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2-(Carboxymethyl)-4,6-dimethoxybenzoic acid

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Key indicators

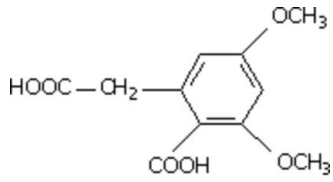
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.125
Data-to-parameter ratio = 14.7

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

The title compound, $C_{11}H_{12}O_6$, is a key intermediate for the synthesis of naturally occurring biologically active isocoumarin derivatives. In the crystal packing, molecules are linked into a two-dimensional network structure *via* intermolecular C—H···O hydrogen bonds.

Comment

The title compound, (I), alternatively named 4,6-dimethoxyhomophthalic acid, is a key intermediate in the synthesis of naturally occurring biologically active isocoumarin and dihydroisocoumarin derivatives.



(I)

The molecular structure of (I) is shown in Fig. 1. The C9/O3/O4 carboxyl group is tilted by 30.34 (9)° with respect to the plane of the benzene ring, possibly as a result of the steric hindrance of the *ortho* substituents. The O3—C9—C2—C3 and O3—C9—C2—C1 torsion angles are 149.68 (14) and −29.6 (2)°, respectively.

In the crystal packing, intermolecular C—H···O hydrogen-bond interactions (Table 1) link the molecules, forming a two-dimensional network (Fig. 2).

Experimental

The title compound was synthesized from 3,5-dimethoxybenzaldehyde according to a literature procedure (Furniss *et al.*,

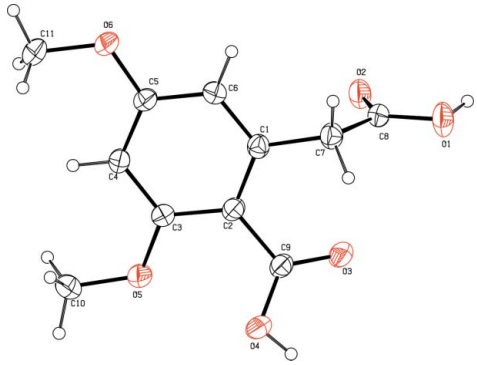


Figure 1
View of the title compound, with displacement ellipsoids drawn at the 30% probability level.

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1989). Light-yellow single crystals suitable for X-ray analysis were obtained by recystallization from an ethyl acetate solution.

Crystal data

$C_{11}H_{12}O_6$
M_r = 240.21
Triclinic, $P\overline{1}$
a = 7.2886 (10) Å
b = 7.9631 (11) Å
c = 10.2758 (14) Å
α = 105.937 (2)°
β = 103.444 (2)°
γ = 93.211 (2)°
V = 553.17 (13) Å^3
Z = 2
D_x = 1.442 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2688 reflections
θ = 2.7–28.2°
μ = 0.12 mm^{-1}
T = 292 (2) K
Block, light yellow
0.24 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.966, T_{max} = 0.977
4782 measured reflections
2380 independent reflections
2074 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 27.0°
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -12 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.126
S = 1.08
2380 reflections
162 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0658P)^2 + 0.0652P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7B···O3	0.97	2.43	2.7829 (19)	101
C10—H10C···O2^{i}	0.96	2.56	3.4144 (19)	148
O4—H4A···O3^{ii}	0.850 (10)	1.821 (11)	2.6606 (14)	169 (2)
O1—H1···O2^{iii}	0.853 (10)	1.801 (10)	2.6532 (14)	178 (2)

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

The H atoms of the carboxylic groups were located in a difference Fourier map and refined isotropically. All other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.96 Å, and U_{iso}(H) = 1.2 (aromatic and methylene H atom) or 1.5 (methyl H atoms) times U_{eq}(C).

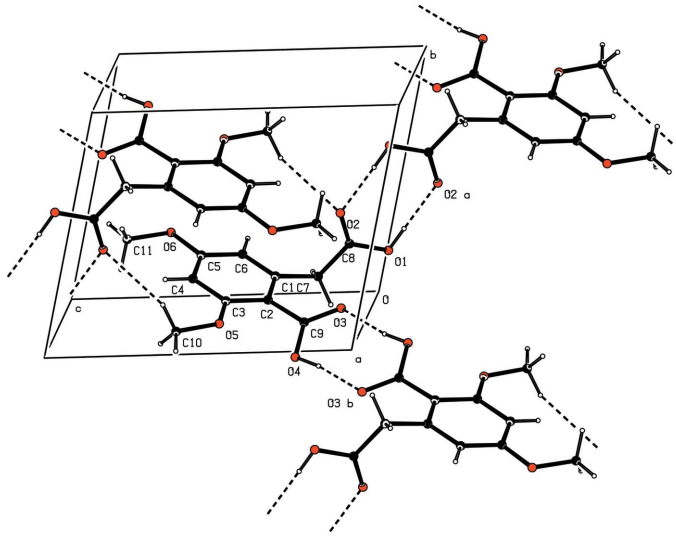

Figure 2
The crystal packing of the title compound, viewed approximately along the *a* axis. Hydrogen bonds are shown as dashed lines [symmetry codes: (*a*) $1-x, 1-y, 2-z$; (*b*) $-x, 2-y, 2-z$; (*c*) $-x, 1-y, 1-z$].

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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