Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Benzene-1,2,4,5-tetracarboxylic acid dihydrate at 160 K

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Received 16 June 2003
Accepted 26 June 2003
Online 10 July 2003

Key indicators

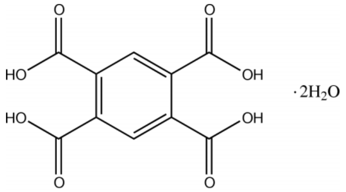
Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.096
Data-to-parameter ratio = 10.2

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

The title compound, $C_{10}H_6O_8\cdot 2H_2O$, has been previously studied by single-crystal X-ray diffraction at room temperature [Takusagawa *et al.* (1971) *Bull. Chem. Soc. Jpn*, **44**, 1274–1278]. We present here a redetermination of the structure at 160 K, resulting in a considerably improved precision. The organic molecule has crystallographic inversion symmetry.

Comment

The title compound, (I), has been previously studied at room temperature (Cambridge Structural Database, Version 5.24, April 2003 Update; Allen, 2002; reference code PYMELL10; Takusagawa *et al.*, 1971) using single-crystal X-ray diffraction.



(I)

The use of low-temperature (160 K) data collection here results in an improvement in the precision of the structure $\{R1[F^2 > 2\sigma(F^2)] = 0.035$ at 160 K, compared to $R1 = 0.074$ at room temperature (Takusagawa *et al.*, 1971)}.

This improved structure (Fig. 1) shows considerable similarities to the original, with almost identical molecular geometry (Table 1) and only slightly differing dihedral angles between the two independent carboxyl groups and the plane of the aromatic ring [dihedral angles with respect to the C1–C3

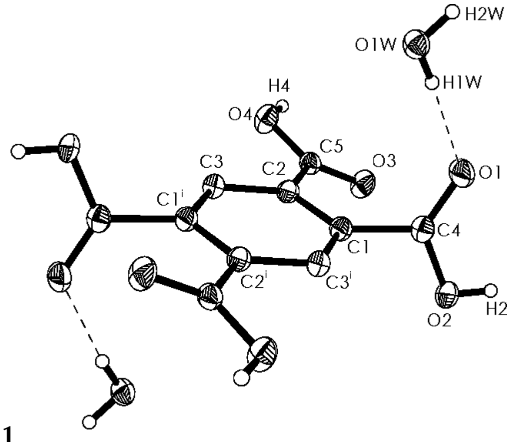


Figure 1
View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Aromatic H atoms have been removed for clarity and OH atoms are represented by circles of arbitrary radius. Hydrogen bonds are represented by dashed lines. [Symmetry code: (i) $1 - x, 1 - y, 1 - z$.]

DOI: 10.1107/S1600536803014351

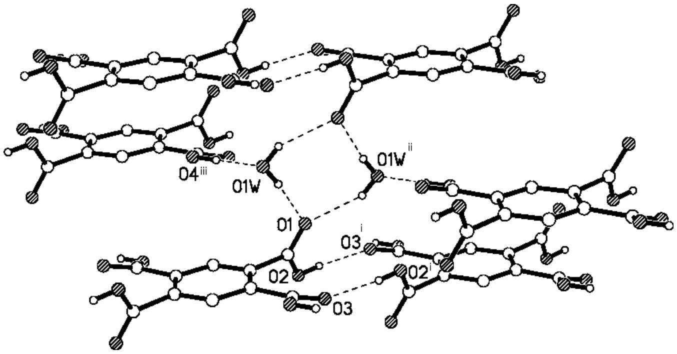


Figure 2
The three-dimensional hydrogen bonding array maintained by O—H···O hydrogen bonds. Aromatic H atoms have been removed for clarity, other atoms are represented by circles of arbitrary radius and hydrogen bonds are represented by dashed lines. [Symmetry codes: (i) $1 - x, 1 - y, -z$; (ii) $-x, 2 - y, -z$; (iii) $x, y + 1, z$.]

ring: C4/O1/O2 71.44 (12)° (*cf.* 74.4°); C5/O3/O4 19.3 (3)° (*cf.* 17.9°)]. Half the pyromellitic acid and one H_2O molecule constitute the asymmetric unit. The acid is located on an inversion centre.

The hydrogen-bonding array extends the structure into three dimensions through O—H···O hydrogen bonds (Table 2), with hydrogen bonds between carboxyl groups in neighbouring molecules creating $R_2^2(14)$ graph-set motifs (Etter, 1990; Etter & MacDonald, 1990; Bernstein *et al.*, 1995) and hydrogen bonding between two H_2O molecules and two carbonyl O atom acceptors forming an almost square $R_4^2(8)$ ring motif (Fig. 2).

Experimental

Colourless X-ray quality crystals of the title compound were grown by slow evaporation of an acetonitrile solution of pyromellitic acid at room temperature. The water originated from wet acetontrile and/or atmospheric moisture.

Crystal data

$C_{10}H_6O_8{\cdot}2H_2O$
$M_r = 290.18$
Triclinic, $P\bar{1}$
$a = 5.4651$ (6) Å
$b = 6.4044$ (7) Å
$c = 9.1145$ (11) Å
$\alpha = 71.941$ (3)°
$\beta = 88.612$ (3)°
$\gamma = 72.933$ (3)°
$V = 289.16$ (6) Å^3
$Z = 1$
$D_x = 1.666$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1682 reflections
$\theta = 3.5$–28.4°
$\mu = 0.15$ mm^{-1}
$T = 160$ (2) K
Plate, colourless
0.48 × 0.42 × 0.09 mm

Data collection

Bruker SMART 1K CCD diffractometer
Thin-slice ω scans
Absorption correction: none
1735 measured reflections
1010 independent reflections
890 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.012$
$\theta_{\rm max} = 25.0°$
$h = -6 \rightarrow 6$
$k = -7 \rightarrow 6$
$l = -10 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.096$
$S = 1.06$
1010 reflections
99 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 0.025P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.21$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å).

C1—C3$^{\rm i}$	1.391 (2)	C4—O1	1.2120 (18)
C1—C2	1.402 (2)	C4—O2	1.3113 (18)
C1—C4	1.502 (2)	C5—O3	1.2191 (18)
C2—C3	1.391 (2)	C5—O4	1.3033 (19)
C2—C5	1.500 (2)		

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*···O1	0.85 (2)	1.92 (2)	2.7664 (16)	171.6 (18)
O2—H2···O3$^{\rm i}$	0.84	1.85	2.6724 (14)	165
O4—H4···O1*W*$^{\rm iii}$	0.84	1.73	2.5656 (14)	175
O1*W*—H2*W*···O1$^{\rm ii}$	0.81 (2)	2.11 (2)	2.8586 (16)	152.0 (19)
O1*W*—H2*W*···O3$^{\rm iv}$	0.81 (2)	2.55 (2)	3.0642 (16)	122.4 (17)

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

Aromatic H and carboxyl OH atoms were placed in geometrically calculated positions, and refined using a riding model (with an aromatic C—H distance of 0.95 Å and an O—H distance of 0.84 Å), while the coordinates of the water H atoms were freely refined. $U_{\rm iso}$ values were set to $1.2U_{\rm eq}$(C) for aryl H [$1.5U_{\rm eq}$(O) for OH].

Data collection: *SMART* (Siemens, 1994); cell refinement: *SAINT* (Siemens, 1994); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

We acknowledge the EPSRC for the provision of a studentship (SHD).

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