Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,4-Dihydroxybenzoic acid acetonitrile solvate at 120 K

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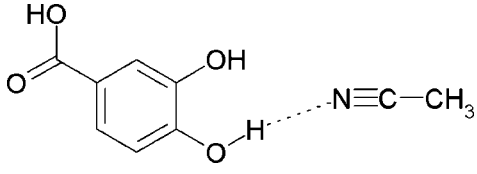
Received 5 June 2007; accepted 19 June 2007

Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.001 Å; R factor = 0.045; wR factor = 0.131; data-to-parameter ratio = 26.0.

The title solvate, $C_7H_6O_4\cdot C_2H_3N$, comprises two molecules, one acid and one solvent. The main molecules are arranged in layers by a two-dimensional hydrogen-bond network. In this way, channels are formed along the [100] axis, which are occupied by tail-to-tail solvent molecules in a manner similar to that observed previously in related structures. However, the hydrogen bond between an acetonitrile N atom and one of the hydroxyl groups is relatively normal, with an N···O separation of 2.8189 (10) Å. The placement of the solvent molecules makes the whole structure unstable under ambient conditions. Several minutes after removing the crystal from the mother solution it became milky, which probably indicates release of the solvent from the crystal structure.

Related literature

Related structures have been described, in which solvent molecules fill channels generated by a supramolecular arrangement (Aitipamula & Nangia, 2005; Gerkensmeier *et al.*, 1999).



Experimental

Crystal data

$C_7H_6O_4\cdot C_2H_3N$
M_r = 195.17
Monoclinic, $P2_1/c$
a = 6.3792 (6) Å
b = 10.4941 (8) Å
c = 13.7634 (12) Å
β = 99.4790 (14)°
V = 908.80 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 120 (2) K
0.4 × 0.2 × 0.2 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
9774 measured reflections
3948 independent reflections
3466 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.131
S = 1.05
3948 reflections
152 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	0.93 (2)	1.68 (2)	2.6056 (9)	174 (2)
O7—H7···O9^{ii}	0.87 (2)	1.84 (2)	2.6878 (8)	164.8 (17)
O9—H9···N12	0.82 (2)	2.07 (2)	2.8189 (10)	153.0 (18)

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

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *HKL* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2117).

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 doi:10.1107/S160053680702987X

supplementary materials

Acta Cryst. (2007). E**63**, o3289 [**doi:10.1107/S160053680702987X**]

3,4-Dihydroxybenzoic acid acetonitrile solvate at 120 K

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Experimental

An amount of 3,4-dihydroxybenzoic acid monohydrate (0.2 g) were diluted in 6 ml of acetonitrile. The resulting mixture was warmed to 333 K and then slowly cooled to 293 K. After several hours, colourless crystals were formed.

Refinement

All H atoms for the 3,4-dihydroxybenzoic acid molecule were found in a difference map and refined isotropically. H atoms of the methyl group in acetonitrile were placed in idealized positions, with C—H bond lengths constrained to 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C14). This group was considered as a rigid group but allowed to rotate about C13—C14 σ bond.

Figures

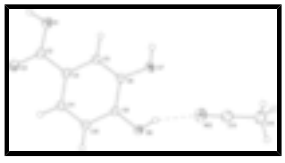

Fig. 1. *ORTEPII* view of the title solvate. The dashed bond shows the hydrogen bond connecting main molecule and solvent in the asymmetric unit.

3,4-Dihydroxybenozic acid acetonitile solvate

Crystal data

$C_7H_6O_4·C_2H_3N$	F_{000} = 408
M_r = 195.17	D_x = 1.427 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2242 reflections
a = 6.3792 (6) Å	θ = 1.0–35.0º
b = 10.4941 (8) Å	μ = 0.11 mm^{-1}
c = 13.7634 (12) Å	T = 120 (2) K
β = 99.4790 (14)º	Parallelepiped, colourless
V = 908.80 (14) Å^3	0.4 × 0.2 × 0.2 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	3466 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.026
T = 120(2) K	θ_{max} = 35.0º

CCD scans	θ_{min} = 3.6º
Absorption correction: none	h = −10→10
9774 measured reflections	k = −12→16
3948 independent reflections	l = −22→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.131	$w = 1/[\sigma^2(F_o^2) + (0.0745P)^2 + 0.1499P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.025
3948 reflections	$\Delta\rho_{max}$ = 0.49 e Å^{-3}
152 parameters	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.25161 (9)	1.05508 (6)	0.06328 (5)	0.02453 (13)
H1	−0.381 (4)	1.080 (2)	0.0265 (17)	0.073 (6)*
C2	−0.24757 (11)	0.93132 (7)	0.07528 (5)	0.01851 (13)
O3	−0.39826 (10)	0.86069 (6)	0.04151 (5)	0.02659 (14)
C4	−0.04961 (10)	0.87984 (7)	0.13234 (5)	0.01694 (13)
C5	0.12150 (11)	0.96085 (7)	0.16568 (5)	0.01803 (13)
H5	0.113 (2)	1.0546 (14)	0.1515 (10)	0.030 (3)*
C6	0.30668 (11)	0.91081 (7)	0.21885 (5)	0.01761 (13)
O7	0.48099 (9)	0.98127 (6)	0.25608 (5)	0.02552 (14)
H7	0.464 (3)	1.059 (2)	0.2347 (13)	0.052 (5)*
C8	0.32202 (10)	0.77978 (7)	0.23852 (5)	0.01706 (13)
O9	0.50348 (9)	0.72937 (5)	0.29073 (5)	0.02233 (13)
H9	0.596 (3)	0.7824 (19)	0.3070 (14)	0.056 (5)*
C10	0.15149 (11)	0.69939 (7)	0.20561 (6)	0.01884 (13)
H10	0.1655 (19)	0.6080 (12)	0.2212 (9)	0.023 (3)*
C11	−0.03439 (11)	0.74969 (7)	0.15260 (5)	0.01867 (13)
H11	−0.155 (2)	0.6970 (14)	0.1283 (10)	0.030 (3)*
N12	0.88052 (13)	0.84338 (8)	0.38793 (6)	0.02975 (16)
C13	1.05256 (13)	0.86299 (8)	0.42443 (6)	0.02431 (15)
C14	1.26991 (13)	0.89099 (10)	0.47069 (7)	0.02865 (18)
H14A	1.3432	0.9365	0.4239	0.043*
H14B	1.3448	0.8111	0.4902	0.043*
H14C	1.2680	0.9442	0.5291	0.043*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0197 (2)	0.0181 (3)	0.0325 (3)	0.00133 (19)	−0.0057 (2)	0.0037 (2)
C2	0.0154 (3)	0.0175 (3)	0.0212 (3)	0.0009 (2)	−0.0013 (2)	−0.0003 (2)
O3	0.0183 (3)	0.0207 (3)	0.0363 (3)	−0.00038 (19)	−0.0086 (2)	−0.0010 (2)
C4	0.0134 (3)	0.0165 (3)	0.0196 (3)	0.0007 (2)	−0.0014 (2)	−0.0001 (2)
C5	0.0147 (3)	0.0148 (3)	0.0232 (3)	0.0003 (2)	−0.0010 (2)	0.0014 (2)
C6	0.0135 (3)	0.0138 (3)	0.0241 (3)	−0.00094 (19)	−0.0012 (2)	0.0004 (2)
O7	0.0162 (2)	0.0147 (2)	0.0414 (3)	−0.00371 (18)	−0.0077 (2)	0.0035 (2)
C8	0.0130 (2)	0.0140 (3)	0.0227 (3)	0.00081 (19)	−0.0012 (2)	0.0000 (2)
O9	0.0148 (2)	0.0150 (2)	0.0338 (3)	0.00113 (17)	−0.00611 (19)	0.00066 (19)
C10	0.0158 (3)	0.0135 (3)	0.0257 (3)	−0.0006 (2)	−0.0012 (2)	−0.0003 (2)
C11	0.0151 (3)	0.0157 (3)	0.0237 (3)	−0.0012 (2)	−0.0014 (2)	−0.0011 (2)
N12	0.0237 (3)	0.0309 (4)	0.0329 (4)	0.0007 (3)	−0.0004 (3)	−0.0005 (3)
C13	0.0226 (3)	0.0236 (3)	0.0258 (3)	0.0024 (3)	0.0012 (3)	−0.0013 (3)
C14	0.0204 (3)	0.0325 (4)	0.0312 (4)	0.0012 (3)	−0.0013 (3)	−0.0052 (3)

Geometric parameters (Å, °)

O1—C2	1.3089 (9)	C8—O9	1.3646 (8)
O1—H1	0.93 (2)	C8—C10	1.3922 (10)
C2—O3	1.2419 (9)	O9—H9	0.82 (2)
C2—C4	1.4753 (10)	C10—C11	1.3900 (10)
C4—C11	1.3942 (10)	C10—H10	0.983 (13)
C4—C5	1.3999 (10)	C11—H11	0.960 (14)
C5—C6	1.3864 (10)	N12—C13	1.1478 (12)
C5—H5	1.003 (14)	C13—C14	1.4572 (12)
C6—O7	1.3631 (9)	C14—H14A	0.9800
C6—C8	1.4017 (10)	C14—H14B	0.9800
O7—H7	0.87 (2)	C14—H14C	0.9800
C2—O1—H1	110.1 (14)	C10—C8—C6	120.37 (6)
O3—C2—O1	123.16 (7)	C8—O9—H9	113.2 (13)
O3—C2—C4	121.51 (7)	C11—C10—C8	119.60 (6)
O1—C2—C4	115.33 (6)	C11—C10—H10	121.6 (7)
C11—C4—C5	120.30 (6)	C8—C10—H10	118.8 (7)
C11—C4—C2	119.37 (6)	C10—C11—C4	120.16 (6)
C5—C4—C2	120.33 (6)	C10—C11—H11	121.9 (9)
C6—C5—C4	119.58 (6)	C4—C11—H11	118.0 (9)
C6—C5—H5	119.2 (8)	N12—C13—C14	178.69 (10)
C4—C5—H5	121.2 (8)	C13—C14—H14A	109.5
O7—C6—C5	124.35 (6)	C13—C14—H14B	109.5
O7—C6—C8	115.65 (6)	H14A—C14—H14B	109.5
C5—C6—C8	119.99 (6)	C13—C14—H14C	109.5
C6—O7—H7	109.3 (12)	H14A—C14—H14C	109.5
O9—C8—C10	119.08 (6)	H14B—C14—H14C	109.5
O9—C8—C6	120.54 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3^{i}	0.93 (2)	1.68 (2)	2.6056 (9)	174 (2)
O7—H7···O9^{ii}	0.87 (2)	1.84 (2)	2.6878 (8)	164.8 (17)
O9—H9···N12	0.82 (2)	2.07 (2)	2.8189 (10)	153.0 (18)

Symmetry codes: (i) −*x*−1, −*y*+2, −*z*; (ii) −*x*+1, *y*+1/2, −*z*+1/2.

Fig. 1