

Vratislav Langer,^{a*} Shiming Li^b
and Knut Lundquist^c^aDepartment of Environmental Inorganic Chemistry, Chalmers University of Technology, SE-41296 Göteborg, Sweden, ^bDepartment of Organic Chemistry, Chalmers University of Technology, SE-41296 Göteborg, Sweden, and ^cDepartment of Forest Products and Chemical Engineering, Chalmers University of Technology, SE-41296 Göteborg, SwedenCorrespondence e-mail:
langer@inoc.chalmers.se

Key indicators

Single-crystal X-ray study
T = 183 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.056
wR factor = 0.133
Data-to-parameter ratio = 24.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.*erythro*-2-(2,6-Dimethoxyphenoxy)-1-(4-hydroxy-3,5-dimethoxyphenyl)-1,3-propanediol

In the crystals of the title compound, $\text{C}_{19}\text{H}_{24}\text{O}_8$, the molecules adopt a conformation in which the bulky 2,6-dimethoxyphenoxy and 4-hydroxy-3,5-dimethoxyphenyl groups are distant from each other. The $\text{O}(\text{phenoxy})-\text{C}-\text{C}-\text{C}(\text{phenyl})$ torsion angle between these groups is $-177.27(10)^\circ$. The conformation is compared with those of related lignin model compounds. The hydrogen-bonding pattern is discussed in terms of graph-set theory.

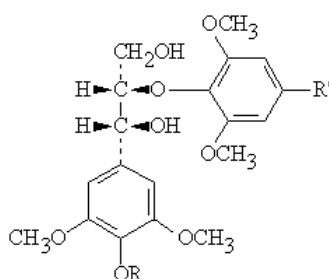
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Comment

Crystal structures of *erythro* forms (1) and (2) of model compounds, representative of structural elements in lignin of the syringylglycerol β -syringyl ether type, have been described previously (Stomberg & Lundquist, 1989; Langer & Lundquist, 2001). The conformations of the compounds differed considerably. The torsion angle between the 1-aryl and the 2-aryloxy groups was, for instance, $-71.0(4)^\circ$ in (1) and $178.58(7)^\circ$ in (2). The conformational differences were attributed to the different hydrogen-bonding patterns (Langer & Lundquist, 2001). To obtain a better basis for judging which factors govern the conformation of syringylglycerol β -syringyl ethers, we have now determined the crystal structure of a third model compound representative of such ethers, *erythro*-2-(2,6-dimethoxyphenoxy)-1-(4-hydroxy-3,5-dimethoxyphenyl)-1,3-propanediol, (3). This compound adopts a conformation in



- 1 R = H, R' = CH_2OH
- 2 R = CH_3 , R' = H
- 3 R = R' = H

which the bulky aromatic groups are distant from each other. The torsion angle $\text{C}_9-\text{C}_{10}-\text{O}_6-\text{C}_{12}$ is $-75.26(13)^\circ$ and the torsion angle $\text{C}_1-\text{C}_9-\text{C}_{10}-\text{O}_6$ is $-177.27(10)^\circ$. This leads to a C_1-C_{12} distance of $4.4458(17) \text{ \AA}$. Corresponding data for (2) are $-89.24(9)^\circ$, $178.58(7)^\circ$ and $4.5030(13) \text{ \AA}$. The angle between the aromatic ring planes is $57.27(5)^\circ$ in (3) and $76.00(3)^\circ$ in (2). It is obvious that (2) and (3) adopt similar conformations. Nevertheless, the hydrogen-bonding patterns of (2) and (3) are different.

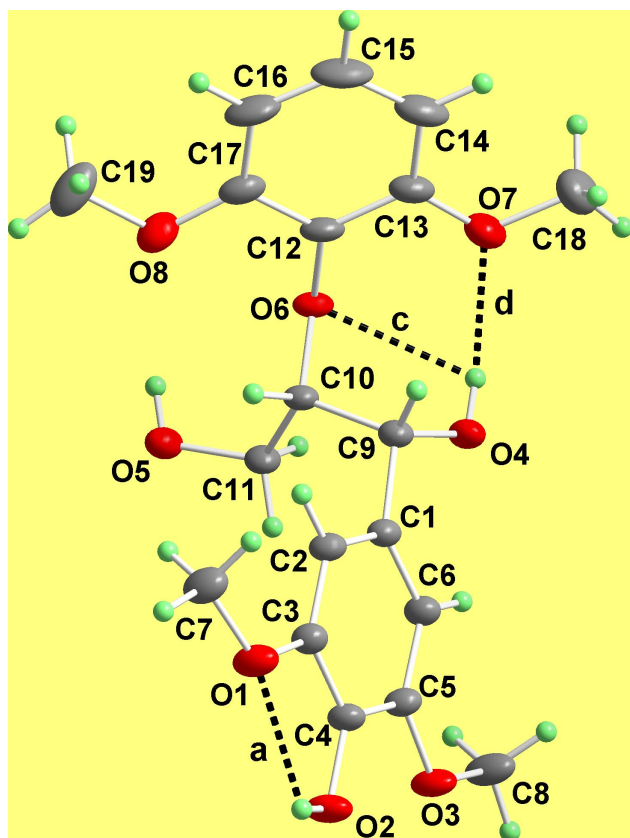


Figure 1
The numbering scheme for the title compound. Displacement ellipsoids are shown at the 50% probability level. Intramolecular contacts are shown by broken lines. For hydrogen-bond codes, see Table 1.

Both structures exhibit intramolecular hydrogen bonds of approximately the same strength, but in (2), they are formed by the hydroxyl-H atom in the methylol group and the O atoms in a methoxy group and the phenoxy group (Langer & Lundquist, 2001), while in (3), they are formed by H(O4) and the atoms O6 and O7 (Fig. 1 and Table 1). The phenolic H(O2) atom, which is lacking in (2), forms an intramolecular hydrogen bond with O1 as acceptor. H(O2) also takes part in intermolecular hydrogen bonds. Assignment of the hydrogen-bond descriptors using program *PLUTO* (Motherwell *et al.*, 1999) in terms of the graph-set theory (Bernstein *et al.*, 1995) has been carried out. For this purpose, we use symbols of type $Xa,d(n)$, where X is the pattern descriptor, a is the number of acceptors, d is the number of donors and n is the number of atoms comprising the pattern. For (3), there are, on the first-level of intermolecular contacts, a chain $C1,1(6)$ formed by hydrogen bonds of type $[e]$ (Fig. 2 and Table 1) and a ring $R2,2(20)$, formed by hydrogen bonds of type $[b]$ (Fig. 3). On the second-level of graph-set descriptors, chains $C2,2(10)$ and $C2,2(16)$ were identified, both formed by hydrogen bonds of types $[b]$ and $[e]$. The structure of (2) exhibits (apart from intramolecular contacts) just one first-level descriptor, a chain $C1,1(6)$ with the benzylic oxygen as donor and the oxygen in the methylol groups as acceptor (Langer & Lundquist, 2001).

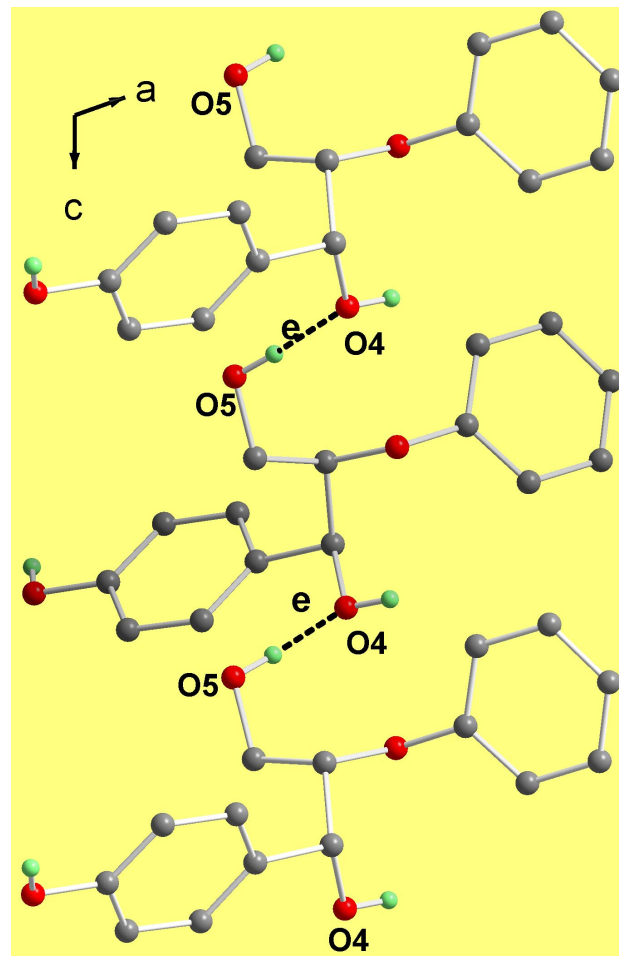


Figure 2
The hydrogen-bond network showing the $C1,1(6)$ chain of molecules. Methoxy groups and carbon-bonded H atoms have been omitted for clarity. For hydrogen-bond codes, see Table 1.

Experimental

The title compound was synthesized following the method described by Li *et al.* (2000); m.p. 390–391 K (from ethyl acetate).

Crystal data

$C_{19}H_{24}O_8$
 $M_r = 380.38$
Monoclinic, $P2_1/c$
 $a = 12.9064$ (5) Å
 $b = 15.6222$ (6) Å
 $c = 9.9140$ (4) Å
 $\beta = 109.837$ (1)°
 $V = 1880.31$ (13) Å³
 $Z = 4$

$D_x = 1.344$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 6618 reflections
 $\theta = 2.1$ – 33.0°
 $\mu = 0.11$ mm⁻¹
 $T = 183$ (2) K
Plate, colorless
 $0.24 \times 0.12 \times 0.05$ mm

Data collection

Siemens SMART CCD diffractometer
 ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
 $T_{min} = 0.975$, $T_{max} = 0.995$
30 831 measured reflections

6775 independent reflections
4740 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.047$
 $\theta_{max} = 33.0^\circ$
 $h = -19 \rightarrow 19$
 $k = -23 \rightarrow 23$
 $l = -14 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.057$
 $wR(F^2) = 0.133$
 $S = 1.04$
 6775 reflections
 275 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.7831P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.34 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$

Table 1

 Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O2-H2\cdots O1$ [a]	0.84	2.29	2.7168 (14)	112
$O2-H2\cdots O5^i$ [b]	0.84	1.97	2.7388 (14)	152
$O4-H4\cdots O6$ [c]	0.84	2.51	2.8339 (13)	104
$O4-H4\cdots O7$ [d]	0.84	2.15	2.8863 (14)	146
$O5-H5\cdots O4^{ii}$ [e]	0.84	2.05	2.8580 (14)	161
$C6-H6\cdots O4$	0.95	2.37	2.7319 (16)	102
$C6-H6\cdots O5^{iii}$	0.95	2.47	3.3401 (16)	152
$C11-H11B\cdots O1^i$	0.99	2.58	3.2998 (16)	129
$C15-H15\cdots O3^{iv}$	0.95	2.60	3.308 (2)	132

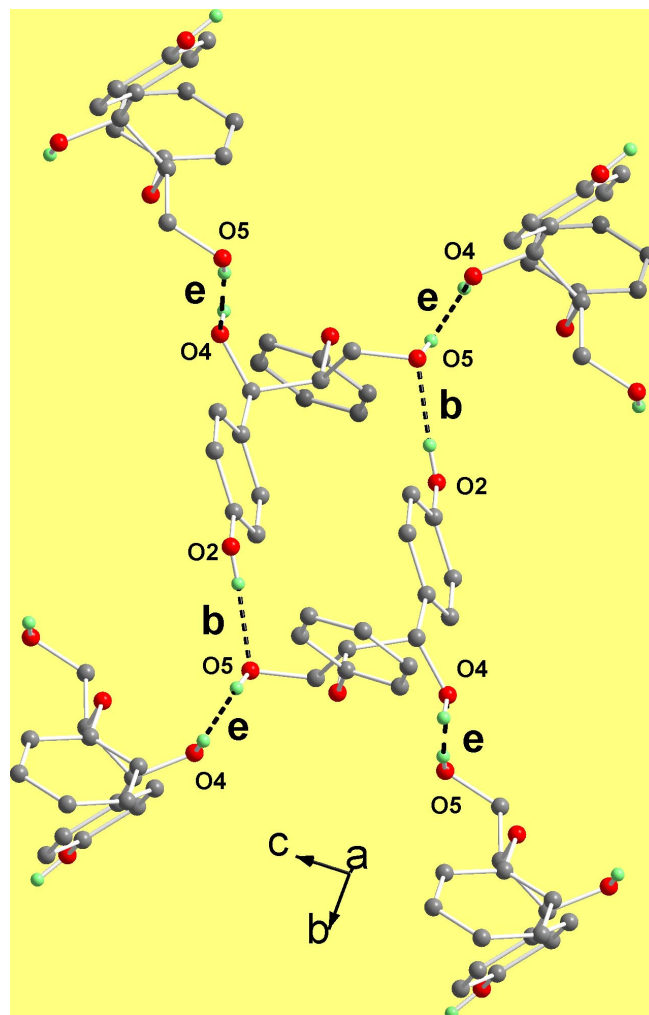
Symmetry codes: (i) $1-x, 1-y, -z$; (ii) $x, \frac{1}{2}-y, z-\frac{1}{2}$; (iii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (iv) $1+x, y, z$.

H atoms were refined isotropically and were constrained to the ideal geometry using an appropriate riding model. For hydroxyl groups, the O–H distances (0.84 \AA) and C–O–H angles (109.5°) were kept fixed, the torsion angle was chosen to maximize the electron density. For methyl groups, the C–H distances (0.98 \AA) and C–C–H angles (109.5°) were kept fixed, while the torsion angles were allowed to refine with the starting position based on the threefold averaged circular Fourier synthesis.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT* and *SADABS* (Sheldrick, 2001); program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 2000); software used to prepare material for publication: *SHELXTL*.

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Figure 3

The hydrogen-bond network showing the $R2,2(20)$ ring together with chains $C2,2(10)$ and $C2,2(16)$. Methoxy groups and carbon-bonded H atoms have been omitted for clarity. For hydrogen-bond codes, see Table 1.

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