Examination of the packing in the unit cell shows that the molecules pack as discrete monomeric units. An analysis of C—H···O interactions yielded none worthy of note. Similarly, no significant ring–ring interactions were observed with neighbouring molecules.

Experimental

The title compound was synthesized as described previously (Roy *et al.*, 1997) and single crystals suitable for diffraction measurements were obtained from methanol/water.

Crystal data

$C_{24}H_{18}ClNO_6S$
$M_r = 483.931$
Monoclinic
$P2_1/n$
$a = 10.4968 (9)$ Å
$b = 13.500 (1)$ Å
$c = 15.626 (1)$ Å
$\beta = 96.986 (6)°$
$V = 2197.9 (6)$ Å^3
$Z = 4$
$D_x = 1.462$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 24 reflections
$\theta = 37.09–39.89°$
$\mu = 2.79$ mm^{-1}
$T = 294$ K
Small rhomb
0.12 × 0.12 × 0.12 mm
Colourless

Data collection

Rigaku AFC-5 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4700 measured reflections
4253 independent reflections
3033 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.032$
$\theta_{\rm max} = 72.65°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 16$
$l = -19 \rightarrow 19$
3 standard reflections every 400 reflections intensity decay: −1.0 (4)%

Refinement

Refinement on F^2
$R(F) = 0.049$
$wR(F^2) = 0.128$
$S = 1.026$
4253 reflections
300 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0762P)^2 + 0.8774P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = -0.001$
$\Delta\rho_{\rm max} = 0.51 (7)$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.32 (7)$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected bond lengths (Å)*

Cl1—C2	1.741 (3)	N8—C9	1.406 (3)
O11—C12	1.353 (4)	N8—C6	1.427 (3)
O11—C10	1.400 (3)	C9—C10	1.337 (4)
O33—C12	1.209 (4)	C9—C19	1.474 (4)
N8—C12	1.369 (4)	C10—C13	1.463 (4)

H atoms were included at calculated positions using a riding model (with this model each methyl group contributes an additional parameter to those varied).

Data collection: AFC-5 software. Cell refinement: AFC-5 software. Data reduction: local software. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: local software.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1376). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 1494–1496

Methyl 2,6-Dihydroxy-4-(2-hydroxy-4,6-dimethoxy-3-methylbenzoyloxy)-3-methylbenzoate

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(*Received 4 March 1998; accepted 3 April 1998*)

Abstract

In the title compound, $C_{19}H_{20}O_9$, the benzene rings are nearly perpendicular to each other [dihedral angle 86.39 (8)°]. All three hydroxy groups are involved in intramolecular O—H···O hydrogen bonds.

Comment

The biosynthetic interrelationship between the common depsides, the depsidones, dibenzofurans and diphenyl ethers has been a subject of speculation for some time (Culberson, 1969; Mosbach, 1973). It has been reported

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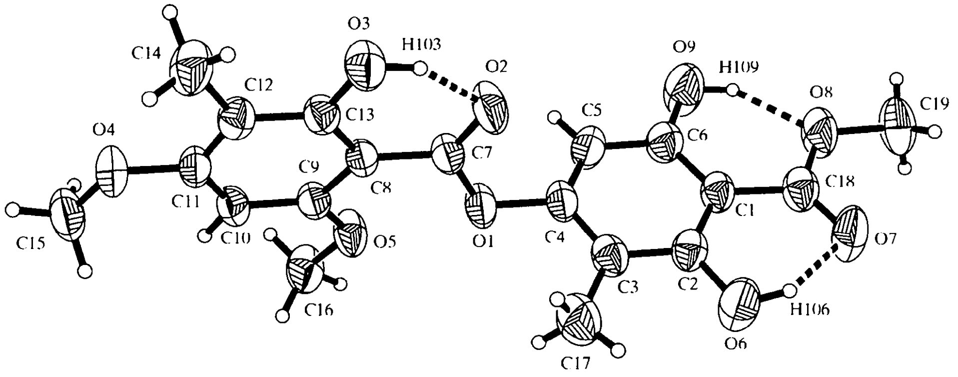
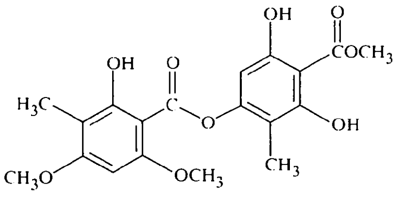


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

that *para*-depsides could be readily converted into the corresponding diphenyl ether *via* an intramolecular Smiles rearrangement (Elix *et al.*, 1984). The present work is part of a project concerned with the synthesis of diphenyl ethers and their analogues. The title compound, (I), a depside, is an intermediate compound in the synthesis of a diphenyl ether molecule. Knowledge of the three-dimensional structure of the title molecule could be useful for the understanding of this synthesis and for predicting the conformation of other depside molecules.



(I)

The bond lengths and angles observed in the structure of (I) agree with values reported for similar compounds (Allen *et al.*, 1987). The two benzene rings are nearly perpendicular to one another [dihedral angle 86.39 (8)°]. The methoxycarbonyl group (C18, C19, O7, O8) is twisted from the plane of the adjacent benzene ring by 2.98 (9)°. Both methoxy groups are twisted slightly out of the plane of benzene ring C8–C13 [torsion angles C10—C11—O4—C15 −3.3 (3) and C10—C9—O5—C16 3.2 (3)°]. All three hydroxy groups in the molecule are involved in intramolecular O—H···O hydrogen bonds with carbonyl or methoxy O atoms. The crystal structure is stabilized by weak intermolecular C—H···O hydrogen bonds and van der Waals interactions.

Experimental

The synthesis of the title compound was carried out by reaction of 2-hydroxy-4,6-dimethoxy-3-methylbenzoic acid and methyl 2,4,6-trihydroxy-3-methylbenzoate (molar ratio 1:1) in dry dichloromethane. 4-Dimethylaminopyridine and *N*,*N*-dicyclohexylcarbodiimide were added and the mixture was stirred in a nitrogen atmosphere at room temperature for 6 h. After purification, the product was recrystallized from a mixture of petroleum ether and dichloromethane, giving the title compound as colourless crystals (m.p. 450–451 K). The structure of the title compound was confirmed by microanalytical evidence as well as spectroscopic data.

Crystal data

$C_{19}H_{20}O_9$
$M_r = 392.35$
Triclinic
$P\bar{1}$
$a = 8.4255\ (2)$ Å
$b = 8.5771\ (2)$ Å
$c = 14.5531\ (2)$ Å
$\alpha = 89.692\ (1)°$
$\beta = 85.374\ (2)°$
$\gamma = 61.295\ (1)°$
$V = 918.78\ (3)$ Å^3
$Z = 2$
$D_x = 1.418$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 3274 reflections
$\theta = 1.5$–$25.0°$
$\mu = 0.114$ mm^{-1}
$T = 293\ (2)$ K
Thin plate
0.70 × 0.36 × 0.04 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
4938 measured reflections
3159 independent reflections

2396 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\max} = 25°$
$h = -10 \rightarrow 12$
$k = -12 \rightarrow 13$
$l = -16 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.149$
$S = 1.054$
3159 reflections
333 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0865P)^2 + 0.1275P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.196$ e Å^{-3}
$\Delta\rho_{\min} = -0.192$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C7	1.353 (2)	O5—C16	1.433 (3)
O1—C4	1.414 (2)	O6—C2	1.352 (3)
O2—C7	1.219 (2)	O7—C18	1.212 (3)
O3—C13	1.355 (2)	O8—C18	1.343 (3)
O4—C11	1.369 (2)	O8—C19	1.456 (3)
O4—C15	1.431 (3)	O9—C6	1.362 (3)
O5—C9	1.360 (2)		
C7—O1—C4—C5	−82.2 (2)	O2—C7—C8—C13	−11.1 (3)
C4—O1—C7—C8	179.5 (2)	C19—O8—C18—O7	2.6 (4)
O1—C7—C8—C9	−12.8 (3)	C6—C1—C18—O8	2.7 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1O3···O2	0.94 (3)	1.69 (3)	2.554 (2)	151 (3)
O6—H1O6···O7	0.94 (4)	1.71 (4)	2.579 (3)	153 (4)
O9—H1O9···O8	0.88 (5)	1.76 (4)	2.570 (3)	152 (4)
C5—H5···O4^{i}	0.91 (3)	2.57 (3)	3.409 (3)	154 (3)

Symmetry code: (i) $1 - x, -y, 1 - z$.

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4.023 cm and the detector swing angle was −35°. Coverage of the unique set is over 91% complete to at least 25° in θ. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible. The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from a difference Fourier map and refined isotropically.

Data collection: *SMART* (Siemens, 1996*b*). Cell refinement: *SAINT* (Siemens, 1996*a*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

This work has received partial support from the King Prajadhipok and Queen Rambhaibarni Memorial Foundation. The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. KC thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1218). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 1496–1499

2-Crotonoyl-5,8-dihydro-1-naphthol, (I), and 4-(1-Naphthoyl)-5,8-dihydro-1-naphthol, (II)†

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(*Received 2 February 1998; accepted 16 April 1998*)

Abstract

In (I), $C_{14}H_{14}O_2$, the dihydrobenzene ring adopts a flattened boat conformation and the hydroxyl group is involved in an O—H···O intramolecular hydrogen bond. In (II), $C_{21}H_{16}O_2$, the dihydronaphthalene ring system is planar and makes a dihedral angle of 70.06 (7)° with the naphthalene ring system; the hydroxyl group forms O—H···O intermolecular hydrogen bonds.

Comment

The hydroxyketone derivatives of naphthalene are useful in the synthesis of the subunits of daunomycinone and adriamycin anticancer drugs (Crouse *et al.*, 1981). The dihydronaphthalene derivatives are useful intermediates in the synthesis of cyclic polymethylene phenols, which are useful antifibrillatory agents, disinfectants and water softeners (Hauck *et al.*, 1977). The crystal structure determinations of the title compounds, (I) and (II), were carried out in order to elucidate the molecular conformation and packing arrangements.

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† IUPAC names: 1-(1-hydroxy-5,8-hydro-2-naphthyl)but-2-en-1-one and 1-hydroxy-5,8-dihydro-4-naphthyl 1-naphthyl ketone.

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Acta Crystallographica Section C
ISSN 0108-2701 © 1998