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2,4,6-Trimethoxybenzoic acid

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.150
Data-to-parameter ratio = 22.0

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

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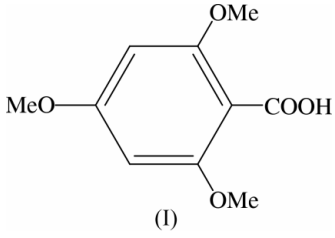
In the crystal structure of 2,4,6-trimethoxybenzoic acid, $C_{10}H_{12}O_5$, the molecules form hydrogen-bonded chains. The carboxyl group is in a *syn* conformation. The lone pair of electrons acting as the hydrogen bond acceptor is in an *anti* orientation.

Comment

ortho-Alkoxybenzoic acids are a class of acids which crystallize with different packing modes. The distinctive behaviour of 2-ethoxybenzoic acid which forms monomers is due to the formation of an intramolecular hydrogen bond (Gopalakrishna & Cartz, 1972). 2,3-Dimethoxybenzoic acid forms the normal acid dimer pattern (Bryan & White, 1982*a*). 2,6-Dimethoxybenzoic acid (Bryan & White, 1982*b*) and 2,6-dimethoxy-3-nitrobenzoic acid (Frankenbach *et al.*, 1991) form catemers. The carboxyl group of 2,6-dimethoxybenzoic acid exists in an *anti* conformation, the carboxyl group of 2,6-dimethoxy-3-nitrobenzoic acid in a *syn* conformation.



(I)

In 2,4,6-trimethoxybenzoic acid, (I), the three methoxy groups are nearly coplanar with the benzene ring (C5—C6—O61—C61 = 7.7°, C5—C4—O41—C41 = −7.0° and C3—C2—O21—C21 = 4.2°). As observed in 2,6-dimethoxybenzoic acid or 2,6-dimethoxy-3-nitrobenzoic acid, the hydrogen interaction from the hydroxyl O11 of one molecule to the remote carbonyl O12 of a neighbour (Table 2) results in catemers. The torsion angle between the plane of the acid group and the benzene ring (C6—C1—C11—O12) is 54.1 (1)°, quite similar to that found in 2,6-dimethoxybenzoic acid. However, in 2,4,6-trimethoxybenzoic acid, we find a *syn–anti* hydrogen-bond mode and in 2,6-dimethoxybenzoic acid an *anti–anti* hydrogen-bond mode. So the hypothesis (Frankenbach *et al.*, 1991) of the stabilization of the *anti–anti* mode by an intramolecular hydrogen bond has to be rejected. More subtle packing effects in the environment of the hydroxyl group have to be considered to give a rational explanation.

Experimental

2,4,6-Trimethoxybenzoic acid was purchased from Lancaster Chemicals. Crystals suitable for X-ray study were obtained by slow evaporation of a solution in ethanol.

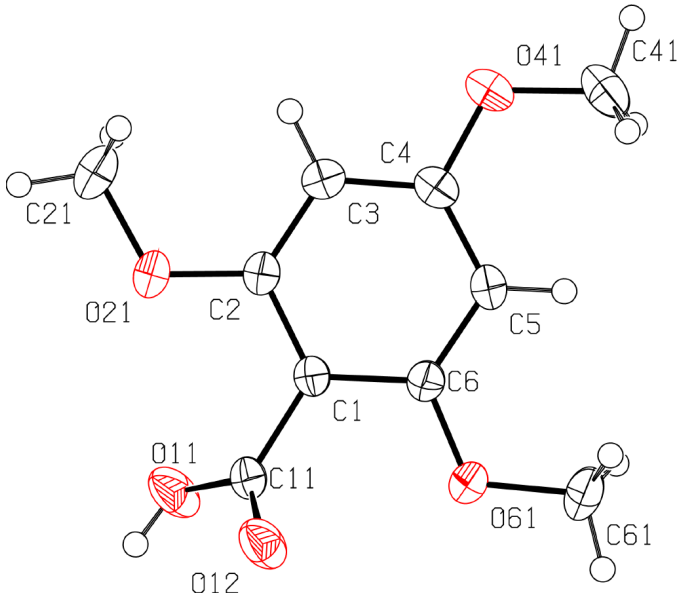


Figure 1
The molecular structure of (I) with the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level.

Crystal data

$C_{10}H_{12}O_5$
M_r = 212.20
Monoclinic, $P2_1/n$
a = 10.602 (3) Å
b = 7.288 (1) Å
c = 13.224 (8) Å
β = 93.80 (2)°
V = 1019.6 Å^3
Z = 4
D_x = 1.382 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 2.4–30.4°
μ = 0.11 mm^{-1}
T = 293 (2) K
Prismatic, white
0.67 × 0.35 × 0.22 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.907, T_{max} = 0.976
3231 measured reflections
3083 independent reflections
2256 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 30.4°
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 10$
$l = -18 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.150
S = 1.08
3083 reflections
140 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1033P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.025
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C6	1.4005 (13)	C5—C6	1.3876 (14)
C1—C2	1.4050 (13)	C6—O61	1.3538 (12)
C1—C11	1.4824 (14)	C11—O12	1.2089 (13)
C2—O21	1.3551 (13)	C11—O11	1.3207 (12)
C2—C3	1.3848 (15)	C21—O21	1.4262 (15)
C3—C4	1.3921 (16)	C41—O41	1.4234 (16)
C4—O41	1.3636 (13)	C61—O61	1.4261 (14)
C4—C5	1.3826 (15)		
C6—C1—C2	118.54 (9)	C4—C5—C6	118.50 (9)
C6—C1—C11	119.63 (8)	O61—C6—C5	123.39 (9)
C2—C1—C11	121.70 (9)	O61—C6—C1	115.17 (9)
O21—C2—C3	123.71 (9)	C5—C6—C1	121.41 (9)
O21—C2—C1	115.64 (9)	O12—C11—O11	122.14 (9)
C3—C2—C1	120.61 (9)	O12—C11—C1	123.85 (9)
C2—C3—C4	119.09 (10)	O11—C11—C1	114.01 (9)
O41—C4—C5	123.62 (10)	C2—O21—C21	118.05 (10)
O41—C4—C3	114.52 (10)	C4—O41—C41	117.94 (10)
C5—C4—C3	121.85 (10)	C6—O61—C61	117.96 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
O11—H11···O12^{i}	0.82	1.88	2.6683 (12)	160

Symmetry code: (i) $\frac{3}{2} - x, y - \frac{1}{2}, -\frac{1}{2} - z$.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLUTON*93 (Spek, 1993); software used to prepare material for publication: *SHELXL*97.

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