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2-Bromo-4'-phenylacetophenone*

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Abstract. C₁₄H₁₁BrO, $M_r = 275.15$, orthorhombic, *Pcab* ($a\bar{c}b$ orientation of *Pbca*), $a = 7.794$ (1), $b = 10.961$ (2), $c = 27.106$ (3) Å, $V = 2316$ (1) Å³, $Z = 8$, $D_x = 1.58$ Mg m⁻³, Cu $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 5.1$ mm⁻¹, $F(000) = 1104$, $T = 293$ K, $R = 0.050$ for 1600 reflections. The bromoacetyl group is tilted at 5.4 (1)° to the adjacent phenyl group and the two phenyl groups of the biphenyl are inclined at an angle of 33.9 (1)°. Bond lengths and angles fall within normal ranges.

Introduction. Although biphenyl is non-planar in the gas phase (Bastiansen & Trætteberg, 1962; Schmid &

Brosa, 1972), the phenyl rings are coplanar in the crystal (Trotter, 1961; Hargreaves & Rizvi, 1962; Charbonneau & Delugeard, 1976). In 4-nitrobiphenyl (Casalone, Gavezzotti & Simonetta, 1973) and 4,4'-dinitrobiphenyl (Boonstra, 1963) the phenyl rings are inclined at 33° and in 4,4'-bitolyl the angle of twist between the aromatic rings is *ca* 38° (Casalone, Mariani, Mugnoli & Simonetta, 1969). A halogen substituent at C(3) can be accommodated with little effect on the twist angle, which is 39° in 4-acetyl-3'-bromobiphenyl (Sutherland & Hoy, 1969). A halogen substituent at C(2) introduces greater steric interactions and several biphenyl derivatives of this type have interplanar angles of *ca* 48° (Young, Tollin & Sutherland, 1968; Sutherland & Hoy, 1968; Sutherland, 1969).

* 4-Phenylphenacyl bromide; 4-bromoacetyl biphenyl.

An X-ray study of the title compound, $4\text{-C}_6\text{H}_5\text{-C}_6\text{H}_4\text{COCH}_2\text{Br}$, was undertaken to determine the interplanar angle in this biphenyl.

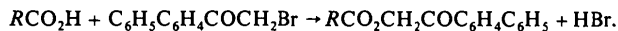
Experimental. Colourless crystal, dimensions $0.35 \times 0.15 \times 0.15$ mm. Enraf-Nonius CAD-4 diffractometer, graphite monochromator, $\text{Cu K}\alpha$ radiation. Cell dimensions from setting angles of 25 independent reflections with θ $20\text{--}24^\circ$. 2300 intensities surveyed in the range θ $2\text{--}65^\circ$; h $0\text{--}9$, k $0\text{--}12$, l $0\text{--}31$; 1600 independent reflections with $I > 2.5\sigma(I)$. Two reference reflections monitored periodically showed no significant variation in intensity. No absorption correction. Structure determined by Patterson and Fourier methods. H atoms located in a difference Fourier synthesis. Least-squares calculations on F with anisotropic thermal parameters for C, N and O atoms and isotropic for H atoms. Convergence at R 0.050 , wR 0.074 , S 3.45 , $\Delta/\sigma \leq 0.22$ for C, N and O atoms and ≤ 0.41 for H, $w = 1/\sigma^2(|F_o|)$. Final $\Delta\rho$ max. 0.37 , min. -0.88 e \AA^{-3} in vicinity of Br. Scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations on a SEL 32/27 computer with the *GX* system of programs (Mallinson & Muir, 1985).

Discussion. Atomic coordinates are listed in Table 1 and molecular dimensions in Table 2.* Fig. 1, drawn with *ORTEP* (Johnson, 1965), illustrates the molecular geometry.

The bromoacetyl group is closely coplanar and inclined at only $5.4(1)^\circ$ to the plane of the adjacent phenyl ring. The dihedral angle between the phenyl rings is $33.9(1)^\circ$, in good agreement with values obtained for other 4-substituted biphenyls. Because of the rotation around the $\text{C}(4)\text{--}\text{C}(7)$ bond the $\text{H}(5)\cdots\text{H}(8)$ and $\text{H}(3)\cdots\text{H}(12)$ separations are greater than 2 \AA .

The length of the bond connecting the phenyl rings, $1.475(6) \text{ \AA}$, is close to that of the phenyl-acyl bond, $1.481(7) \text{ \AA}$. Both values are close to the standard value, 1.48 \AA , for a $\text{C}_{sp^2}\text{--}\text{C}_{sp^2}$ single bond.

The $\text{C}\text{--}\text{Br}$ bond in 4-phenylphenacyl bromide is labile and the compound can be used for the preparation of derivatives of carboxylic acids:



The $\text{C}\text{--}\text{Br}$ bond length, however, is normal, $1.915(6) \text{ \AA}$.

* Lists of coordinates and bond lengths involving H atoms, structure factors, anisotropic thermal parameters and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43031 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

$$U_{\text{eq}} = (U_{11}U_{22}U_{33})^{1/3}.$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Br	0.17492 (9)	0.19864 (6)	0.09484 (2)	0.070
C(1)	0.3790 (6)	0.2287 (4)	-0.0505 (2)	0.039
C(2)	0.4268 (7)	0.3228 (4)	-0.0824 (2)	0.044
C(3)	0.4869 (7)	0.2990 (4)	-0.1285 (2)	0.042
C(4)	0.5047 (5)	0.1794 (4)	-0.1460 (2)	0.034
C(5)	0.4576 (6)	0.0863 (4)	-0.1139 (2)	0.040
C(6)	0.3964 (6)	0.1085 (4)	-0.0672 (2)	0.040
C(7)	0.5615 (5)	0.1535 (4)	-0.1969 (2)	0.033
C(8)	0.6553 (5)	0.0487 (4)	-0.2083 (2)	0.041
C(9)	0.7000 (7)	0.0219 (5)	-0.2563 (2)	0.049
C(10)	0.6559 (7)	0.1005 (5)	-0.2937 (2)	0.049
C(11)	0.5650 (7)	0.2048 (5)	-0.2834 (2)	0.047
C(12)	0.5190 (6)	0.2314 (4)	-0.2356 (2)	0.039
C(13)	0.3088 (6)	0.2596 (4)	-0.0013 (2)	0.043
C(14)	0.2721 (9)	0.1535 (5)	0.0326 (2)	0.055
O(1)	0.2814 (7)	0.3625 (3)	0.0111 (1)	0.075

Table 2. *Interatomic distances (\AA) and angles ($^\circ$)*

Br-C(14)	1.915 (6)	C(1)-C(2)	1.396 (7)
C(1)-C(6)	1.400 (7)	C(1)-C(13)	1.481 (7)
C(2)-C(3)	1.359 (8)	C(3)-C(4)	1.402 (7)
C(4)-C(5)	1.392 (7)	C(4)-C(7)	1.475 (6)
C(5)-C(6)	1.374 (7)	C(7)-C(8)	1.397 (7)
C(7)-C(12)	1.392 (7)	C(8)-C(9)	1.377 (8)
C(9)-C(10)	1.375 (8)	C(10)-C(11)	1.373 (8)
C(11)-C(12)	1.377 (8)	C(13)-C(14)	1.509 (8)
C(13)-O(1)	1.196 (7)		
C(2)-C(1)-C(6)	117.9 (5)	C(2)-C(1)-C(13)	119.2 (5)
C(6)-C(1)-C(13)	122.9 (5)	C(1)-C(2)-C(3)	121.3 (5)
C(2)-C(3)-C(4)	121.7 (5)	C(3)-C(4)-C(5)	116.6 (5)
C(3)-C(4)-C(7)	121.8 (4)	C(5)-C(4)-C(7)	121.5 (4)
C(4)-C(5)-C(6)	122.5 (5)	C(1)-C(6)-C(5)	119.9 (5)
C(4)-C(7)-C(8)	121.5 (4)	C(4)-C(7)-C(12)	121.0 (4)
C(8)-C(7)-C(12)	117.5 (5)	C(7)-C(8)-C(9)	121.1 (5)
C(8)-C(9)-C(10)	120.0 (5)	C(9)-C(10)-C(11)	120.0 (6)
C(10)-C(11)-C(12)	120.2 (5)	C(7)-C(12)-C(11)	121.2 (5)
C(1)-C(13)-C(14)	116.3 (5)	C(1)-C(13)-O(1)	122.2 (5)
C(14)-C(13)-O(1)	121.5 (5)	Br-C(14)-C(13)	114.4 (4)

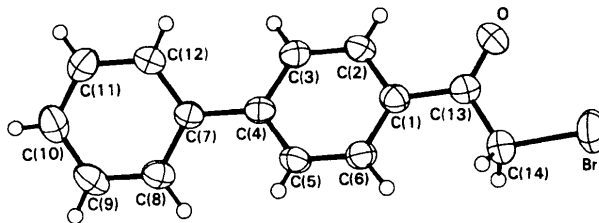


Fig. 1. Molecular structure and atomic numbering: the thermal ellipsoids of the C, O and Br atoms are drawn at the 50% probability level and H atoms are represented by spheres of radius 0.1 \AA .

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X-ray Structure of 8 α -Acetoxy-1,3,4,10-tetrahydro-1 α ,10 α -epoxyachillin

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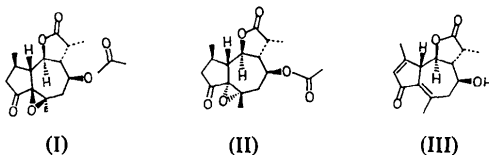
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Abstract. (5 α H,6 β H,11 α H)-8 α -Acetoxy-1,5-dihydro-2-oxo-1 α ,10 α -epoxyguaian-6,12-olide, C₁₇H₂₂O₆, M_r = 322.3, orthorhombic $P2_12_12_1$, a = 14.964 (6), b = 17.057 (4), c = 6.218 (4) Å, V = 1587 (1) Å³, Z = 4, D_m = 1.349 (5), D_x = 1.349 Mg m⁻³, λ (Mo $K\alpha$) = 0.7107 Å, μ = 0.09 mm⁻¹, $F(000)$ = 688, room temperature, final wR = 0.068 for 1899 observed reflexions. The conformations displayed by the cyclopentanone, γ -lactone and cycloheptane rings are near to a twist, an envelope and a chair form, respectively. The cyclopentanone–cycloheptane and γ -lactone–cycloheptane ring fusions are *cis* and *trans*, respectively. Packing in the crystal is due to weak C–H...O hydrogen-bond interactions giving chains of molecules parallel to [010] and [001].

Introduction. The title compound (I) (m.p. 469–471 K, M^+ m/z 322) was prepared, along with its stereoisomer at C(1) and C(10) (II), from 8 α -hydroxyachillin (III), a natural product isolated from *Artemisia lanata* Willd (González, Bermejo, de la Rosa & Massanet, 1976).



The detailed ¹H NMR analysis and the criteria based on the H(6) paramagnetic shift (Bates, Procházka & Cekan, 1963; Ando, Akahane & Takase, 1978), usually

employed to establish the stereochemistry of the 1,10-epoxyguaianolides (Bhacca & Williams, 1964), seem to fail in this case (Massanet, 1986). An X-ray diffraction analysis was suggested to establish definitely the conformational details of (I).

Experimental. Single crystals in the form of colourless needles elongated along [001], prepared in the Organic Chemistry Department of the University of Cádiz. D_m by flotation. Crystal 0.12 × 0.10 × 0.16 mm. Unit-cell parameters from 25 reflexions, $5 < \theta < 15^\circ$. Enraf–Nonius CAD-4 diffractometer, graphite monochromator, $2 \leq \theta \leq 30^\circ$ ($0 \leq h \leq 21$, $0 \leq k \leq 24$, $0 \leq l \leq 8$), ω - 2θ scan mode. Two standard reflexions (410 , $4\bar{1}0$), variation in intensity < 3% of mean value. 2638 independent reflexions measured, 739 considered unobserved [$I < 2\sigma(I)$]. Lorentz and polarization correction, no correction for absorption or extinction. Structure solved by direct methods using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The chirality was assumed on the basis of chemical information. Full-matrix least-squares refinement; $\sum w(|F_o| - |F_c|)^2$ minimized with weighting scheme based on statistical-count criterion ($w = 1/\sigma_p^2$). Difference Fourier synthesis revealed the 22 H-atom positions; isotropic temperature factor $B = 4.0 \text{ \AA}^2$ for H atoms; further least-squares refinement including positional parameters of H atoms. At final convergence $\Delta/\sigma < 0.4$, $R = 0.081$, $wR = 0.068$, $S = 2.7$ for 274 refined variables; number of reflexions/number of parameters = 7.87. Final accuracy of the refinement is limited by the poor quality of the crystals.