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2′,4′,6′-Trihydroxy-1-methoxyacetophenone monohydrate at 150 K

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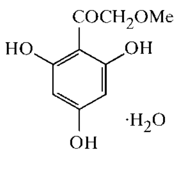
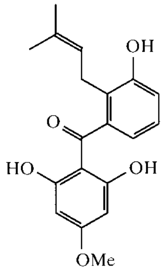
The geometry of the title compound, 2-methoxy-1-(2,4,6-trihydroxyphenyl)ethanone, $C_9H_{10}O_5 \cdot H_2O$, is determined by the presence of an intramolecular hydrogen bond; the geometry of the benzene ring is distorted by a flanking carbonyl group.

Comment

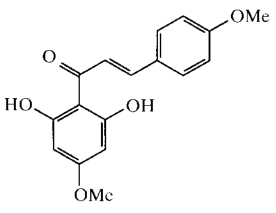
The molecular geometry in the title compound, (I), was determined in order to study the geometric distortions in a tetrasubstituted benzene derivative and to investigate the possibilities for inter- and intramolecular hydrogen bonding in the solid state. Only a few closely analogous structures have been reported. For example, cudranone, (II), and the chalcone (III) are both ketones which contain the 2,6-dihydroxybenzene substructure, the former being a benzophenone derivative and the latter having a saturated side chain (Ottersen *et al.*, 1977; Ulrich *et al.*, 1990). Both have methoxy groups at the 4-position of the hydroxylated ring, whereas (I) is a 2,4,6-trihydroxybenzene derivative.



(I)



(II)



(III)

The intramolecular geometry of (I) is dominated by a cyclic hydrogen bond involving the O6—H6 group and the O11 atom of the carbonyl group [O6···O11 2.4860 (14) Å and H6···O11 1.66 (2) Å]. The molecular structure, labelling scheme and partial hydrogen bonding is depicted in Fig. 1. The O···O distances in (II) and (III) are 2.501 (3) and 2.406 (3) Å, respectively. Intramolecular hydrogen bonding has little effect on the length of the C—O bond of the participating phenol group of (I), *e.g.* C6—O6 is 1.3527 (16) Å and C4—O4 is 1.3537 (16) Å. The bond lengths of the benzene ring of (I) are distorted: those flanking the carbonyl group, C1—C2 and C1—C6, are 1.4190 (18) and 1.4265 (18) Å, respectively, whereas the others lie between 1.380 and 1.390 Å. The longest ring bond is that which is involved in the cyclic hydrogen bond. Similar features are present in the structures of (II) and (III).

The internal bond angles in the benzene ring of (I) range from 116.34 (11) to 122.20 (12)°, the former being at the point of substitution of the acyl group, and the latter at that of the intramolecular hydrogen-bonding hydroxy group. The bond angles generally agree well with those calculated using the angular substituent parameters described by Domenicano (1992). The largest deviation (1.3°) is found at C1. Once again similar features are apparent in (II) and (III). The geometry about the C═O group is very similar in (I) and (II). In (I), the aryl–carbon distance, C1—C11 [1.4499 (17) Å], is substantially shorter than that of the alkyl–carbon distance, C11—C12 [1.5096 (18) Å], as expected. In (II), the similarity of the magnitude of these two features is more surprising; however, the longer bond length is that to the monohydroxylated ring, which is twisted substantially out of plane and is therefore unable to participate in conjugation.

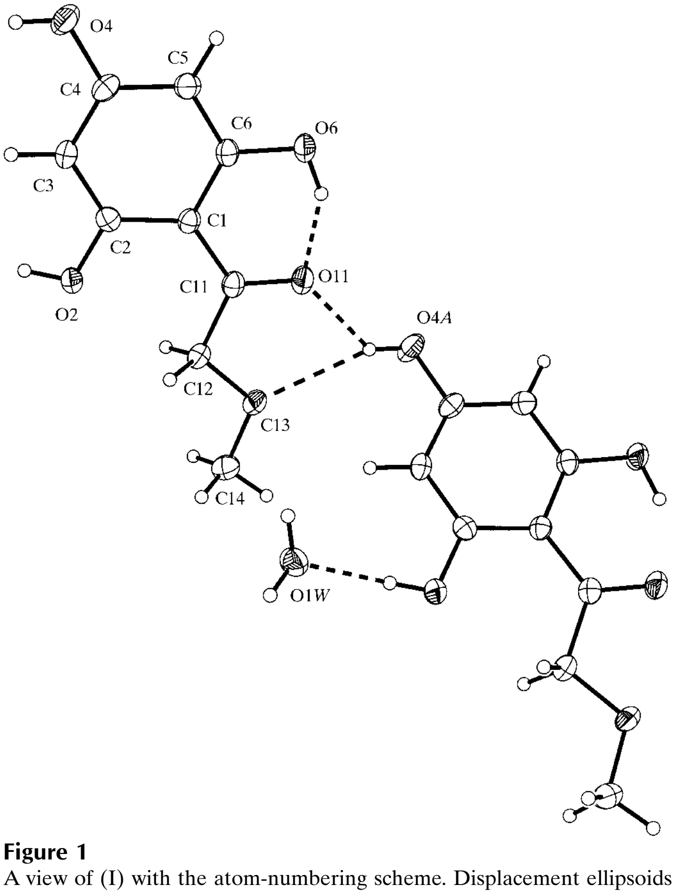


Figure 1
A view of (I) with the atom-numbering scheme. Displacement ellipsoids enclose 50% probability surfaces. The symmetry element (*A*) relating the two molecules is (x, $-y$, $z + \frac{1}{2}$).

The entire molecule of (I), including the methoxy group, is planar (mean deviation 0.006 Å from the plane of the ring; maximum deviation 0.201 Å for C14). This is ascribable to the formation of a bifurcated intermolecular hydrogen bond from H4 of a neighbouring molecule to O11 and O13 (Fig. 1). The interaction shown in Fig. 1 extends to form corrugated sheets of molecules which are hydrogen bonded together by molecules of water of crystallization (see Table 2).

Experimental

Compound (I) was prepared by the Houben–Hoesch reaction of phloroglucinol with methoxyacetonitrile in the presence of anhydrous HCl and $ZnCl_2$ (Deng *et al.*, 1997). Crystals were obtained by recrystallization from water.

Crystal data

$C_9H_{10}O_5{\cdot}H_2O$
$M_r = 216.19$
Monoclinic, $C2/c$
$a = 17.3138\ (5)$ Å
$b = 7.33300\ (10)$ Å
$c = 16.8507\ (5)$ Å
$\beta = 117.8380\ (10)^\circ$
$V = 1891.81\ (8)$ Å^3
$Z = 8$
$D_x = 1.518$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24172 reflections
$\theta = 2.66$–27.56°
$\mu = 0.129$ mm^{-1}
$T = 150\ (2)$ K
Block, colourless
0.35 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
ω–φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
$T_{\min} = 0.811$, $T_{\max} = 1.000$
24172 measured reflections
2180 independent reflections
1594 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.076$
$\theta_{\max} = 27.56^\circ$
$h = -22 \rightarrow 22$
$k = -9 \rightarrow 9$
$l = -21 \rightarrow 21$

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.4190 (18)	C2—O2	1.3495 (15)
C1—C6	1.4265 (18)	C2—C3	1.3831 (18)
C1—C11	1.4499 (17)	C3—C4	1.3872 (19)
C11—O11	1.2466 (15)	C4—O4	1.3537 (16)
C11—C12	1.5096 (18)	C4—C5	1.3849 (19)
C12—O13	1.4084 (16)	C5—C6	1.3808 (18)
O13—C14	1.4246 (17)	C6—O6	1.3527 (16)
C2—C1—C6	116.34 (11)	C3—C2—C1	121.45 (12)
C2—C1—C11	124.61 (11)	C2—C3—C4	119.69 (12)
C6—C1—C11	119.06 (11)	O4—C4—C5	117.42 (12)
O11—C11—C1	120.54 (12)	O4—C4—C3	121.18 (12)
O11—C11—C12	116.80 (11)	C5—C4—C3	121.39 (12)
C1—C11—C12	122.66 (11)	C6—C5—C4	118.91 (12)
O13—C12—C11	108.82 (10)	O6—C6—C5	117.23 (12)
C12—O13—C14	111.25 (10)	O6—C6—C1	120.57 (11)
O2—C2—C3	120.47 (12)	C5—C6—C1	122.20 (12)
O2—C2—C1	118.08 (11)		

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O2—H2⋯O1$W^{\rm i}$	0.85 (2)	1.85 (2)	2.7017 (16)	171 (2)
O4—H4⋯O11$^{\rm ii}$	0.86 (3)	1.98 (2)	2.6727 (15)	137 (2)
O4—H4⋯O13$^{\rm ii}$	0.86 (3)	2.32 (3)	3.0881 (17)	149 (2)
O6—H6⋯O11	0.90 (2)	1.66 (2)	2.4860 (14)	150 (2)
O1W—H1W⋯O6$^{\rm iii}$	0.86 (2)	2.00 (2)	2.8514 (16)	172 (2)
O1W—H2W⋯O4$^{\rm iv}$	0.92 (2)	2.02 (2)	2.9345 (14)	175 (2)

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

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.109$
$S = 1.050$
2180 reflections
185 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2 + 0.0798P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0031 (7)

The refined C—H distances are in the range 0.94 (2)–1.03 (2) Å.

Data collection and refinement: *COLLECT* (Hooft, 1998); data reduction: *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BJ1012). Services for accessing these data are described at the back of the journal.

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