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2-(2,4-Dihydroxybenzoyl)benzoic acid

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In the title structure, $C_{14}H_{10}O_5$, the angle between the planes formed by the 2,4-dihydroxybenzoyl and *o*-benzoic acid moieties is 87.12 (4)°. In addition to an intramolecular O—H···O hydrogen bond, intermolecular O—H···O hydrogen bonds (H···O = 1.76 and 1.89 Å) connect molecules to form a two-dimensional network parallel to $(10\bar{1})$.

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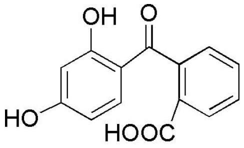
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.049
wR factor = 0.146
Data-to-parameter ratio = 14.3

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

Comment

The title compound, (I), is an intermediate in the synthesis of fluorescein and was first prepared by Baeyer (1876) and also by Bollmann (1922) in his study of resorcinbenzein. Further details about the title compound and its derivatives have been reported (Orndorff & Adamson, 1918; Orndorff & Kelley, 1922 Orndorff & Kline, 1924). Despite extensive investigations with repect to its synthesis, there has not been a crystallographic study of (I). The present study reports the crystal structure of 2-(2,4-dihydroxybenzoyl)benzoic acid at room temperature.



(I)

Selected bond lengths and angles for (I) are given in Table 1. The 2,4-dihydroxybenzoyl and *o*-benzoic acid moieties are each essentially planar, with maximum deviations from each plane of 0.0170 (21) Å for C5 and 0.0417 (17) Å for O5, and the angle between these planes is 86.79 (4)°. In addition to an intramolecular O—H···O hydrogen bond, intermolecular O—H···O hydrogen bonds connect molecules to form a two-dimensional network parallel to $(10\bar{1})$ (see Table 2 and Fig. 2).

Experimental

The title compound was prepared according to the method described by Orndorff & Kline (1924). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution in methanol and water. ^{1}H NMR (DMSO-d_6): δ 6.21 (*dd*, ^4J = 2.0 Hz, ^3J = 8.8 Hz, 1H), 6.32 (*d*, J = 2.4 Hz, 1H), 6.92 (*d*, J = 8.8 Hz, 1H), 7.36 (*dd*, ^4J = 0.8 Hz, ^3J = 7.6 Hz, 1H), 7.58–7.70 (*m*, 2H), 8.08 (*dd*, ^4J = 0.8 Hz, ^3J = 8.0 Hz, 1H), 10.68 (*s*, 1H), 12.22 (*s*, 1H), 13.15 (*s*, 1H); ^{13}C NMR (DMSO-d_6): δ 103.63, 109.03, 114.95, 128.63, 130.66, 131.48, 133.42, 136.06, 142.00, 166.59, 168.70, 202.82.

Crystal data

$C_{14}H_{10}O_5$
M_r = 258.22
Monoclinic, $P2_1/n$
a = 10.331 (3) Å
b = 11.628 (4) Å
c = 11.640 (4) Å
β = 116.034 (5)°
V = 1256.4 (7) Å^3
Z = 4

D_x = 1.365 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2633 reflections
θ = 2.6–26.5°
μ = 0.11 mm^{-1}
T = 294 (2) K
Block, colourless
0.30 × 0.22 × 0.22 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{\min}$ = 0.963, $T_{\max}$ = 0.977
6755 measured reflections

2626 independent reflections
1762 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.099
$\theta_{\max}$ = 26.7°
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 14$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.146
S = 1.07
2626 reflections
184 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.3516P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.27 e Å^{-3}
$\Delta\rho_{\min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O2—C1	1.316 (2)	C3—C8	1.513 (3)
O4—C10	1.353 (3)	C8—C9	1.436 (3)
C1—C2	1.488 (3)		
O1—C1—O2	122.6 (2)	O3—C8—C9	121.81 (17)
C4—C3—C8	116.49 (17)	C14—C9—C8	121.67 (17)
C2—C3—C8	124.45 (17)	C10—C9—C8	121.12 (18)
O1—C1—C2—C7	−179.5 (2)	O2—C1—C2—C3	178.9 (2)
O2—C1—C2—C7	0.0 (3)	O3—C8—C9—C14	178.77 (19)
O1—C1—C2—C3	−0.7 (3)	O3—C8—C9—C10	−0.5 (3)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O2—H2$\cdots$O3$^{\rm i}$	0.89 (3)	1.76 (3)	2.643 (2)	177 (3)
O4—H4$\cdots$O3	0.90 (3)	1.80 (3)	2.602 (2)	148 (3)
O5—H5$\cdots$O1$^{\rm ii}$	0.89 (4)	1.89 (4)	2.764 (2)	169 (3)

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

All H atoms bonded to C atoms were included in calculated positions, with C—H = 0.93 Å. They were included in the refinement in riding-model approximation, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The H atoms bonded to O atoms were refined independently with isotropic displacement parameters.

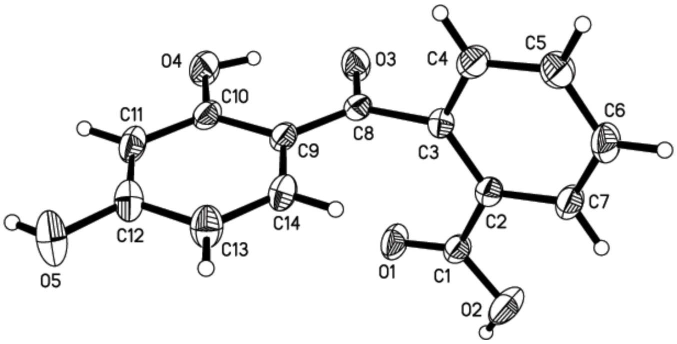


Figure 1
A view of the molecular of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

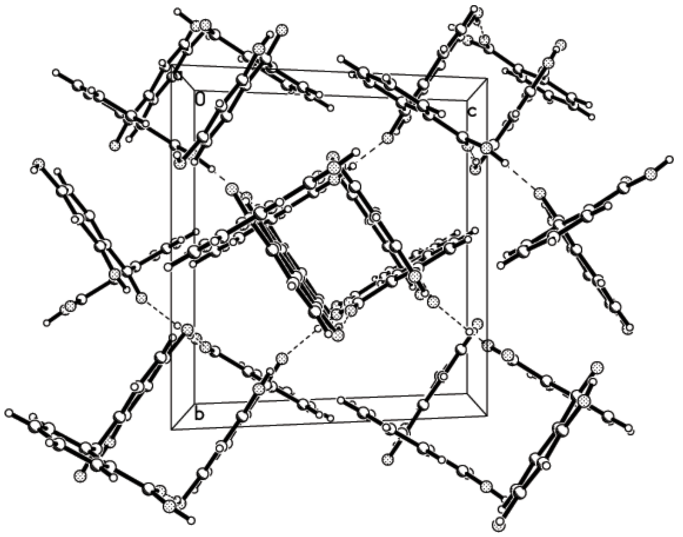


Figure 2
The molecular structure of (I), viewed along the a axis. Dashed lines indicate hydrogen-bond interactions.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*.

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