Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,4-Methylenedioxybenzoic acid

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Received 14 February 2006
Accepted 23 March 2006.

Key indicators

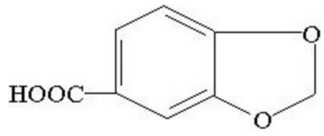
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.134
Data-to-parameter ratio = 14.8

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

The crystal structure of the title compound, $C_8H_6O_4$, consists of layers of characteristic dimers formed by intermolecular O—H···O hydrogen bonds.

Comment

3,4-Methylenedioxybenzoic acid (I), an important intermediate in the synthesis of some types of inhibitor (Shen, 2001) and antibiotics such as Cinoxacin, is also used in the production of some spices and dyes.



(I)

An intermolecular hydrogen bond, involving O3—H3*B* as donor and O4 as acceptor, links the molecules into dimers (Table 1 and Fig. 2).

Experimental

The most common method for the synthesis of 3,4-methylenedioxybenzoic acid was followed, in which 3,4-(methylenedioxy)benz-

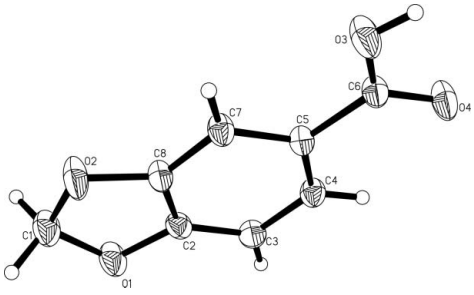


Figure 1
A view of the molecule of (I). Displacement ellipsoids are drawn at the 30% probability level.

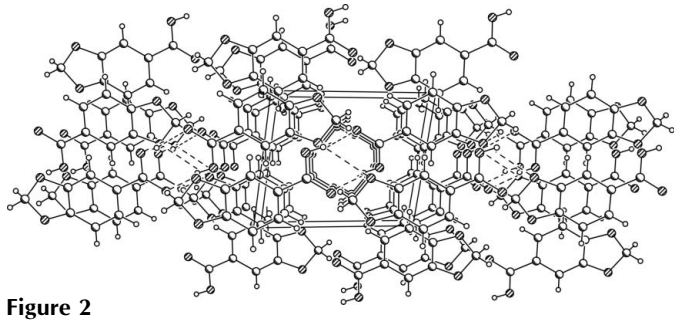


Figure 2
The packing of (I). Hydrogen bonds are shown as dashed lines.

aldehyde is prepared by oxidation of isoxafrole (200 ml) in a pressuse vessel with an ozone pressure of 1.0 MPa. 3,4-Methylenedioxybenzoic acid was obtained by further oxidation with potassium permanganate (Shriner & Kleiderder, 1930).

Crystal data

$C_8H_6O_4$
M_r = 166.13
Triclinic, $P\overline{1}$
a = 7.0685 (14) Å
b = 7.1553 (14) Å
c = 8.1436 (16) Å
α = 90.39 (3)°
β = 107.51 (3)°
γ = 114.06 (3)°
V = 354.67 (12) Å^3
Z = 2
D_x = 1.555 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2904 reflections
θ = 3.2–27.5°
μ = 0.13 mm^{-1}
T = 298 (2) K
Column, colorless
0.37 × 0.26 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
ω and φ scans
Absorption correction: multi-scan *SADABS*
T_{min} = 0.954, T_{max} = 0.981
3553 measured reflections
1613 independent reflections
1158 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.5°
$h = -9 \rightarrow 9$
$k = -8 \rightarrow 9$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.134
S = 1.03
1613 reflections
109 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.085P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3B···O4^{i}	0.82	1.81	2.618 (2)	167

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

H atoms were positioned geometrically (O—H = 0.82, C—H = 0.93–0.97 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C,O).

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/NT* (Bruker, 1997); software used to prepare material for publication: *SHELXTL/NT*.

We gratefully acknowledge support from the SRCICT of Tianjin University.

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