicted as spheres of arbitrary radii. Displacement ellipsoids are shown at the 40% probability level.

Experimental

The title compound was prepared by Michael addition of lithiated 4-methoxyphenylacetonitrile to 2-hexenolide followed by 1-bromo-2-methyl-2-propene alkylation. After usual treatment of the reaction, the residue was crystallized from ethanol. Crystals were grown by slow evaporation of the alcoholic solution at room temperature (m.p. 377 K).

Crystal data

C ₁₇ H ₂₀ O ₄	Mo K α radiation
$M_r = 288.35$	$\lambda = 0.71073 \text{ \AA}$
Orthorhombic	Cell parameters from 25 reflections
<i>Pbca</i>	$\theta = 8-12^\circ$
$a = 8.057 (2) \text{ \AA}$	$\mu = 0.085 \text{ mm}^{-1}$
$b = 17.128 (6) \text{ \AA}$	$T = 295 \text{ K}$
$c = 21.578 (7) \text{ \AA}$	Platelet
$V = 2978 (3) \text{ \AA}^3$	$0.80 \times 0.50 \times 0.20 \text{ mm}$
$Z = 8$	Colourless
$D_x = 1.286 \text{ Mg m}^{-3}$	

Data collection

Enraf-Nonius CAD-4 diffractometer	$R_{int} = 0.025$
$\omega/2\theta$ scans	$\theta_{max} = 25^\circ$
Absorption correction: refined from ΔF (DIFABS; Walker & Stuart, 1983)	$h = 0 \rightarrow 9$
3071 measured reflections	$k = 0 \rightarrow 20$
2600 independent reflections	$l = -25 \rightarrow 3$
1401 observed reflections	3 standard reflections
$[I > 3\sigma(I)]$	frequency: 60 min
	intensity decay: insignificant

Refinement

Refinement on F	$\Delta\rho_{max} = 0.18 \text{ e \AA}^{-3}$
$R = 0.045$	$\Delta\rho_{min} = -0.21 \text{ e \AA}^{-3}$
$wR = 0.049$	Extinction correction: none
$S = 1.570$	Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
1401 reflections	
190 parameters	
Unit weights applied	
$(\Delta/\sigma)_{max} = 0.01$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	$B_{eq} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$			
	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
O(1)	0.8101 (4)	-0.0304 (2)	0.5421 (1)	4.39 (7)
O(2)	0.6864 (4)	0.0184 (2)	0.4608 (1)	4.57 (7)
O(3)	0.6584 (3)	0.1315 (2)	0.6844 (1)	3.70 (6)
O(4)	1.0745 (4)	0.4328 (2)	0.7484 (2)	4.66 (7)
C(2)	0.7192 (5)	0.0258 (2)	0.5147 (2)	3.38 (8)
C(3)	0.6639 (5)	0.0924 (2)	0.5560 (2)	2.74 (8)
C(4)	0.8120 (4)	0.1290 (2)	0.5909 (2)	2.78 (7)
C(5)	0.9416 (5)	0.0658 (2)	0.6063 (2)	4.4 (1)
C(6)	0.8647 (6)	-0.0141 (2)	0.6049 (2)	4.8 (1)
C(7)	0.5664 (5)	0.1522 (2)	0.5174 (2)	3.43 (8)
C(8)	0.4757 (5)	0.2123 (2)	0.5554 (2)	3.13 (8)

C(9)	0.5138 (6)	0.2871 (2)	0.5524 (2)	4.8 (1)
C(10)	0.3352 (5)	0.1826 (3)	0.5940 (2)	4.6 (1)
C(11)	0.7598 (5)	0.1652 (2)	0.6520 (2)	2.72 (7)
C(12)	0.8451 (4)	0.2367 (2)	0.6744 (2)	2.59 (7)
C(13)	0.8490 (5)	0.2521 (2)	0.7376 (2)	3.66 (9)
C(14)	0.9285 (6)	0.3168 (2)	0.7603 (2)	4.2 (1)
C(15)	1.0020 (5)	0.3702 (2)	0.7205 (2)	3.32 (8)
C(16)	0.9985 (5)	0.3572 (2)	0.6576 (2)	3.43 (8)
C(17)	0.9216 (5)	0.2902 (2)	0.6351 (2)	3.26 (8)
C(18)	1.1526 (6)	0.4894 (2)	0.7103 (2)	5.3 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$)

O(1)—C(2)	1.346 (5)	O(1)—C(6)	1.451 (5)
O(2)—C(2)	1.199 (5)	O(3)—C(11)	1.222 (4)
O(4)—C(15)	1.361 (5)	O(4)—C(18)	1.418 (5)
C(2)—C(3)	1.515 (5)	C(3)—C(4)	1.544 (5)
C(3)—C(7)	1.536 (5)	C(4)—C(5)	1.540 (5)
C(4)—C(11)	1.516 (5)	C(5)—C(6)	1.504 (6)
C(7)—C(8)	1.505 (5)	C(8)—C(9)	1.319 (5)
C(8)—C(10)	1.494 (6)	C(11)—C(12)	1.485 (5)
C(2)—O(1)—C(6)	116.0 (3)	C(15)—O(4)—C(18)	118.2 (4)
O(1)—C(2)—O(2)	118.1 (3)	O(1)—C(2)—C(3)	116.1 (4)
O(2)—C(2)—C(3)	125.7 (3)	C(2)—C(3)—C(4)	111.4 (3)
C(2)—C(3)—C(7)	109.5 (3)	C(4)—C(3)—C(7)	112.9 (3)
C(3)—C(4)—C(5)	110.1 (3)	C(3)—C(4)—C(11)	112.1 (3)
C(5)—C(4)—C(11)	106.7 (3)	C(4)—C(5)—C(6)	110.9 (3)
O(1)—C(6)—C(5)	108.6 (3)	C(3)—C(7)—C(8)	114.1 (3)
C(7)—C(8)—C(9)	121.7 (4)	C(7)—C(8)—C(10)	116.0 (4)
C(9)—C(8)—C(10)	122.2 (4)	O(3)—C(11)—C(4)	119.4 (3)
O(3)—C(11)—C(12)	120.9 (3)	C(4)—C(11)—C(12)	119.5 (3)
O(4)—C(15)—C(16)	124.9 (4)	O(4)—C(15)—C(14)	115.4 (3)

Data were collected using Enraf-Nonius CAD-4 diffractometer software. Lorentz-polarization corrections were applied. The structure solved by direct methods using *MULTAN11/82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Refinement was by full-matrix least-squares methods. H atoms were refined using a riding model with C—H 0.95 \AA , B = 6 \AA^2 . Heavy atoms were refined with anisotropic displacement parameters. All calculations were performed on a VAX4200 computer. Programs used were *MolEN* (Fair, 1990), *MULTAN11/82* and *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and torsion angles have been deposited with the IUCr (Reference: KA1079). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf-Nonius, Delft, The Netherlands.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Klyne, W. & Prelog, V. (1960). *Experientia*, **16**, 521-568.
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Roux, M. C., Seyden-Penne, J., Wartski, L., Posner, G. J., Nierlich, M., Vigner, D. & Lance, M. (1993). *J. Org. Chem.* **58**, 3969-3973.
- Roux, M. C., Wartski, L., Nierlich, M., Vigner, D. & Lance, M. (1994). *Tetrahedron*, **50**, 8445-8456.
- Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158-166.