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(*RS*)-2,6-Dimethoxy-4-(2-methoxypropyl)phenol

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.007 Å
R factor = 0.057
wR factor = 0.164
Data-to-parameter ratio = 8.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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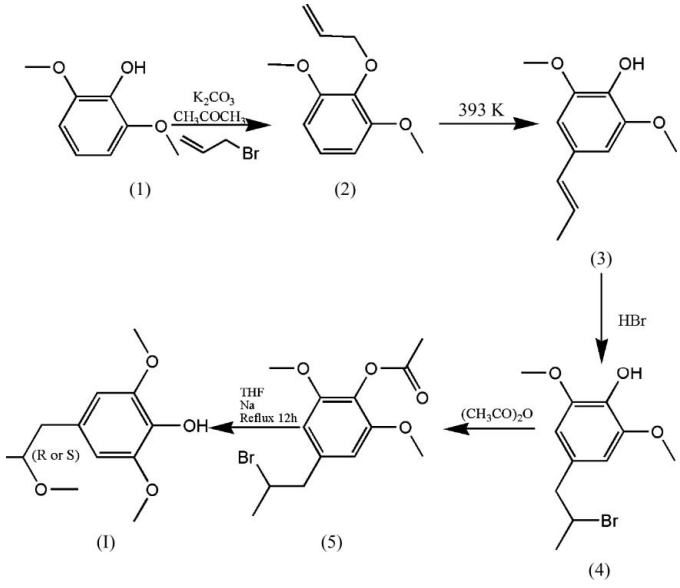
The title compound, $C_{12}H_{18}O_4$, crystallizes with two molecules in the asymmetric unit. The structure features O—H···O hydrogen-bonded double chains.

Comment

The mechamism of phenol oxidation by tyrosinase has been well studied (Krol & Bolton, 1997). However, little work has been done to determine the influence of substituents on the reaction. Recently, we have synthesized the title compound, (I), and its crystal structure is reported here.



Compound (I) crystallizes in the non-centrosymmetric space group *Pc*, with two independent molecules in the asymmetric unit. In the molecular structure (Fig. 1 and Table 1), the two molecules have similar geometric parameters about the two chiral centres, C10 and C22; the bond lengths and angles are unremarkable. The crystal structure is stabilized by intermolecular O—H···O hydrogen bonds, with the phenol groups acting as donors and the ether O atoms as acceptors (Table 2). These hydrogen bonds link the molecules into double chains (Fig. 2).

Experimental

2,6-Dimethoxyphenol, (1), was synthesized according to the literature procedure of Wu *et al.* (1997). To a basic acetone solution of 2,6-dimethoxyphenol (0.35 g, 2 mmol) was added K_2CO_3 (0.14 g); a solution of 3-bromoprop-1-ene (15 ml) in acetone was then added dropwise. The mixture was stirred at 308 K for 5 h, filtered and

purified by column chromatography on silical gel, using petroleum–ethyl acetate (3:2 *v*/*v*), to give compound (2) (0.30 g, 1.5 mmol). Product (2) (0.19 g, 1 mmol) in tetrahydrofuran (THF, 20 ml) was refluxed for 1 h at 393 K and then cooled to room temperature. To the solution was added hydrobromic acid (5 ml, 6 *M*) and the mixture stirred for 1 h. Removal of the solvent under reduced pressure and subsequent purification by flash chromatography (using acetone as eluent) afforded compound (4) (0.15 g, 0.5 mmol). The hydroxyl group of compound (4) was protected with acetic andydride, yielding compound (5). To a solution of (5) in THF (20 ml) was added sodium (0.07 g) and the mixture was refluxed for 12 h at 323 K to afford the title compound, (I). Crystals suitable for X-ray analysis were obtained by recrystallization from ethanol.

Crystal data

$C_{12}H_{18}O_4$	D_x = 1.190 Mg m^{-3}
M_r = 226.26	Mo $K\alpha$ radiation
Monoclinic, *Pc*	Cell parameters from 2536 reflections
a = 13.740 (3) Å	θ = 2.6–25.4°
b = 9.405 (2) Å	μ = 0.09 mm^{-1}
c = 9.997 (2) Å	*T* = 292 (2) K
β = 102.190 (4)°	Block, colorless
V = 1262.8 (5) Å^3	0.30 × 0.20 × 0.10 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1809 reflections with $I > 2\sigma(I)$
	R_{int} = 0.081
ω scans	θ_{max} = 26.0°
Absorption correction: none	*h* = −16 → 16
6654 measured reflections	*k* = −11 → 6
2481 independent reflections	*l* = −12 → 11

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.057	$w = 1/[\sigma^2(F_o^2) + (0.1154P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.164	$(\Delta/\sigma)_{max} < 0.001$
S = 0.97	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
2481 reflections	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
299 parameters	

Table 1
Selected geometric parameters (Å, °).

C9—C10	1.502 (6)	C21—C22	1.501 (8)
C10—O4	1.437 (5)	C22—O8	1.431 (6)
C10—C11	1.491 (7)	C22—C23	1.555 (10)
O4—C10—C11	111.5 (4)	O8—C22—C21	105.3 (4)
O4—C10—C9	105.4 (3)	O8—C22—C23	112.4 (5)
C11—C10—C9	113.1 (4)	C21—C22—C23	111.8 (5)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1	0.82	2.26	2.676 (4)	112
O2—H2···O8^{i}	0.82	2.05	2.774 (5)	148
O6—H6*A*···O5	0.82	2.25	2.661 (4)	111
O6—H6*A*···O4^{ii}	0.82	2.05	2.823 (4)	156

Symmetry codes: (i) $x+1, y, z$; (ii) $x, y-1, z+1$.

H atoms were positioned geometrically (methyl C—H = 0.96 Å, methylene C—H = 0.97 Å, methine C—H = 0.98 Å, aromatic C—H = 0.93 Å and hydroxy O—H = 0.82 Å) and included in the refinement in the riding-model approximation, with U_{iso}(H) = xU_{eq}(carrier atom), where x = 1.5 for methyl and hydroxy, and x = 1.2 for the others. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

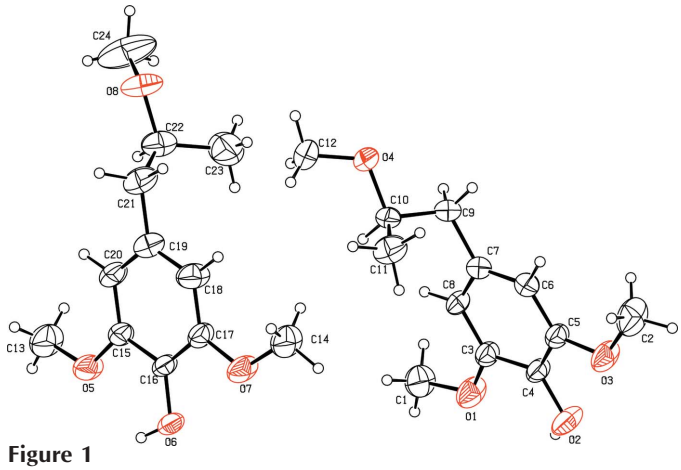


Figure 1
The molecular structure of the asymmetric unit of the title compound, showing 30% probablity displacement ellipsoids.

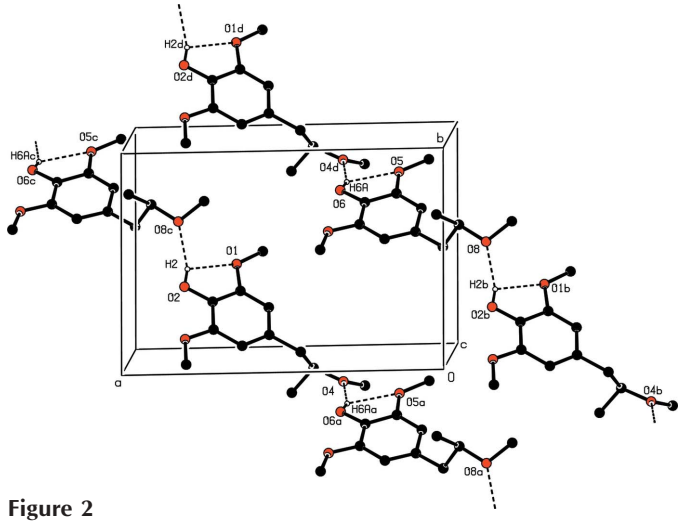


Figure 2
Plot of the crystal packing, showing the linkage of molecules by O—H···O hydrogen bonding (dashed lines). Labels *a*, *b*, *c*, *d* correspond to the symmetry positions $(x, -1+y, z)$, $(-1+x, y, -1+z)$, $(x+1, y, z+1)$ and $(x, y+1, z)$, respectively.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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