

(scan rate); where 0.02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction for Lp effects and decay. $\sigma(I)$ was estimated from counting statistics: $\sigma(I) = [(I_{\text{peak}} + I_{\text{background}})^{1/2} \times (\text{scan rate})]$. Final $R = 0.0536$ for 1673 reflections, with $wR = 0.0582$ ($R_{\text{all}} = 0.0894$, $wR_{\text{all}} = 0.0687$) and goodness of fit = 1.636. Maximum $|\Delta/\sigma| < 0.1$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were -0.26 and $0.50 \text{ e } \text{Å}^{-3}$, respectively. Scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with anomalous-dispersion corrections taken from the work of Cromer & Liberman (1970). Scattering factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear-absorption coefficient were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55). * Fig. 1, showing the atom-labelling scheme, was generated using *SHELXTL-Plus* (Sheldrick, 1991). The positional and thermal parameters for non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are

* Lists of anisotropic thermal parameters, H-atom positional parameters, bond distances and angles involving H atoms, torsion angles, and structure-factor amplitudes, as well as a unit-cell packing diagram, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55205 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0565]

listed in Table 2. Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

Related literature. The structure of (I) was determined as part of a generalized approach to the syntheses of alkaloids of the indole family (Martin, Rüeger, Williamson & Grzejszczak, 1987; Martin, Benage & Hunter, 1988).

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References

- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
 CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
 GADOL, S. M. & DAVIS, R. E. (1982). *Organometallics*, **1**, 1607–1613.
 HIRAI, Y., TERADA, T. & YAMAZAKI, T. (1988). *J. Am. Chem. Soc.* **110**, 958–960.
 MARTIN, S. F., BENAGE, B. & HUNTER, J. E. (1988). *J. Am. Chem. Soc.* **110**, 5925–5927.
 MARTIN, S. F. & CORBETT, J. W. (1991). Unpublished results.
 MARTIN, S. F., RÜEGER, H., WILLIAMSON, S. A. & GRZEJSZCZAK, S. (1987). *J. Am. Chem. Soc.* **109**, 6124–6134.
 MELCHIO, J., BOUQUET, A., PAIS, M. & GOUTAREL, R. (1977). *Tetrahedron Lett.* pp. 315–316.
 SHELDRICK, G. M. (1991). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1992). **C48**, 1880–1881

2-Menthyl (2-Hydroxyphenyl)glycolate

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Abstract. 2-Menthyl hydroxy(2-hydroxyphenyl)ethanoate, C₁₈H₂₆O₄, $M_r = 306.40$, monoclinic, $P2_1$, $a = 13.686$ (2), $b = 5.874$ (2), $c = 11.030$ (3) Å, $\beta = 96.62$ (3)°, $V = 880.8$ Å³, $Z = 2$, $D_x = 1.16 \text{ g cm}^{-3}$, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 6.14 \text{ cm}^{-1}$, $F(000) = 332$, room temperature, $R = 0.044$ for 1715 unique observed reflections with $I \geq 2\sigma(I)$. The cyclohexane ring adopts the chair conformation. The absolute configuration at C7 was inferred to be *R* on the basis of those of the compounds used in the synthesis. A short intramolecular bond [O2...H1O1 = 1.84 (6) Å] is observed.

Experimental. A prismatic crystal $0.11 \times 0.19 \times 0.27$ mm was used for data collection on a Siemens AED single-crystal diffractometer equipped with an IBM PS2/30 personal computer (Belletti, Cantoni & Pasquinelli, 1988), and Cu $K\alpha$ radiation. Cell parameters were determined by least-squares fit of the setting angles of 27 reflections with $11.02 \leq \theta \leq 31.32^\circ$. 1933 reflections were measured ($3 \leq \theta \leq 70^\circ$) using a modified version (Belletti *et al.*, 1988) of the Lehmann & Larsen (1974) procedure; 1847 were unique ($R_{\text{int}} = 0.017$) and 1715 with $I \geq 2\sigma(I)$ observed ($-16 \leq h \leq 16$, $0 \leq k \leq 7$, $0 \leq l \leq 13$). One

Table 1. Atomic fractional coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$)

	x	y	z	U_{eq}
O1	8482 (2)	-1551 (7)	6177 (3)	1034 (10)
O2	9601 (1)	1923*	5682 (2)	745 (7)
O3	9231 (2)	367 (7)	3375 (2)	881 (8)
O4	8125 (1)	3124 (6)	2903 (1)	575 (5)
C1	7810 (2)	2103 (6)	5457 (2)	537 (6)
C2	7721 (2)	-4 (7)	6014 (3)	692 (9)
C3	6831 (3)	-662 (8)	6406 (4)	875 (13)
C4	6029 (2)	746 (9)	6236 (3)	843 (11)
C5	6104 (2)	2839 (10)	5696 (3)	843 (12)
C6	6995 (2)	3509 (8)	5309 (2)	683 (9)
C7	8756 (2)	2847 (7)	4972 (2)	565 (6)
C8	8747 (1)	1962 (6)	3670 (2)	537 (6)
C9	7993 (2)	2398 (7)	1623 (2)	536 (7)
C10	6903 (2)	2573 (6)	1175 (2)	537 (7)
C11	6778 (2)	1952 (8)	-186 (3)	677 (10)
C12	7393 (2)	3473 (10)	-911 (2)	781 (11)
C13	8485 (2)	3342 (10)	-444 (3)	771 (12)
C14	8618 (2)	3913 (8)	915 (2)	666 (9)
C15	9098 (3)	4881 (7)	-1161 (4)	1322 (27)
C16	6240 (2)	1290 (7)	1976 (3)	649 (8)
C17	6439 (4)	-1260 (9)	2041 (6)	1033 (18)
C18	5153 (2)	1698 (10)	1550 (4)	894 (14)

* Coordinate fixed to define origin.

Table 2. Bond distances (\AA), bond angles ($^\circ$) and selected torsion angles ($^\circ$)

O1—C2	1.378 (5)	C7—C8	1.526 (4)
O2—C7	1.427 (30)	C9—C10	1.520 (4)
O3—C8	1.213 (5)	C9—C14	1.512 (5)
O4—C8	1.319 (3)	C10—C11	1.535 (4)
O4—C9	1.466 (3)	C10—C16	1.535 (5)
C1—C2	1.393 (5)	C11—C12	1.517 (6)
C1—C6	1.382 (5)	C12—C13	1.526 (4)
C1—C7	1.521 (4)	C13—C14	1.526 (4)
C2—C3	1.393 (5)	C13—C15	1.516 (8)
C3—C4	1.370 (6)	C16—C17	1.523 (7)
C4—C5	1.375 (7)	C16—C18	1.526 (4)
C5—C6	1.394 (5)		
C8—O4—C9	117.8 (2)	O3—C8—O4	124.0 (2)
C6—C1—C7	119.7 (2)	O4—C9—C10	108.2 (2)
C2—C1—C7	122.1 (2)	O4—C9—C14	107.5 (2)
C2—C1—C6	118.2 (2)	C10—C9—C14	112.6 (2)
O1—C2—C1	122.7 (2)	C9—C10—C16	113.8 (2)
C1—C2—C3	120.5 (3)	C9—C10—C11	107.2 (2)
O1—C2—C3	116.8 (3)	C11—C10—C16	116.1 (2)
C2—C3—C4	120.4 (4)	C10—C11—C12	111.7 (2)
C3—C4—C5	120.0 (3)	C11—C12—C13	111.9 (2)
C4—C5—C6	119.8 (3)	C12—C13—C15	111.9 (3)
C1—C6—C5	121.2 (3)	C12—C13—C14	108.8 (2)
O2—C7—C1	111.5 (2)	C14—C13—C15	111.8 (2)
C1—C7—C8	108.6 (2)	C9—C14—C13	111.6 (2)
O2—C7—C8	107.8 (2)	C10—C16—C18	111.4 (3)
O4—C8—C7	111.2 (2)	C10—C16—C17	113.3 (3)
O3—C8—C7	124.8 (2)	C17—C16—C18	109.4 (3)
C6—C1—C7—C8	-91.1 (3)	C8—O4—C9—C10	139.3 (3)
C2—C1—C7—C8	86.5 (4)	C8—O4—C9—C14	-98.8 (3)
C6—C1—C7—O2	150.3 (3)	C1—C7—C8—O4	73.3 (3)
C2—C1—C7—O2	-32.0 (4)	C1—C7—C8—O3	-105.2 (3)
O2—C7—C8—O4	-165.8 (2)	O4—C9—C10—C16	-53.7 (3)
O2—C7—C8—O3	15.7 (4)		

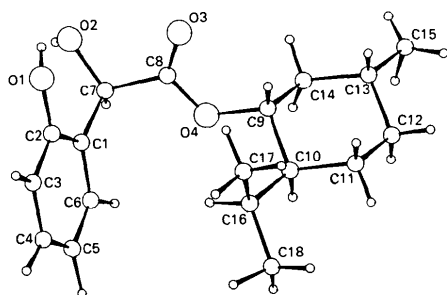


Fig. 1. Perspective view of the molecule.

check reflection monitored every 50 reflections showed no significant crystal movement or decay. Data were corrected for Lorentz and polarization effects but not for absorption. Structure solution was by automatic direct methods (Sheldrick, 1986). Full-matrix refinement was via *SHELX76* (Sheldrick, 1976), minimizing $\sum w\Delta F^2$, with heavy atoms anisotropic and H atoms (from ΔF map) isotropic; 298 parameters refined. $(\Delta/\sigma)_{\max} = 0.36$, $\Delta\rho_{\min/\max} = -0.15/0.19 \text{ e \AA}^{-3}$. Final $R = 0.044$, $wR = 0.052$ with $w = 1.0/(\sigma^2 F + 0.00433 F^2)$. Scattering factors were inlaid (Sheldrick, 1976). The final non-H atomic coordinates with their equivalent isotropic temperature factors (Hamilton, 1959) are given in Table 1. Table 2 lists bond lengths, bond angles and selected torsion angles of the molecule illustrated in Fig. 1.* All calculations were performed on an IBM PS2/80 personal computer with the *CRYSRULER* package (Rizzoli, Sangermano, Calestani & Andreotti, 1987).

Related literature. This structure determination is part of a general program aimed at developing coordinated Friedel–Crafts reactions of phenols and, in particular, on stereocontrolled electrophilic aromatic substitution. Only two structures of compounds similar to the title compound have been reported previously (Bigi, Casiraghi, Casnati, Sartori, Soncini, Fava Gasparri & Ferrari Belicchi, 1985; Casiraghi, Bigi, Casnati, Sartori, Soncini, Fava Gasparri & Ferrari Belicchi, 1988).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55207 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA0237]

References

- BELLETTI, D., CANTONI, A. & PASQUINELLI, G. (1988). *Gestione On-Line di Diffratometro a Cristallo Singolo Siemens AED con Sistema IBM PS2/30*. Internal report 1/88. Centro di Studio per la Strutturistica Diffratometrica del CNR, Parma, Italy.
- BIGI, F., CASIRAGHI, G., CASNATI, G., SARTORI, G., SONCINI, P., FAVA GASPARRI, G. & FERRARI BELICCHI, M. (1985). *Tetrahedron Lett.* **26**, 2021–2024.
- CASIRAGHI, G., BIGI, F., CASNATI, G., SARTORI, G., SONCINI, P., FAVA GASPARRI, G. & FERRARI BELICCHI, M. (1988). *J. Org. Chem.* **53**, 1779–1785.
- HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.
- LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* **A30**, 580–589.
- RIZZOLI, C., SANGERMANO, V., CALESTANI, G. & ANDRETTI, G. D. (1987). *J. Appl. Cryst.* **20**, 436–439.
- SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- SHELDRICK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.