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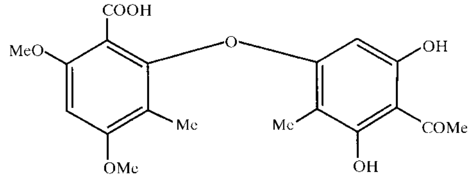
In the title compound, $C_{19}H_{20}O_8$, the benzene rings are nearly perpendicular to each other [dihedral angle 80.2 (2)°]. The carboxy group is twisted out while both the methoxy and acetyl groups are almost coplanar with their attached benzene rings. The hydroxy group is involved in an intramolecular O—H···O hydrogen bond with the acetyl O atom and the compound is connected through an intermolecular O—H···O contact to form a dimer. The crystal structure is stabilized by intermolecular O—H···O hydrogen bonds.

Comment

It has been reported that *p*-depsides could be readily converted into the corresponding diphenyl ether *via* an intramolecular Smiles rearrangement (Elix *et al.*, 1984). A number of depsides have been prepared as intermediate compounds in the synthesis of corresponding diphenyl ether molecules. Suitable reaction conditions for an intramolecular Smiles rearrangement of the prepared depsides have also been studied extensively (Elix & Jenie, 1989; Elix *et al.*, 1990). The crystal structure determination of the title compound, (I), was undertaken as part of structural studies on diphenyl ether derivatives. Knowledge of the three-dimensional structure of (I) could be useful for the understanding of this synthesis and predicting selectivity of (I).



(I)

It is very difficult to obtain good quality crystals for this compound. Preliminary tests also show that this compound processes antibacterial action against *S. aureus*, though not against *E. coli*.

The bond lengths and bond angles observed in the structure are normal and agree reasonably with the reported values (Elix *et al.*, 1978; Allen *et al.*, 1987; Chantrapromma *et al.*, 1998). The benzene rings are essentially planar [the atoms having the largest deviations in the two benzene rings are C6 −0.036 (5) Å and C9 0.017 (5) Å] and they are nearly perpendicular to each other [dihedral angle 80.2 (2)°]. The carboxy group is twisted out of the benzene plane [C7—C12—C19—O8 −38.5 (6)°] while the two methoxy groups and the acetyl group are nearly coplanar with the benzene rings [C18—O6—C11—C10 8.6 (6)°, C17—O5—C9—C10 −5.1 (7)° and C2—C3—C13—C14 3.9 (8)°]. The hydroxy group in the molecule is involved in an intramolecular O—H···O hydrogen bond with the acetyl O atom. The compound is connected by intermolecular O—H···O contact to form a dimer (O8—H8*A*···O7 in Table 2).

Experimental

The title compound was synthesized according to the procedure of Elix *et al.* (1984). A solution of 4,6-dimethoxy-2-hydroxy-3-methylbenzoic acid (0.07 mmol) and 2,4,6-trihydroxy-3-methylacetophenone (0.07 mmol) in dry dichloromethane was added to a mixture of *N*,*N*-dimethylaminopyridine (0.01 mmol) and *N*,*N*-dicyclohexylcarbodiimide(0.17 mmol). The reaction mixture was stirred in a nitrogen atmosphere at room temperature for 6 h. After purification, the corresponding depside (0.05 mmol) was obtained. For the Smiles rearrangement of the depside, a mixture of the depside (0.27 mmol) and anhydrous potassium carbonate (0.40 mmol) in dimethyl sulfoxide (5 ml) was stirred in a nitrogen atmosphere at room temperature for 16 h. The reaction mixture was acidified with cold diluted hydrochloric acid and extracted with ethyl acetate. The combined organic extracts were washed with water to remove the dimethyl sulfoxide. After evaporation of the solvent, the crude product was recrystallized from methanol, giving the title compound as light-brown crystals, m.p. 447–449 K.

Crystal data

$C_{19}H_{20}O_8$
$M_r = 376.35$
Monoclinic, $P2_1/n$
$a = 6.0228$ (3) Å
$b = 24.792$ (1) Å
$c = 12.189$ (1) Å
$\beta = 100.928$ (1)°
$V = 1787.1$ (2) Å^3
$Z = 4$

$D_x = 1.399$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 3937 reflections
$\theta = 1.64$–28.15°
$\mu = 0.110$ mm^{-1}
$T = 293$ (2) K
Plate, light brown
0.34 × 0.18 × 0.10 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
6669 measured reflections
2848 independent reflections
1529 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.075$
$\theta_{\rm max} = 25°$
$h = -7 \rightarrow 6$
$k = -29 \rightarrow 29$
$l = -11 \rightarrow 14$
Intensity decay: none

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Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.080$
$wR(F^2) = 0.231$
$S = 0.961$
2848 reflections
244 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1483P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.47$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.374 (5)	O5—C17	1.438 (5)
O1—C7	1.402 (5)	O6—C11	1.366 (5)
O2—C2	1.369 (6)	O6—C18	1.427 (5)
O3—C13	1.248 (5)	O7—C19	1.239 (5)
O4—C4	1.358 (5)	O8—C19	1.303 (5)
O5—C9	1.349 (5)		
C6—O1—C7	117.3 (3)	C11—O6—C18	117.9 (3)
C9—O5—C17	118.8 (4)		
C6—C1—C2—O2	174.4 (4)	C17—O5—C9—C10	−5.1 (7)
C1—C2—C3—C4	5.6 (7)	C17—O5—C9—C8	175.9 (4)
O2—C2—C3—C13	9.6 (7)	C18—O6—C11—C10	8.6 (6)
C2—C3—C4—O4	177.0 (4)	C9—C10—C11—O6	−179.1 (4)
C13—C3—C4—C5	176.3 (4)	C8—C7—C12—C19	176.7 (4)
O4—C4—C5—C6	177.2 (4)	C2—C3—C13—C14	3.8 (8)
C7—O1—C6—C1	−29.9 (6)	C7—C12—C19—O8	−38.5 (6)
C4—C5—C6—O1	−176.3 (4)		

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O2—H2A⋯O7$^{\rm i}$	0.82	2.10	2.914 (4)	178
O4—H4A⋯O3	0.82	1.77	2.506 (6)	148
O8—H8A⋯O7$^{\rm ii}$	0.82	1.89	2.697 (5)	170

Symmetry codes: (i) $2-x, 1-y, -z$; (ii) $3-x, 1-y, -z$.

After checking their presence in the difference map, all H atoms were placed in calculated positions with O—H = 0.82 Å and C—H = 0.93–0.96 Å, and were allowed to ride on their attached atoms with $U_{\rm iso} = 1.5U_{\rm eq}$ for the attached atoms. Due to large fraction of weak data at higher angles, the 2θ maximum is limited to 50°.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.

Chantrapromma, K., Sortiruk, R., Chantrapromma, S., Ponglimanont, C., Fun, H.-K. & Chinnakali, K. (1998). *Acta Cryst.* C**54**, 1494–1496.

Elix, J. A., Engkaninan, U., Jones, A. J., Raston, C. L., Sargen, M. V. & White, A. L. (1978). *Aust. J. Chem.* **31**, 2057–2068.

Elix, J. A. & Jenie, U. A. (1989). *Aust. J. Chem.* **42**, 987–994.

Elix, J. A., Jiang, H. & Wardlaw, J. H. (1990). *Aust. J. Chem.* **43**, 1745–1758.

Elix, J. A., Jones, A. J., Lajide, L., Coppins, B. J. & James, P. W. (1984). *Aust. J. Chem.* **37**, 2349–2364.

Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M.(1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.