Stereochemistry of Arylglycerol β-Aryl Ethers. Crystal Structure of *erythro*-3-Hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic Acid

Anders Johansson,[a] Knut Lundquist[a] and Rolf Stomberg[b]

[a] Department of Organic Chemistry and [b] Department of Inorganic Chemistry, Chalmers University of Technology and University of Göteborg, S-412 96 Göteborg, Sweden

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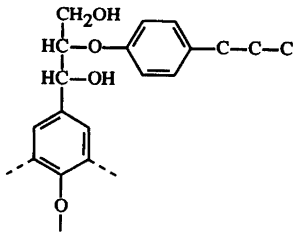
The *threo* and *erythro* forms of 3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid have been synthesized. Reduction with borane–dimethyl sulfide complex gave the *threo* and *erythro* forms of the lignin model 2-phenoxy-1-phenyl-1,3-propanediol. The steric assignments were derived from a crystal structure determination of *erythro*-3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid by single-crystal X-ray diffraction methods. The compound crystallizes as colourless prisms in the triclinic space group $P\bar{1}$ with a = 9.785(1), b = 13.369(2), c = 5.768(1) Å, α = 101.74(1), β = 92.43(1), γ = 102.77(1) °, V = 717.4(1) Å^3 and Z = 2. A final R-value of 0.035 was obtained.

The structure, stereochemistry and conformation of a series of lignin model compounds representative of structural elements in lignins of the β-ether type have been discussed in previous papers[1–5] on the basis of crystal structure determinations. Compounds hitherto investigated have been of the β-syringyl ether or β-guaiacyl ether types. Minor amounts of structural elements of an additional type of β-ether are assumed to be present in lignins, namely those of type **1**. Their proportion may be very small since it has been shown that only a minor fraction of the lignin units lacking methoxy groups are etherified.[6] The biosynthesis of structural elements of type **1** is expected to involve an enzymic oxidation of *p*-coumaryl alcohol. Experimental evidence for this was obtained by Nakatsubo and Higuchi[7] who isolated a mixture of the diastereomers of **2** from the dehydrogenation product obtained on enzymic oxidation of *p*-coumaryl alcohol. They assigned the *erythro* and *threo* forms of **2** on the basis of ^{1}H NMR spectral comparison with arylglycerol β-aryl ethers of known configuration. To obtain an independent basis for the assignment of diastereomers of compounds related to lignin structures of type **1** we have prepared the diastereomers of 2-phenoxy-1-phenyl-1,3-propanediol by reduction of diastereomers of 3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid of known configuration. The assignments of the *erythro* (**3a**) and *threo* (**3b**) forms of 2-phenoxy-1-phenyl-1,3-propanediol were deduced on the basis of a crystal structure determination of one of the synthetic intermediates, *erythro*-3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid (**4a**). Attempts were also made to determine the crystal structure of **3a** which was obtained in the crystalline state (m.p. 111–112 °C). Although the molecules of **3a** in the crystals investigated seem to be disordered, a prelimi-



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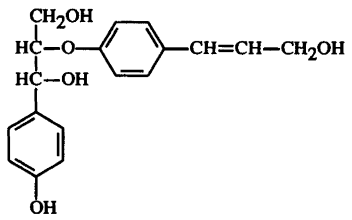

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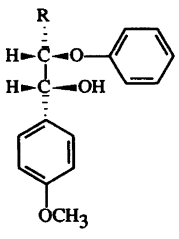

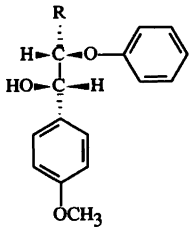

3a R=CH_2OH **3b** R=CH_2OH

4a R=COOH **4b** R=COOH

Table 1. Atomic fractional coordinates and B_{eq} (B_{iso} for H) for *erythro*-3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid (**4a**), $C_{16}H_{16}O_5$. T = 296 K. $B_{eq} = \frac{4}{3} \Sigma_i \Sigma_j \beta_{ij} \boldsymbol{a}_i \cdot \boldsymbol{a}_j$.

Atom	*x*	*y*	*z*	B_{eq}
C(1)	0.7799(2)	0.8676(1)	0.0485(3)	2.97(6)
C(2)	0.7506(2)	0.8154(2)	−0.1860(3)	3.60(7)
C(3)	0.8532(2)	0.7785(2)	−0.3152(4)	4.01(8)
C(4)	0.9870(2)	0.7955(1)	−0.2067(4)	3.91(7)
C(5)	1.0186(2)	0.8510(2)	0.0246(4)	4.61(9)
C(6)	0.9164(2)	0.8863(2)	0.1506(4)	3.97(7)
C(7)	1.0656(4)	0.6873(3)	−0.5296(6)	6.8(1)
C(8)	0.6720(2)	0.9083(1)	0.1970(3)	2.93(6)
C(9)	0.5251(2)	0.8329(1)	0.1513(3)	2.90(6)
C(10)	0.4259(2)	0.8784(1)	0.3158(3)	2.84(6)
C(11)	0.4296(2)	0.6510(1)	0.1438(3)	3.51(7)
C(12)	0.4348(3)	0.5720(2)	0.2579(6)	6.7(1)
C(13)	0.3283(4)	0.4822(2)	0.2108(6)	8.2(1)
C(14)	0.2175(3)	0.4707(2)	0.0545(6)	7.0(1)
C(15)	0.2095(3)	0.5503(2)	−0.0543(6)	7.2(1)
C(16)	0.3157(2)	0.6418(2)	−0.0114(4)	5.5(1)
O(1)	1.0964(2)	0.7608(1)	−0.3118(3)	5.96(7)
O(2)	0.6612(1)	1.0100(1)	0.1683(2)	3.47(5)
O(3)	0.5439(1)	0.73652(9)	0.1964(2)	3.45(5)
O(4)	0.3594(1)	0.9386(1)	0.2658(2)	3.68(5)
O(5)	0.4239(1)	0.8500(1)	0.5207(2)	3.75(5)
H(C2)	0.661(2)	0.802(1)	−0.262(3)	3.5(4)
H(C3)	0.827(2)	0.742(2)	−0.469(4)	4.5(5)
H(C5)	1.112(2)	0.865(2)	0.097(4)	5.5(5)
H(C6)	0.938(2)	0.922(2)	0.319(4)	4.9(5)
H1(C7)	0.994(4)	0.628(3)	−0.502(6)	11(1)
H2(C7)	1.022(3)	0.713(3)	−0.655(6)	11(1)
H3(C7)	1.153(3)	0.664(2)	−0.555(5)	8.4(7)
H(C8)	0.704(2)	0.918(1)	0.363(3)	2.7(3)
H(C9)	0.487(2)	0.826(1)	−0.009(3)	2.3(3)
H(C12)	0.517(3)	0.577(2)	0.346(5)	7.8(7)
H(C13)	0.342(3)	0.430(3)	0.289(6)	11(1)
H(C14)	0.149(3)	0.410(2)	0.030(5)	7.9(7)
H(C15)	0.123(3)	0.548(2)	−0.155(6)	9.3(8)
H(C16)	0.305(2)	0.702(2)	−0.084(4)	6.3(6)
H(O2)	0.658(2)	1.013(2)	0.019(4)	5.0(5)
H(O5)	0.383(3)	0.895(2)	0.623(5)	7.4(7)

nary structure determination supports the *erythro* configuration.

Crystal structure of erythro-*3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid* (**4a**). Atomic fractional coordinates and thermal parameters are given in Table 1. Table 2 lists bond distances, bond angles and selected torsion angles. Fig. 1 shows a stereoscopic view of the molecule and the atomic numbering is shown in Fig. 2.

The crystals of *erythro*-3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid (**4a**), consist of monomeric molecules held together by van der Waals forces and rather strong hydrogen bonds (Table 2, Fig. 3). The shortest intermolecular contact distance other than those concerned with the bond distances listed in Table 2 is 2.47(2) Å [between O(4) and H(C5)].

It appears in Figs. 1 and 2 that the compound examined has the *erythro* configuration. All bond distances and bond angles are normal (see Table 2). Average bond distances

Table 2. Bond distances (Å) and angles (°) in *erythro*-3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid (**4a**), $C_{16}H_{16}O_5$. The average C–H and O–H bond distances are 0.96(3) and 0.90(3) Å, respectively.

Bond distances			
C(1)–C(2)	1.376(3)	C(9)–C(10)	1.522(2)
C(1)–C(6)	1.386(3)	C(9)–O(3)	1.414(2)
C(1)–C(8)	1.512(2)	C(10)–O(4)	1.211(2)
C(2)–C(3)	1.392(3)	C(10)–O(5)	1.313(2)
C(3)–C(4)	1.378(3)	C(11)–C(12)	1.362(3)
C(4)–C(5)	1.374(3)	C(11)–C(16)	1.367(3)
C(4)–O(1)	1.374(2)	C(11)–O(3)	1.384(2)
C(5)–C(6)	1.371(3)	C(12)–C(13)	1.376(4)
C(7)–O(1)	1.403(3)	C(13)–C(14)	1.341(4)
C(8)–C(9)	1.540(2)	C(14)–C(15)	1.355(4)
C(8)–O(2)	1.429(2)	C(15)–C(16)	1.390(3)
Bond angles			
C(2)–C(1)–C(6)	118.0(2)	C(10)–C(9)–O(3)	111.7(1)
C(2)–C(1)–C(8)	123.3(2)	C(9)–C(10)–O(4)	123.0(1)
C(6)–C(1)–C(8)	118.6(2)	C(9)–C(10)–O(5)	113.8(1)
C(1)–C(2)–C(3)	121.3(2)	O(4)–C(10)–O(5)	123.2(2)
C(2)–C(3)–C(4)	119.4(2)	C(12)–C(11)–C(16)	119.6(2)
C(3)–C(4)–C(5)	119.7(2)	C(12)–C(11)–O(3)	115.9(2)
C(3)–C(4)–O(1)	125.1(2)	C(16)–C(11)–O(3)	124.6(2)
C(5)–C(4)–O(1)	115.1(2)	C(11)–C(12)–C(13)	120.1(3)
C(4)–C(5)–C(6)	120.3(2)	C(12)–C(13)–C(14)	121.1(3)
C(1)–C(6)–C(5)	121.2(2)	C(13)–C(14)–C(15)	119.1(3)
C(1)–C(8)–C(9)	113.5(1)	C(14)–C(15)–C(16)	121.1(3)
C(1)–C(8)–O(2)	112.2(1)	C(11)–C(16)–C(15)	118.9(2)
C(9)–C(8)–O(2)	109.6(1)	C(4)–O(1)–C(7)	118.0(2)
C(8)–C(9)–C(10)	109.5(1)	C(9)–O(3)–C(11)	117.8(1)
C(8)–C(9)–O(3)	106.0(1)		
Hydrogen bonds			
O(2)···O(5)	(1−*x*, 2−*y*, 1−*z*)	2.620(2)	O(2)···H(O5) 1.73(3)
O(2)···O(4)	(1−*x*, 2−*y*, −*z*)	2.738(2)	O(4)···H(O2) 1.90(2)
Selected torsion angles			
C(1)–C(8)–C(9)–C(10)		177.6(1)	
C(1)–C(8)–C(9)–O(3)		56.9(2)	
C(1)–C(8)–C(9)–H(C9)		−64(1)	
C(1)–C(8)–O(2)–H(O2)		46(1)	
C(2)–C(1)–C(8)–C(9)		39.7(2)	
C(2)–C(1)–C(8)–O(2)		−85.3(2)	
C(2)–C(1)–C(8)–H(C8)		159(1)	
C(3)–C(4)–O(1)–C(7)		−11.9(3)	
C(8)–C(9)–C(10)–O(4)		87.3(2)	
C(8)–C(9)–C(10)–O(5)		−89.9(2)	
C(8)–C(9)–O(3)–C(11)		−173.2(1)	
C(9)–C(8)–O(2)–H(O2)		−81(1)	
C(9)–C(10)–O(5)–H(O5)		164(2)	
C(9)–O(3)–C(11)–C(12)		−158.9(2)	
C(10)–C(9)–C(8)–O(2)		−56.0(2)	
C(10)–C(9)–C(8)–H(C8)		58(1)	
C(10)–C(9)–O(3)–C(11)		67.5(2)	
C(11)–O(3)–C(9)–H(C9)		−53(1)	
O(2)–C(8)–C(9)–O(3)		−176.7(1)	
O(2)–C(8)–C(9)–H(C9)		62(1)	
O(3)–C(9)–C(8)–H(C8)		−63(1)	
O(3)–C(9)–C(10)–O(4)		−155.5(2)	
O(3)–C(9)–C(10)–O(5)		27.3(2)	
O(4)–C(10)–C(9)–H(C9)		−32(1)	
O(4)–C(10)–O(5)–H(O5)		−13(2)	
O(5)–C(10)–C(9)–H(C9)		151(1)	
H(C8)–C(8)–C(9)–H(C9)		176(1)	
H(C8)–C(8)–O(2)–H(O2)		163(2)	

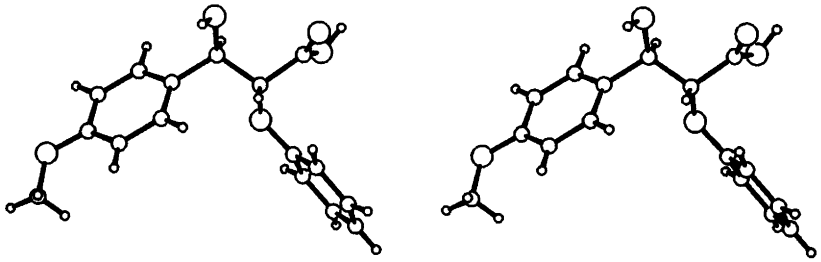

Fig. 1. Stereoscopic view[12] of a molecule of *erythro*-3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid (**4a**).

are (r.m.s. deivations are given in parentheses): $C(sp^2)$ – $C(sp^2)$ (in the benzene rings) 1.372(14) Å, $C(sp^2)_{aromatic}$ – O, 1.379(5) Å and $C(sp^3)$ – O, 1.415(11) Å. Within the limits of experimental error, the ring carbon atoms are situated in the aromatic ring planes, mean deviations being close to 0.01 Å. The benzylic carbon atom [C(8)] is almost coplanar with the aromatic ring plane [the deviation is 0.018(3) Å]. The oxygen atom in the β-ether linkage is slightly twisted [0.045(3) Å] out of the benzene ring plane. The angle between the aromatic ring planes is 88.6(3)°. The maximum deviation of C(9), C(10), O(4) and O(5) from the plane which they define, is 0.015(3) Å. H(O5) is 0.23(3) Å out of this plane. The torsion angle C(1)–C(8)–C(9)–O(3) is 57° in **4a**. In the related propanoic acid derivative *threo*-2-(2,6-dimethoxyphenoxy)-3-(3,4-dimethoxyphenyl)-3-hydroxypropanoic acid, the corresponding torsion angle (denoted C(4)–C(9)–C(10)–O(6) in Ref. 5) is close to 180°.[5] The difference may be due to the existence of an intramolecular hydrogen bond in this latter compound between the oxygen atom in the hydroxy group [denoted O(3) in Ref. 5] and one of the methoxy oxygen atoms [denoted O(7) in Ref. 5] in the 2,6-dimethoxyphenoxy group [O(3···O(7) is 2.907(3) Å and the angle O(3)–H(O3)···O(7) is 162°]. In **4a** there is no intramolecular hydrogen bond (Fig. 3).

Experimental

NMR spectra. ^1H NMR spectra (400 MHz) and ^{13}C NMR spectra (100.6 MHz) were recorded with a Varian VXR-5000 instrument (temperature, 300 K). Deuteriochloroform was used as the solvent (internal reference, TMS).

Thin layer chromatography (TLC) was performed on silica gel plates (Merck, Kieselgel 60 F_{254}) with dichloromethane–ethyl acetate–acetic acid (7:2:1) as the eluent. Spots were made visible with UV light and by spraying with formalin–H_2SO_4 (1:9) subsequent heating. R_f values: **4b**, 0.29; **4a**, 0.39.

Preparation of the erythro (**4a**) *and* threo (**4b**) *forms of 3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid.* A mixture of acids **4a** and **4b** was obtained according to a procedure previously described[8] for the synthesis of analogous compounds. A crude product (ca. 7 g) was obtained from anisaldehyde (3.0 g) and phenoxyacetic acid (3.4 g). About equal amounts of **4a** and **4b** were present in the reaction mixture (^1H NMR). Purification/separation of **4a** and **4b** was accomplished by flash chromatography on silica gel (200 g; Merck Kieselgel 60, 230–400 mesh) using dichloromethane–ethyl acetate–acetic acid (14:4:1) as the eluent. Effluent fractions were combined on the basis of TLC examinations. Eluate 460–530 ml gave a crystalline product (0.81 g) consisting of **4a** contaminated by phenoxyacetic

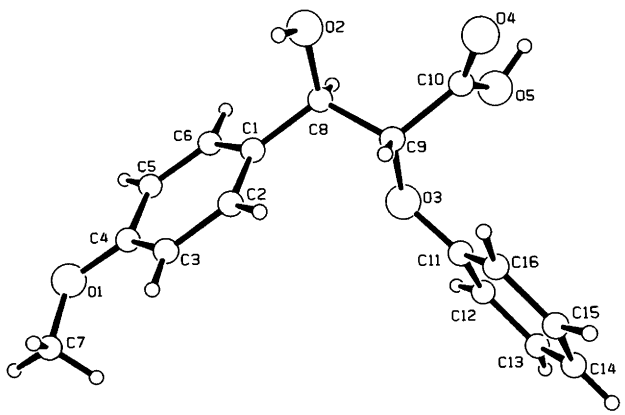

Fig. 2. A perspective drawing[12] of the *erythro*-3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid (**4a**) molecule showing the atomic numbering.

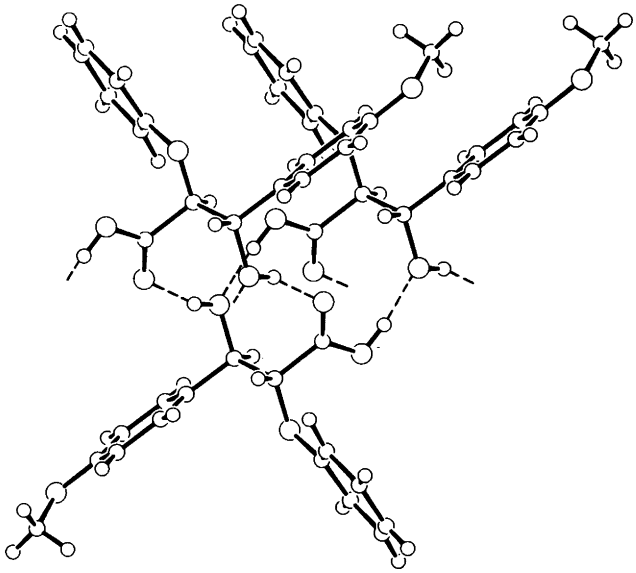

Fig. 3. The hydrogen bonding in *erythro*-3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid (**4a**). The connections between three neighbouring molecules are shown.

Table 3. Crystal and experimental data for *erythro*-3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid (**4a**), $C_{16}H_{16}O_5$.

Crystal data	
Formula weight	288.30
Crystal colour; habit	Colourless; prism
Crystal dimensions (mm)	0.27×0.35×0.44
Crystal system	Triclinic
No. of reflections used for unit cell determination (2θ range; °)	25 (36.6–41.2)
Omega scan peak width at half-height	0.19
Lattice parameters	a = 9.785(1) Å b = 13.369(2) Å c = 5.768(1) Å α = 101.74(1) ° β = 92.43(1) ° γ = 102.77(1) ° V = 717.4(1) Å^3
Space group	$P\bar{1}$ (No. 2)
Z	2
D_{calc} (g cm^{-3})	1.334
F(000)	304
μ(Mo K_α)/cm^{-1}; no correction	0.93
Intensity measurements	
Diffractometer	Rigaku AFC6R
Radiation	Mo K_α (λ = 0.71069 Å)
Temperature (°C)	23
Scan type	ω–2θ
Scan rate (°/min)	4.0 (maximum of 3 rescans)
Scan width (°)	1.47 + 0.30 θ
2θ range (°)	3.5–50.0
No. of reflections measured	Total: 2678 Unique: 2517 (R_{int} = 0.012)
Corrections	Lorentz polarization
Structure solution and refinement	
Structure solution	Direct methods. Electron density difference maps
Hydrogen atom treatment	Refined
Refinement	Full-matrix least-squares
Function minimized	$\Sigma w(\|F_o\|-\|F_c\|)^2$
Least-squares weights	$w = 1/\sigma^2(F_o)$
Anomalous dispersion	All non-hydrogen atoms
No. of observed independent reflections [I>3.00σ(I)]	1859
No. of variables	255
Reflection/parameter ratio	7.29
Residuals: R; R_w	0.035; 0.047
Goodness-of-fit indicator	2.09
Maximum shift/error in final cycle	0.02
Maximum peak in final diff. map (e$^-$ Å^{-3})	0.19
Minimum peak in final diff. map (e$^-$ Å^{-3})	−0.15

acid (^{1}H NMR). Recrystallization from ethyl acetate gave pure crystals of **4a** (0.18 g) melting at 128–131 °C. Eluate 530–970 ml gave a mixture of **4a** and **4b** (4.1 g). Eluate 970–1120 ml gave **4b** (0.14 g). ^{1}H NMR of **4a**: δ 3.78 (3 H, s; OCH_3), 4.78 (1 H, d, J = 4.8 Hz; Hβ), 5.16 (1 H, d, J = 4.8 Hz; Hα), 6.80–7.5 (9 H, m; aromatic protons). ^{1}H NMR of **4b**: δ 3.78 (3 H, s; OCH_3), 4.74 (1 H, s, d, J = 3.4 Hz; Hβ), 5.18 (1 H, d, J = 3.4 Hz; Hα), 6.74–7.39 (9 H, m; aromatic protons).

Preparation of the erythro (**3a**) *and* threo (**3b**) *forms of 2-phenoxy-1-phenyl-1,3-propanediol.* Acids **4a** and **4b** (or mixtures of these two compounds) were reduced with borane–dimethyl sulfide complex according to a previously described procedure.[8] The *erythro* form (**3a**) and the *threo* form (**3b**) of 2-phenoxy-1-phenyl-1,3-propanediol could be separated by ion-exchange chromatography on an anion exchanger using a borate solution as the eluent (cf. Ref. 8); the *threo* form was eluted before the *erythro* form.

^{1}H NMR spectrum of the acetate derivative of **3a**: δ 2.01 (3 H, s; $COCH_3$), 2.05 (3 H, s; $COCH_3$), 3.79 (3 H, s; OCH_3), 4.20 (1 H, dd, J = 4.0 and 11.8 Hz; Hγ), 4.73 (1 H, m; Hβ), 5.99 (1 H, d, J = 4.9 Hz; Hα), 6.84–7.38 (9 H, m; aromatic protons). ^{13}C NMR spectrum of the acetate derivative of **3a**: δ 20.9 (CH_3–C), 21.2 (CH_3–C), 55.5 (CH_3O), 62.8 (Cγ), 74.1 (Cα), 78.7 (Cβ), 110–180 [114.1 (2 C), 117.1 (2 C), 122.2 (1 C), 128.5 (1 C), 128.9 (2 C), 129.7 (2 C), 158.4 (1 C), 159.9 (1 C); aromatic carbon], 169.8 (CO, benzylic acetate), 170.9 (CO, acetate at Cγ).

^{1}H NMR spectrum of the acetate derivative of **3b**: δ 1.98 (3 H, s; $COCH_3$), 2.00 (3 H, s; $COCH_3$), 3.79 (3 H, s; OCH_3), 3.99 (1 H, dd, J = 7.3 and 11.9 Hz; Hγ), 4.22 (1 H, dd, J = 3.8 and 11.9 Hz; Hγ), 4.72 (1 H, m; Hβ), 6.04 (1 H, d, J = 7.0 Hz; Hα), 6.83–7.37 (9 H, m; aromatic protons). ^{13}C NMR spectrum of the acetate derivative of **3b**: δ 20.8 (CH_3–C), 21.2 (CH_3–C), 55.4 (CH_3O), 63.2 (Cγ), 74.6 (Cα), 78.6 (Cβ), 110–180 [114.3 (2 C), 116.7 (2 C), 122.0 (1 C), 128.4 (1 C), 128.8 (2 C), 129.7 (2 C), 158.8 (1 C), 160.0 (1 C); aromatic carbon], 170.0 (CO, benzylic acetate), 170,7 (CO, acetate at Cγ).

Determination of the crystal structure of erythro-*3-hydroxy-3-(4-methoxyphenyl)-2-phenoxypropanoic acid.* Crystal data and conditions for the data collection are given in Table 3. Rotation and Weissenberg photographs (Cu K_α radiation) were taken of the X-ray diffraction patterns. From these, symmetry and approximate cell dimensions were derived. Intensity data were recorded with a Rigaku AFC6R X-ray diffractometer with graphite-monochromated Mo K_α radiation from a RU200 rotating anode source operated at 9 kW (50 kV; 180 mA). The weak reflections [I<10.0σ(I)] were rescanned 4.35 (1 H, dd, J = 6.3 and 11.8 Hz; Hγ), and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 0.5 mm and the crystal-to-detector distance was 40 cm. Five very strong reflections were measured with an attenuating filter (1 mm Al) in front of the detector and were scaled to the level of the rest of the data set.

The intensities of three representative reflections, which were measured after every 150 reflections, remained constant throughout the data collection indicating crystal and

electronic stability (no decay correction was applied). Azimuthal scans of several reflections indicated no need for an absorption correction. The intensities were corrected for Lorentz and polarization effects. The unit cell dimensions were determined from a least-squares fit of refined diffratometer setting angles for 25 reflections.

The structure was solved by direct methods using the program SHELXS[9] and by electron density calculations. The SHELXS solution gave the positions of all non-hydrogen atoms. A full-matrix least-squares refinement of positional and anisotropic thermal parameters for these atoms was performed. All hydrogen atoms were located in the subsequent electron density difference map. Inclusion of positional and isotropic thermal parameters for the hydrogen atoms in the refinement reduced R to 0.035 ($R = \Sigma||F_o|-|F_c||/\Sigma|F_o|$). The weighting scheme (see Table 3) was based on counting statistics. Plots of $\Sigma w(|F_o|-|F_c|)^2$ versus $|F_o|$, reflection order in data collection, $\sin\theta/\lambda$ and various classes of index showed no unusual trends.

Some further details concerning the refinement of the structure are given in Table 3. Neutral atomic scattering factors as well as $\Delta f'$ and $\Delta f''$ were taken from *International Tables for X-Ray Crystallography*.[10] Calculations were carried out on a VAX computer using the TEXSAN crystallographic software package of Molecular Structure Corporation.[11]

Tables of U-values and structure factor tables are available from the author (R.S.) on request.

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