Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 2,5-dihydroxybenzoate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.140
Data-to-parameter ratio = 16.2

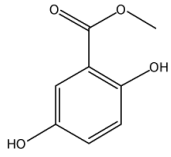
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 31 March 2003
Accepted 1 April 2003
Online 16 April 2003

The crystal structure of the title compound, $C_8H_8O_4$, is characterized by extensive hydrogen-bonding interactions to yield centrosymmetrically related dimers linked to each other by intermolecular interactions between the hydroxy groups and between the hydroxy and methyl groups.

Comment

The title compound, (I), was prepared as a prototype receptor with multiple functionality for coordination to analytes. The structure of (I) shows the molecules to be planar (Fig. 1), with bond lengths and angles in accord with conventional values (Allen *et al.*, 1987). Pairs of centrosymmetrically related molecules are associated through bifurcated intra- and intermolecular hydrogen-bonding interactions between the α-hydroxy group and carbonyl O atoms to form a carboxylic acid dimer motif. Further hydrogen-bonding is observed between the 2- and 5-hydroxy groups on adjacent molecules (Fig. 2). In addition, the structure is stabilized through weak C—H···O hydrogen-bonding interactions between the ester methyl group and 5-hydroxy groups on adjacent molecules.



(I)

Experimental

2,5-Dihydroxybenzoic acid (24.6 g, 159.5 mmol) was dissolved in methanol (500 ml). Sulfuric acid (98%, 10 ml) was added dropwise and the mixture stirred under reflux for 72 h. Removal of the solvent under reduced pressure yielded a yellow oil that was partitioned between chloroform (200 ml) and deionized water (100 ml). The organic layer was dried (magnesium sulfate) and condensed to yield the title compound as a crystalline white solid. Yield 24.5 g, 80.0%; m.p. 357–359 K (literature: 357–359 K; Malcolm, 1981). Crystals suitable for X-ray diffraction studies were prepared by slow evaporation at room temperature of a solution of 30 mg of (I) in a solution of diethyl ether (2 ml) and hexane (5 ml).

Crystal data

$C_8H_8O_4$
M_r = 168.14
Monoclinic, $P2_1/n$
a = 12.316 (2) Å
b = 12.986 (2) Å
c = 4.8795 (6) Å
β = 96.406 (12)°
V = 775.5 (2) Å^3
Z = 4
D_x = 1.440 Mg m^{-3}
Mo Kα radiation
Cell parameters from 25 reflections
θ = 12.6–17.2°
μ = 0.12 mm^{-1}
T = 295 K
Prism, colorless
0.40 × 0.30 × 0.30 mm

 DOI: 10.1107/S1600536803007347

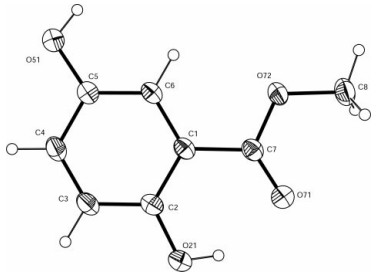


Figure 1
ORTEP-3 (Farrugia, 1997) plot, showing the atomic numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level for non-H atoms.

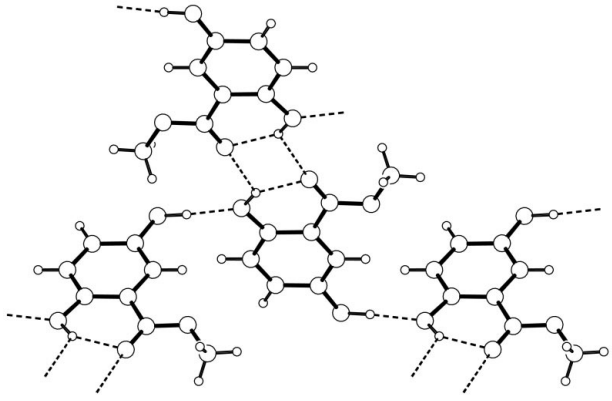


Figure 2
The hydrogen-bonding scheme for (I).

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
Absorption correction: none
2012 measured reflections
1784 independent reflections
1353 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 27.5°$
$h = -7 \rightarrow 15$
$k = 0 \rightarrow 16$
$l = -6 \rightarrow 6$
3 standard reflections
every 150 reflections
intensity decay: 0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.140$
$S = 1.03$
1784 reflections
110 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.092P)^2 + 0.1P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.033 (9)

Table 1
Selected geometric parameters (Å, °).

O21—C2	1.361 (2)	O72—C7	1.3306 (18)
O51—C5	1.363 (2)	O72—C8	1.444 (2)
O71—C7	1.2133 (18)		
C7—O72—C8	115.78 (12)	O51—C5—C6	123.55 (13)
O21—C2—C3	117.31 (13)	O72—C7—C1	113.07 (12)
O21—C2—C1	123.50 (13)	O71—C7—O72	123.09 (13)
O51—C5—C4	117.16 (13)	O71—C7—C1	123.84 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
O21—H21···O71	0.85	1.89	2.6205 (17)	142
O21—H21···O71^{i}	0.85	2.43	3.0012 (18)	125
O51—H51···O21^{ii}	0.85	1.92	2.7622 (17)	169
C8—H8*B*···O51^{iii}	0.95	2.55	3.283 (2)	134

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$; (iii) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$.

H atoms were placed at calculated positions with C—H set to 0.95 Å. Hydroxy H atoms were located from a difference Fourier map and the O—H bond length set to 0.85 Å. U_{iso} values for the H atoms were set at $1.2U_{eq}$ of the parent atom.

Data collection: *MSC/AFC*-7 *Diffractometer Control Software for Windows* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC*-7 *Diffractometer Control Software for Windows*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997–2001); program(s) used to solve structure: *TEXSAN for Windows*; program(s) used to refine structure: *TEXSAN for Windows* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *TEXSAN for Windows* and *PLATON* (Spek, 1980–2001).

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