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Crystal structure of 4-acetamidobenzoic acid monohydrate

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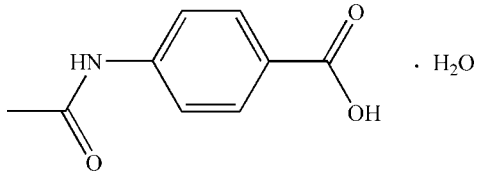
In the title compound, $C_9H_9NO_3\cdot H_2O$, the plane of the acetamide group is oriented at 20.52 (8)° with respect to the benzene ring, whereas the plane of the carboxylic acid group is essentially coplanar with the benzene ring [maximum deviation = 0.033 (1) Å]. In the crystal, classical O—H···O and N—H···O hydrogen bonds and weak C—H···O hydrogen bonds link the organic molecules and water molecules of crystallization into a three-dimensional supramolecular architecture.

Keywords: crystal structure; 4-acetamidobenzoic acid; hydrogen bonding; hydrated carboxylic acid.

CCDC reference: 906509

1. Related literature

For applications of 4-acetamidobenzoic acid in coordination chemistry, see: Yin *et al.* (2011); Wang *et al.* (2009). For related structures, see: Kashino *et al.* (1986); Jedrzejas *et al.* (1995).



2. Experimental

2.1. Crystal data

$C_9H_9NO_3\cdot H_2O$
M_r = 197.19
Monoclinic, $P2_1/c$
a = 6.6712 (13) Å
b = 28.870 (6) Å
c = 4.992 (1) Å
β = 100.01 (3)°
V = 946.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.31 × 0.28 × 0.26 mm

2.2. Data collection

Rigaku MM007-HF CCD (Saturn 724+) diffractometer
7525 measured reflections
1819 independent reflections
1448 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.116
S = 1.06
1819 reflections
140 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H4···O4^{i}	0.89 (2)	2.17 (2)	3.003 (2)	155.4 (17)
O1—H1···O2^{ii}	0.88 (2)	1.75 (2)	2.6285 (18)	174 (2)
O4—H2W···O4^{iii}	0.92 (2)	1.98 (2)	2.8920 (14)	176 (2)
O4—H1W···O3^{iv}	0.83 (3)	1.93 (3)	2.7549 (18)	173 (2)
C9—H9C···O3^{i}	0.96	2.57	3.484 (2)	160

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

Data collection: *CrystalStructure* (Rigaku/MSC, 2006); cell refinement: *CrystalStructure*; data reduction: *CrystalStructure*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: XU5824).

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supporting information

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Crystal structure of 4-acetamidobenzoic acid monohydrate

Wen-Juan Cai, Shao-Ming Chi, Jun-Feng Kou and Feng-Yi Liu

S1. Comment

In the past few years, 4-acetamidobenzoic acid has attracted increasing attention as a multifunctional ligand (Kashino *et al.*, 1986; Jedrzejas *et al.*, 1995). It shows potential diversified coordination and exhibits various coordination modes, such as monodentate, chelating, and multidentate bridging, *etc* in those reported complexes (Yin *et al.*, 2011; Wang *et al.*, 2009). Herein we report the synthesis and structure of the title compound.

The structure of the title compound is shown in Fig. 1, Fig. 2 and hydrogen bond geometry is given in Table 1. In the title compound, the acetamide moiety is oriented with respect to the benzene ring at 20.52 (8)° whereas the carboxyl group is essentially co-planar with the benzene ring [the maximum deviation = 0.033 (1) Å]. In the crystal, classic O—H···O, N—H···O hydrogen bonds and weak C—H···O hydrogen bond link organic molecules and crystalline water molecules into the three dimensional supramolecular architecture.

S2. Experimental

4-Aminobenzoic acid (5 mmol, 686 mg) was added to 20 ml acetic anhydride. The mixture was then refluxed under argon for 8 h. The excess of acetic anhydride was then evaporated under reduced pressure. Water was then added to the resulting solid. The colorless block crystals were obtained after 48 h. Yield: 90% (based on 4-aminobenzoic acid).

S3. Refinement

H atoms attached to N and O atoms were located in a difference Fourier map and refined with positional parameters, U_{iso}(H) = 1.2U_{eq}(N) and U_{iso}(H) = 1.5U_{eq}(O). H atoms attached to carbons were geometrically fixed and allowed to ride on the corresponding non-H atom with C—H = 0.96 Å, U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other.

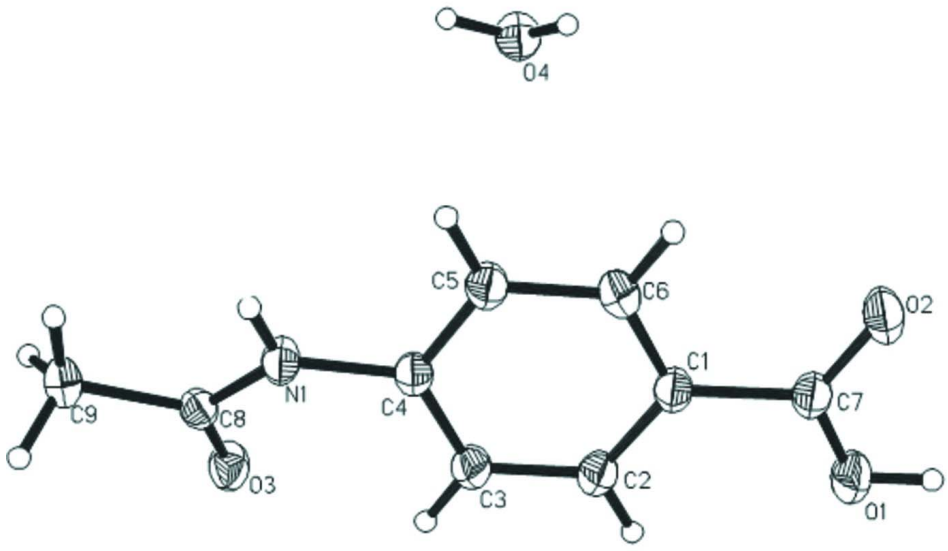

Figure 1

The molecular structure of the title compound, with atom labels and 30% probability displacement ellipsoids.

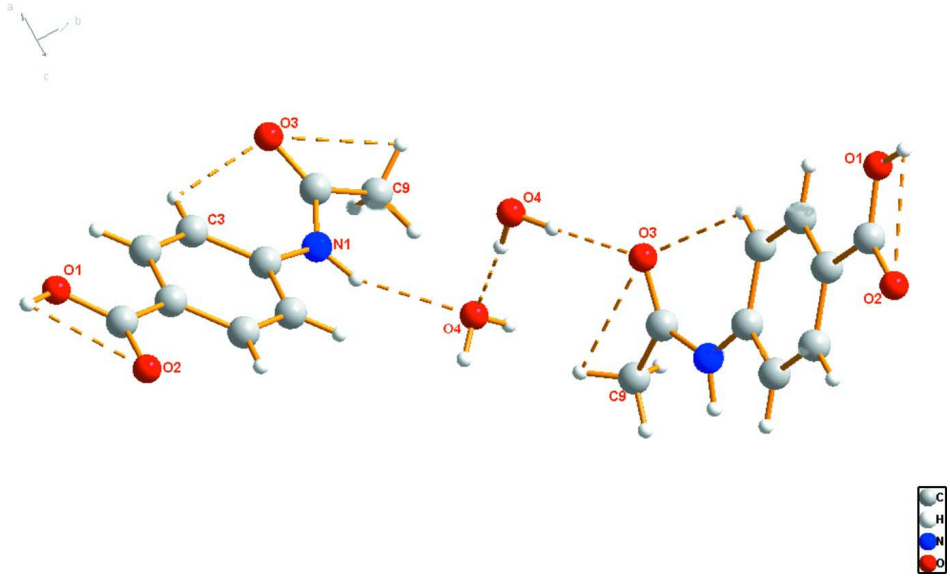

Figure 2

Hydrogen bonds are shown as brown dashed lines.

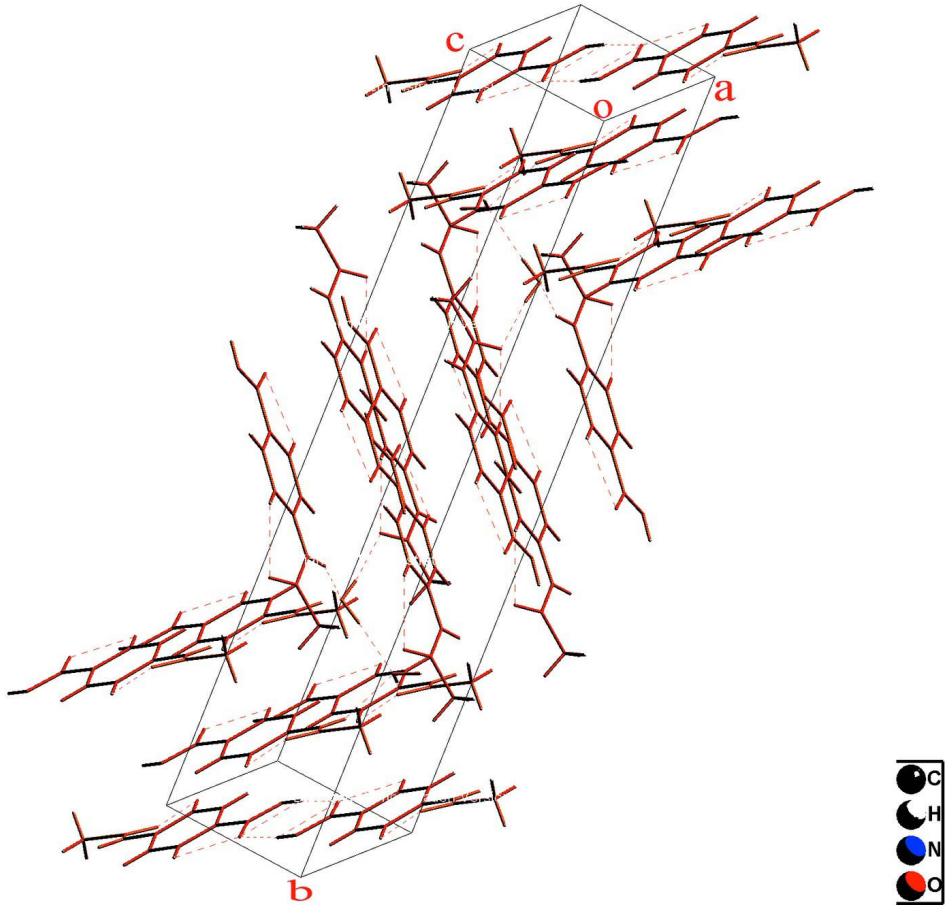

Figure 3

A view of the crystal packing.

4-Acetamidobenzoic acid monohydrate

Crystal data

$C_9H_9NO_3·H_2O$
M_r = 197.19
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 6.6712 (13) Å
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c = 4.992 (1) Å
β = 100.01 (3)°
V = 946.8 (3) Å^3
Z = 4
$F(000)$ = 416
D_x = 1.383 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8729 reflections
θ = 3.1–26.0°
μ = 0.11 mm^{-1}
T = 293 K
Block, colorless
0.31 × 0.28 × 0.26 mm

Data collection

Rigaku MM007-HF CCD (Saturn 724+) diffractometer
Radiation source: rotating anode
Confocal monochromator
ω scans at fixed χ = 45°
7525 measured reflections
1819 independent reflections
1448 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 26.0°, θ_{min} = 3.1°
h = −8→8
k = −35→35
l = −6→5

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.116$
$S = 1.06$
1819 reflections
140 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0607P)^2 + 0.1929P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6802 (3)	0.43025 (5)	0.5354 (3)	0.0370 (4)
C2	0.8849 (3)	0.42983 (5)	0.6534 (3)	0.0432 (4)
H2	0.9751	0.4497	0.5886	0.052*
C3	0.9563 (3)	0.40000 (5)	0.8671 (3)	0.0420 (4)
H3	1.0932	0.4000	0.9459	0.050*
C4	0.8203 (2)	0.37010 (5)	0.9622 (3)	0.0346 (4)
C5	0.6157 (3)	0.37085 (6)	0.8459 (4)	0.0430 (4)
H5	0.5249	0.3511	0.9107	0.052*
C6	0.5461 (3)	0.40082 (6)	0.6342 (3)	0.0439 (4)
H6	0.4086	0.4012	0.5578	0.053*
C7	0.6019 (3)	0.46170 (5)	0.3049 (3)	0.0394 (4)
C8	1.0650 (2)	0.32340 (5)	1.2903 (3)	0.0344 (3)
C9	1.0717 (3)	0.29285 (6)	1.5361 (3)	0.0423 (4)
H9A	1.1507	0.2657	1.5161	0.063*
H9B	0.9358	0.2839	1.5528	0.063*
H9C	1.1329	0.3095	1.6961	0.063*
H1	0.684 (3)	0.5062 (7)	0.080 (4)	0.063*
H4	0.775 (3)	0.3275 (7)	1.251 (4)	0.051*
H1W	0.452 (4)	0.2928 (8)	0.293 (5)	0.063*
H2W	0.545 (4)	0.2597 (7)	0.471 (5)	0.063*
N1	0.8780 (2)	0.33893 (5)	1.1819 (3)	0.0386 (3)
O1	0.7308 (2)	0.48727 (4)	0.2144 (3)	0.0554 (4)
O2	0.4148 (2)	0.46159 (4)	0.2106 (3)	0.0546 (4)
O3	1.21944 (18)	0.33300 (4)	1.1992 (2)	0.0475 (3)

O4	0.5523 (2)	0.27530 (5)	0.3128 (3)	0.0486 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0446 (9)	0.0327 (7)	0.0322 (8)	0.0049 (6)	0.0030 (7)	0.0027 (6)
C2	0.0428 (9)	0.0409 (8)	0.0444 (9)	−0.0030 (7)	0.0037 (7)	0.0100 (7)
C3	0.0355 (8)	0.0426 (8)	0.0452 (9)	−0.0011 (6)	−0.0006 (7)	0.0091 (7)
C4	0.0379 (8)	0.0341 (7)	0.0311 (7)	0.0040 (6)	0.0041 (6)	0.0028 (6)
C5	0.0369 (9)	0.0463 (9)	0.0445 (9)	−0.0028 (7)	0.0031 (7)	0.0120 (7)
C6	0.0384 (9)	0.0465 (9)	0.0428 (9)	0.0018 (7)	−0.0037 (7)	0.0064 (7)
C7	0.0490 (10)	0.0339 (7)	0.0338 (8)	0.0049 (7)	0.0033 (7)	0.0020 (6)
C8	0.0387 (8)	0.0331 (7)	0.0297 (7)	0.0021 (6)	0.0012 (6)	−0.0010 (5)
C9	0.0467 (10)	0.0463 (9)	0.0326 (8)	0.0083 (7)	0.0035 (7)	0.0071 (6)
N1	0.0358 (7)	0.0428 (7)	0.0369 (7)	0.0029 (6)	0.0056 (6)	0.0113 (5)
O1	0.0595 (9)	0.0540 (7)	0.0495 (7)	−0.0018 (6)	0.0003 (7)	0.0215 (6)
O2	0.0504 (8)	0.0573 (7)	0.0515 (7)	0.0061 (6)	−0.0043 (6)	0.0177 (6)
O3	0.0349 (6)	0.0563 (7)	0.0500 (7)	0.0022 (5)	0.0041 (5)	0.0156 (5)
O4	0.0409 (7)	0.0569 (8)	0.0496 (7)	0.0028 (5)	0.0122 (6)	0.0053 (6)

Geometric parameters (Å, º)

C1—C6	1.386 (2)	C7—O2	1.255 (2)
C1—C2	1.390 (2)	C7—O1	1.274 (2)
C1—C7	1.487 (2)	C8—O3	1.228 (2)
C2—C3	1.389 (2)	C8—N1	1.347 (2)
C2—H2	0.9300	C8—C9	1.505 (2)
C3—C4	1.394 (2)	C9—H9A	0.9600
C3—H3	0.9300	C9—H9B	0.9600
C4—C5	1.388 (2)	C9—H9C	0.9600
C4—N1	1.4193 (19)	N1—H4	0.89 (2)
C5—C6	1.382 (2)	O1—H1	0.881 (16)
C5—H5	0.9300	O4—H1W	0.83 (3)
C6—H6	0.9300	O4—H2W	0.92 (2)
C6—C1—C2	119.44 (14)	O2—C7—O1	123.84 (14)
C6—C1—C7	119.22 (16)	O2—C7—C1	118.76 (15)
C2—C1—C7	121.34 (15)	O1—C7—C1	117.40 (16)
C3—C2—C1	120.74 (15)	O3—C8—N1	123.64 (14)
C3—C2—H2	119.6	O3—C8—C9	121.75 (15)
C1—C2—H2	119.6	N1—C8—C9	114.60 (14)
C2—C3—C4	119.27 (16)	C8—C9—H9A	109.5
C2—C3—H3	120.4	C8—C9—H9B	109.5
C4—C3—H3	120.4	H9A—C9—H9B	109.5
C5—C4—C3	119.93 (14)	C8—C9—H9C	109.5
C5—C4—N1	116.64 (14)	H9A—C9—H9C	109.5
C3—C4—N1	123.41 (15)	H9B—C9—H9C	109.5
C6—C5—C4	120.34 (15)	C8—N1—C4	128.98 (14)

C6—C5—H5	119.8	C8—N1—H4	116.6 (13)
C4—C5—H5	119.8	C4—N1—H4	114.4 (13)
C5—C6—C1	120.27 (16)	C7—O1—H1	117.5 (15)
C5—C6—H6	119.9	H1W—O4—H2W	104 (2)
C1—C6—H6	119.9		
C6—C1—C2—C3	0.6 (2)	C7—C1—C6—C5	179.16 (15)
C7—C1—C2—C3	−179.47 (15)	C6—C1—C7—O2	1.9 (2)
C1—C2—C3—C4	0.3 (3)	C2—C1—C7—O2	−178.08 (15)
C2—C3—C4—C5	−0.9 (2)	C6—C1—C7—O1	−177.98 (15)
C2—C3—C4—N1	−178.96 (15)	C2—C1—C7—O1	2.1 (2)
C3—C4—C5—C6	0.6 (3)	O3—C8—N1—C4	−3.9 (3)
N1—C4—C5—C6	178.79 (15)	C9—C8—N1—C4	176.06 (14)
C4—C5—C6—C1	0.3 (3)	C5—C4—N1—C8	163.45 (15)
C2—C1—C6—C5	−0.9 (2)	C3—C4—N1—C8	−18.4 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H4···O4^{i}	0.89 (2)	2.17 (2)	3.003 (2)	155.4 (17)
O1—H1···O2^{ii}	0.88 (2)	1.75 (2)	2.6285 (18)	174 (2)
O4—H2*W*···O4^{iii}	0.92 (2)	1.98 (2)	2.8920 (14)	176 (2)
O4—H1*W*···O3^{iv}	0.83 (3)	1.93 (3)	2.7549 (18)	173 (2)
C9—H9*C*···O3^{i}	0.96	2.57	3.484 (2)	160

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