disorder. Fig. 1 shows the molecule with adopted numbering. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Data on the geometry are assembled in Table 2. Scattering factors of Cromer & Mann (1968) were used. Geometrical calculations and illustrations were made with the programs *PLATON* and *PLUTON* of the *EUCLID* package (Spek, 1982).

Related literature. The title compound was isolated as an intermediate in the total synthesis of $\mathrm{C_{19}}$-diterpene alkaloids (van Beek, van der Baan, Klumpp & Bickelhaupt, 1986). The related structure of methyl 3,4-epoxy-5,5-ethylenedioxy-*exo*-7,*endo*-11-dihydroxytricyclo[7.2.1.$0^{4,10}$]dodecane-8-carboxylate has been reported previously (Spek & van Eijck, 1987).

Crystals were kindly provided by Dr van der Baan and Professor Bickelhaupt. The investigations were supported in part by the Netherlands Foundation for Chemical Research (SON) with financial aid from the Netherlands Organization for Advancement of Pure Research (ZWO).

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* Lists of anisotropic thermal parameters, H-atom parameters and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44809 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1988). C**44**, 1141–1143

Methyl 2-[2-(2-Hydroxyphenyl)ethyl]benzoate

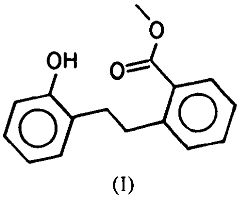
By Jeffrey A. Corkern, Frank R. Fronczek, Richard D. Gandour, Kermin Guo, Michael A. Oliver and Steven F. Watkins

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803-1804, *USA*

(*Received* 23 *December* 1987; *accepted* 26 *January* 1988)

Abstract. $\mathrm{C_{16}H_{16}O_3}$, $M_r = 256\cdot3$, monoclinic, $P2_1/n$, $a = 12\cdot308\ (3)$, $b = 7\cdot300\ (1)$, $c = 16\cdot034\ (2)$ Å, $\beta = 110\cdot17\ (2)^\circ$, $V = 1352\cdot2\ (9)$ Å^3, $Z = 4$, $D_x = 1\cdot259$ g cm^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0\cdot71073$ Å, $\mu = 0\cdot8$ cm^{-1}, $F(000) = 544$, $T = 295\ (1)$ K, $R = 0\cdot033$ for 1605 reflections with $I > 3\cdot0\sigma(I)$ (2368 unique observations). The molecule is a substituted dibenzyl [staggered 1,2-diphenylethane, $\omega(\mathrm{C-C}) = 176\cdot1\ (1)^\circ$, with substituents (hydroxyl and methoxycarbonyl) in the 2- and 2′-ring positions. The rings are nearly parallel [dihedral angle $6\cdot0\ (4)^\circ$] and the methoxycarbonyl group is twisted $18\cdot7\ (1)^\circ$ out of the plane of its attached phenyl ring in order to form a weak intramolecular hydrogen bond: $\mathrm{O\cdots O} = 2\cdot788\ (2)$ Å, $\mathrm{O-H\cdots O} = 159\ (1)^\circ$, $\mathrm{H\cdots O} = 1\cdot91\ (2)$ Å.

Experimental. The compound (I) was obtained by refluxing 2-[2-(2-hydroxyphenyl)ethyl]benzoic acid in acidic methanol and recrystallizing from hexane. A colorless prismatic crystal was mounted with epoxy on a glass fiber in random orientation. Details of data collection and structural refinement are given in Table 1.



(I)

The structure was solved using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All 19 non-H atoms were located from an *E* map. H atoms were located by difference Fourier synthesis and their positions and isotropic thermal parameters were refined. All non-H atoms were treated anisotropically. The structural model was refined by full-matrix least squares with Enraf–Nonius *SDP* (Frenz, 1978), in which the function minimized was

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Table 1. *Experimental details*

Crystal	Colorless prismatic 0·25 × 0·37 × 0·50 mm
Instrument	Enraf–Nonius CAD-4 diffractometer
Monochromator	Incident beam, graphite
Unit cell	25 reflections, $24·1 < 2\theta < 25·7°$
Mode	ω–2θ
Standards	200, 021, 006
R_{int}	0·020
Corrections	Background, Lorentz, polarization. Extinction [$9·6\ (9) \times 10^{-7}$]
2θ range (°)	2·7 – 50·0
hkl ranges	$h = 0$ to 14 $k = 0$ to 8 $l = -19$ to 17
Reflections	2475 total 2368 unique 1605 with $I > 3·0\sigma(I)$
Solution	Direct methods
Weights	$4F_o^2\text{Lp}^2/[S^2(C+R^2B) + (0·020F_o^2)^2]$ S = scan rate, C = integrated count, R = scan time/background time, B = background count
Parameters refined	237
R, wR, R(all)	0·033, 0·037, 0·066
Goodness of fit	1·97
Maximum shift/e.s.d.	0·04
$\Delta\rho$ (e Å^{-3})	0·13 (2), −0·10 (2)

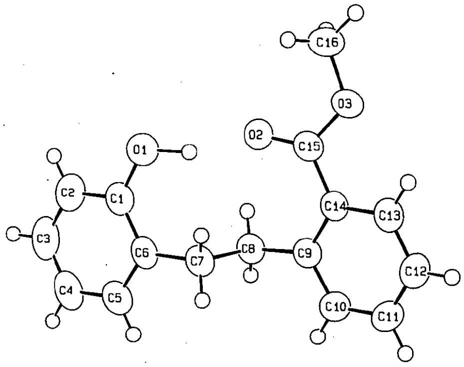

Fig. 1. Single molecule, 50% ellipsoids (Johnson, 1965).

$\sum w(|F_o| - |F_c|)^2$ and the weight w is defined as $4F_o^2\sigma^2(F_o^2)$. The final cycle of refinement included 237 variable parameters including extinction and converged to $R = 0·033$. Atomic scattering factors, including those for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974).

Final positional and equivalent isotropic thermal parameters are shown in Table 2, and selected interatomic distances, angles and torsion angles are shown in Table 3.* Fig. 1 shows the molecule and the atomic numbering scheme.

Table 2. *Positional parameters and their e.s.d.'s*

	x	y	z	B_{eq}(Å^2)
O1	−0·0490 (1)	0·6719 (2)	0·65865 (7)	5·84 (3)
O2	0·05645 (9)	0·3294 (1)	0·69907 (6)	4·65 (3)
O3	0·13974 (9)	0·0580 (2)	0·70250 (6)	4·65 (3)
C1	−0·0726 (1)	0·7848 (2)	0·7183 (1)	4·22 (4)
C2	−0·1585 (1)	0·9137 (2)	0·6831 (1)	5·18 (4)
C3	−0·1874 (1)	1·0342 (3)	0·7386 (1)	5·54 (4)
C4	−0·1314 (1)	1·0254 (2)	0·8284 (1)	5·32 (4)
C5	−0·0443 (1)	0·8978 (2)	0·8632 (1)	4·62 (4)
C6	−0·0126 (1)	0·7758 (2)	0·80927 (9)	3·75 (3)
C7	0·0815 (1)	0·6360 (2)	0·84800 (9)	3·95 (4)
C8	0·0342 (1)	0·4487 (2)	0·86253 (9)	3·68 (3)
C9	0·1294 (1)	0·3080 (2)	0·89545 (9)	3·44 (3)
C10	0·1837 (1)	0·2881 (2)	0·98653 (9)	4·25 (4)
C11	0·2745 (1)	0·1685 (2)	1·02307 (9)	4·91 (4)
C12	0·3129 (1)	0·0624 (2)	0·9683 (1)	4·92 (4)
C13	0·2596 (1)	0·0755 (2)	0·87733 (9)	4·25 (4)
C14	0·1684 (1)	0·1975 (2)	0·83994 (9)	3·37 (3)
C15	0·1151 (1)	0·2055 (2)	0·74172 (9)	3·57 (3)
C16	0·0891 (1)	0·0510 (3)	0·6067 (1)	5·30 (4)

The equivalent isotropic thermal parameter for atoms refined anisotropically is defined by the equation:
$\frac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + abB_{12}\cos\gamma + acB_{13}\cos\beta + bcB_{23}\cos\alpha]$.

Table 3. *Selected internuclear distances* (Å), *angles* (°) *and torsion angles* (°)

O1—H1	0·92 (2)	C5—C6	1·388 (2)
O1—O2	2·788 (2)	C6—C7	1·507 (2)
O1—C1	1·368 (1)	C7—C8	1·535 (2)
O2—C15	1·211 (1)	C8—C9	1·510 (2)
O3—C15	1·333 (1)	C9—C10	1·388 (2)
O3—C16	1·445 (2)	C9—C14	1·403 (1)
C1—C2	1·382 (2)	C10—C11	1·380 (2)
C1—C6	1·390 (2)	C11—C12	1·371 (2)
C2—C3	1·382 (2)	C12—C13	1·381 (2)
C3—C4	1·367 (2)	C13—C14	1·396 (2)
C4—C5	1·383 (2)	C14—C15	1·484 (2)

C1—O1—H1	113 (1)	C7—C8—C9	111·7 (1)
C15—O2—H1	153·4 (5)	C8—C9—C10	118·2 (1)
O1—H1—O2	159 (1)	C8—C9—C14	124·3 (1)
C15—O3—C16	116·4 (1)	C10—C9—C14	117·5 (1)
O1—C1—C2	116·2 (1)	C9—C10—C11	122·6 (1)
O1—C1—C6	122·7 (1)	C10—C11—C12	119·6 (1)
C2—C1—C6	121·0 (1)	C11—C12—C13	119·6 (1)
C1—C2—C3	120·1 (1)	C12—C13—C14	121·1 (1)
C2—C3—C4	120·0 (1)	C9—C14—C13	119·6 (1)
C3—C4—C5	119·7 (1)	C9—C14—C15	121·9 (1)
C4—C5—C6	121·7 (1)	C13—C14—C15	118·5 (1)
C1—C6—C5	117·5 (1)	O2—C15—O3	121·7 (1)
C1—C6—C7	121·3 (1)	O2—C15—C14	126·0 (1)
C5—C6—C7	121·3 (1)	O3—C15—C14	112·3 (1)
C6—C7—C8	112·8 (1)		

O1—C1—C6—C7	−1·5 (2)	C8—C9—C14—C15	−2·2 (2)
C2—C1—C6—C7	−179·7 (1)	C10—C9—C14—C15	178·9 (1)
C4—C5—C6—C7	−179·3 (2)	C12—C13—C14—C15	179·8 (2)
C1—C6—C7—C8	−83·2 (2)	C13—C14—C15—O2	−161·2 (2)
C5—C6—C7—C8	95·3 (2)	C9—C14—C15—O2	19·0 (2)
C6—C7—C8—C9	176·1 (1)	C13—C14—C15—O3	18·0 (2)
C7—C8—C9—C10	90·5 (2)	C9—C14—C15—O3	−161·8 (1)
C7—C8—C9—C14	−88·3 (2)	C16—O3—C15—C14	178·5 (1)
C8—C9—C10—C11	−177·4 (2)	C16—O3—C15—O2	−2·3 (2)
C8—C9—C14—C13	178·0 (1)		

Numbers in parentheses are e.s.d.'s in the least significant digits.

Related literature. Preparation of parent acid: Baker, Ollis & Zealley (1952), Chatteyea, Prasad & Banerji (1964); preparation of isomeric methyl ether carboxylic acid: Vejdelek, Nemecek, Musil & Simek (1964);

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* Lists of structure factors, anisotropic thermal parameters, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44732 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

chemistry of parent acid and its derivatives: Oliver (1986); structure of dibenzyl: Robertson (1935), Jeffrey (1947), Cruickshank (1949); structures of some dibenzyl derivatives: Brown (1954), Sato, Yamato, Hashigaki & Koyama (1978), Corey (1979).

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Acta Cryst. (1988). C**44**, 1143–1144

Structure of 2-Chloro-1-(2,4-dihydroxyphenyl)ethanone

By P. H. van Rooyen*

National Chemical Research Laboratory, Council for Scientific and Industrial Research, PO Box 395, *Pretoria* 0001, *Republic of South Africa*

and J. C. Breytenbach

Department of Pharmaceutical Chemistry, Potchefstroom University for CHE, Potchefstroom 2520, *Republic of South Africa*

(*Received* 3 *September* 1987; *accepted* 18 *February* 1988)

Abstract. ω-Chlororesacetophenone, $C_8H_7ClO_3$, $M_r = 186\cdot8$, monoclinic, $P2_1/a$, $a = 7\cdot335\,(1)$, $b = 15\cdot457\,(10)$, $c = 6\cdot818\,(1)$ Å, $\beta = 92\cdot29\,(1)°$, $U = 772\,(1)$ Å^3, $Z = 4$, $D_x = 1\cdot61$ Mg m^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0\cdot7107$ Å, $\mu = 0\cdot783$ mm^{-1}, $F(000) = 768$, $T = 298$ K. Final $R = 0\cdot077$ for 1275 observed reflections with $F_o > 4\sigma(F_o)$ and 131 variable parameters. The molecule is planar with normal bond lengths and angles. There is strong intramolecular hydrogen bonding through a hydroxyl H atom and the ketone O atom, with an H···O distance of 1·63 (1) Å.

Experimental. The title compound was formed by the Hoesch condensation of resorcinol with chloroacetonitrile (Dawkins & Mulholland, 1959) and yielded almost colourless needles (toluene, m.p. 404 K). Crystal 0·26 × 0·23 × 0·16 mm; Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized radiation; unit cell from 25 reflections ($7 \le \theta \le 20°$); 1759 reflections for $3 \le \theta \le 27°$ in the range ($0 \le h \le 9$, $0 \le k \le 19$ and $-9 \le l \le 9$) using $\omega/2\theta$ scans where ω changed as $0\cdot59 + 0\cdot35\tan\theta°$ with a variable but maximum speed that corresponds to 5·49° min^{-1}. Three standard reflections varied less than 1%, measured every hour; Lorentz–polarization; no absorption correction. 1759 unique reflections, 484 rejected with $F_o \le 4\sigma(F_o)$; structure solved using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); all H atoms from difference Fourier synthesis; least-squares refinement using *SHELX*76 (Sheldrick, 1976), F magnitudes, $\sigma^{-2}(F)$ weights, all non-H atoms anisotropic, H atoms isotropic with a common thermal parameter [U_{iso} (H) 0·092 (10) Å^2], 131 variables refined. Final $wR = 0\cdot066$, $R = 0\cdot077$, $(\Delta/\sigma)_{\max} = 0\cdot07$, residual electron density = 0·56 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Table 1 gives the atom parameters, Fig. 1 shows the molecular structure and the atomic numbering scheme drawn by *ORTEP* (Johnson, 1965).†

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* To whom all correspondence should be addressed.

† Lists of structure factors, anisotropic thermal parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44792 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/88/061143-02$03.00