Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,5-Dicarboxyanilinium nitrate dihydrate

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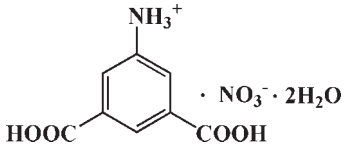
Received 28 January 2010; accepted 9 February 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.054; wR factor = 0.129; data-to-parameter ratio = 14.5.

In the crystal of the title compound, $C_8H_8NO_4^+\cdot NO_3^-\cdot 2H_2O$, the 5-ammonioisophthalic acid cations, the nitrate anions and the water molecules are linked by N—H···O, O—H···O and C—H···O hydrogen bonds into a three-dimensional network. The structure is further stabilized by aromatic π–π stacking interactions, with centroid–centroid separations of 3.827 (2) Å.

Related literature

For the crystal structure of 5-aminoisophthalic acid hemihydrate, see: Dobson *et al.* (1998). For the use of 5-aminoisophthalic acid as a ligand, see: Liao *et al.* (2004).



Experimental

Crystal data

$C_8H_8NO_4^+\cdot NO_3^-\cdot 2H_2O$
M_r = 280.20
Monoclinic, $P2_1/c$
a = 8.3436 (17) Å
b = 8.6234 (17) Å
c = 16.862 (3) Å
β = 97.31 (3)°
V = 1203.4 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 K
0.35 × 0.25 × 0.10 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.960, T_{max} = 0.986
12169 measured reflections
2753 independent reflections
1905 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.129
S = 1.07
2753 reflections
190 parameters
?
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O6^{i}	0.93	2.58	3.324 (3)	138
O9—H9A···O7^{ii}	0.88 (4)	2.39 (4)	3.048 (3)	133 (3)
O9—H9A···O5^{ii}	0.88 (4)	1.94 (4)	2.801 (3)	168 (4)
O8—H8B···O3^{iii}	0.85 (5)	2.29 (5)	3.057 (3)	149 (4)
O8—H8A···O2^{iv}	0.97 (4)	2.00 (4)	2.882 (3)	151 (3)
O4—H4···O3^{v}	0.82	1.84	2.652 (2)	169
N1—H1C···O9^{vi}	0.89	1.98	2.839 (3)	163
N1—H1B···O6^{vii}	0.89	2.00	2.859 (3)	161
N1—H1B···O5^{vii}	0.89	2.55	3.134 (3)	124
O9—H9B···O8	0.84 (5)	1.96 (5)	2.796 (3)	171 (5)
N1—H1A···O7^{i}	0.89	2.46	2.933 (3)	114
N1—H1A···O6^{i}	0.89	2.09	2.963 (3)	165
O1—H1···O9^{viii}	0.82	1.80	2.611 (3)	169

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (iv) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (v) $-x+1, -y, -z+1$; (vi) $x-1, -y+\frac{3}{2}, z-\frac{1}{2}$; (vii) $x, y+1, z$; (viii) $x-1, y, z$.

Data collection: *CrystalClear* (Rigaku 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PRPKAPPA* (Ferguson, 1999).

This work was supported by the Technical Fund Financing Projects (grant Nos. 9207042464 and 9207041482) from Southeast University to Zhi-Rong Qu.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2414).

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Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
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Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
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supporting information

Acta Cryst. (2010). E**66**, o667 [doi:10.1107/S1600536810005362]

3,5-Dicarboxyanilinium nitrate dihydrate

Wen-Xian Liang and Yun-Ti Zhu

S1. Comment

5-Aminobenzene-1,3-dioic acid (5-aminoisophthalic acid) is an important molecule due to its amphoteric property. The report on 5-aminobenzene-1,3-dioic acid hemihydrate (Dobson *et al.* 1998) is one of a series on hydrogen bonding in aminosubstituted carboxylic acids, and follows reports on a novel tetragonal phase of aminobutyric acid, on 8-amino-caprylic acid and on 3-aminoisobutyric acid monohydrate. In addition, 5-aminobenzene-1,3-dioic acid is an attractive ligand for use in the generation of polar coordination polymers (Liao *et al.*, 2004).

The asymmetric unit of the title compound comprises two water molecules, a 5-ammonioisophthalic acid cation and one nitrate anion (Fig. 1). The crystal packing is stabilized by hydrogen bonds of N—H···O, O—H···O, C—H···O (Table 1) connecting neighbouring water molecules, cations and anions into a three-dimensional network (Fig. 2). The structure is further stabiized by aromatic $\pi\cdots\pi$ stacking interactions, with centroid-to-centroid separations of 3.827 (2) Å.

S2. Experimental

5-Aminoisophthalic acid (1.81 g, 10 mmol) was dissolved in water (5 ml), ethanol (20 ml) and nitric acid (0.57 g, 10 mmol) and the solution was filtered. After slowly evaporating over a period of 3 d, colourless prismatic crystals of the title compound suitable for X-ray diffraction analysis were isolated.

S3. Refinement

All the H atoms were calculated geometrically and were allowed to ride on their parent atomsd, with C—H = 0.93–0.97 Å, N—H = 0.89 Å, and with U_{iso}(H) = 1.2Ueq(C) and 1.5U_{eq}(N).

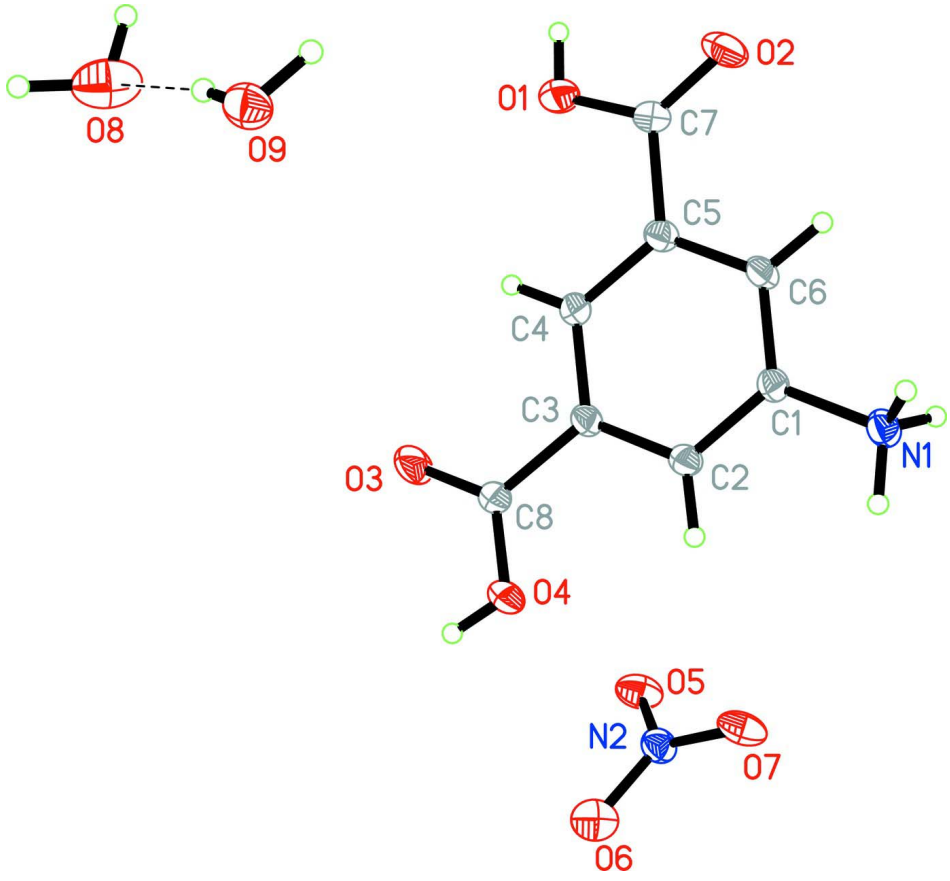

Figure 1

The asymmetric unit of the title compound, with displacement ellipsoids drawn at the 30% probability level. Intermolecular hydrogen bonds are shown as dashed lines.

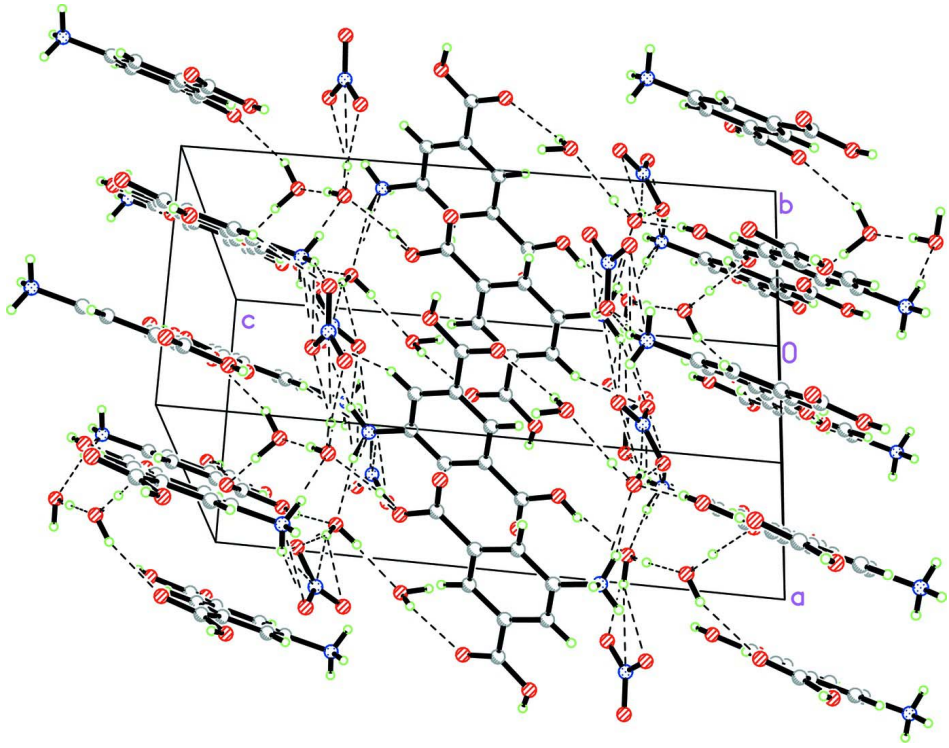

Figure 2

Packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

3,5-Dicarboxyanilinium nitrate dihydrate

Crystal data

$C_8H_8NO_4^+·NO_3^-·2H_2O$
$M_r = 280.20$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.3436$ (17) Å
$b = 8.6234$ (17) Å
$c = 16.862$ (3) Å
$\beta = 97.31$ (3)°
$V = 1203.4$ (4) Å^3
$Z = 4$

$F(000) = 584$
$D_x = 1.547$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1753 reflections
$\theta = 3.1$–27.5°
$\mu = 0.14$ mm^{-1}
$T = 293$ K
Prism, colourless
0.35 × 0.25 × 0.10 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD profile fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.960$, $T_{max} = 0.986$

12169 measured reflections
2753 independent reflections
1905 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.129$
$S = 1.07$
2753 reflections
190 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.7735P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.0014 (1)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O4	0.4952 (2)	0.14675 (19)	0.42859 (10)	0.0442 (5)
H4	0.5275	0.0603	0.4432	0.066*
N1	0.3108 (2)	0.6452 (2)	0.29415 (11)	0.0328 (5)
H1A	0.3651	0.5813	0.2658	0.049*
H1B	0.3632	0.7350	0.3010	0.049*
H1C	0.2126	0.6614	0.2682	0.049*
C2	0.3575 (3)	0.4279 (2)	0.38793 (13)	0.0280 (5)
H2	0.4070	0.3744	0.3498	0.034*
O1	0.1136 (3)	0.6025 (2)	0.62566 (11)	0.0545 (6)
H1	0.0669	0.6559	0.6558	0.082*
O3	0.3885 (3)	0.1368 (2)	0.54276 (11)	0.0513 (5)
C4	0.2667 (3)	0.4405 (2)	0.51780 (13)	0.0296 (5)
H4A	0.2556	0.3946	0.5667	0.036*
C1	0.2969 (3)	0.5756 (2)	0.37216 (13)	0.0264 (5)
C5	0.2069 (3)	0.5891 (3)	0.50071 (14)	0.0299 (5)
C6	0.2225 (3)	0.6570 (2)	0.42699 (13)	0.0300 (5)
H6	0.1830	0.7563	0.4152	0.036*
O2	0.0772 (3)	0.8101 (2)	0.54705 (12)	0.0605 (6)
C3	0.3432 (3)	0.3610 (2)	0.46132 (13)	0.0275 (5)
C7	0.1260 (3)	0.6799 (3)	0.55994 (14)	0.0356 (6)
O5	0.2218 (2)	−0.0029 (2)	0.30373 (12)	0.0461 (5)
O6	0.4607 (2)	−0.06093 (19)	0.27628 (11)	0.0441 (5)
O7	0.3495 (2)	0.15470 (19)	0.23390 (12)	0.0460 (5)
N2	0.3433 (2)	0.0312 (2)	0.27105 (12)	0.0333 (5)

C8	0.4106 (3)	0.2034 (3)	0.47971 (13)	0.0304 (5)
O9	0.9920 (2)	0.7569 (2)	0.73715 (12)	0.0422 (5)
O8	0.8512 (3)	0.5594 (3)	0.84014 (16)	0.0667 (7)
H9A	0.916 (5)	0.828 (5)	0.729 (2)	0.091 (13)*
H8A	0.904 (4)	0.500 (5)	0.885 (2)	0.089 (13)*
H8B	0.781 (6)	0.612 (5)	0.861 (3)	0.111 (16)*
H9B	0.949 (6)	0.706 (6)	0.771 (3)	0.14 (2)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O4	0.0653 (12)	0.0295 (9)	0.0394 (10)	0.0196 (9)	0.0133 (9)	0.0042 (7)
N1	0.0348 (11)	0.0289 (10)	0.0359 (11)	0.0043 (8)	0.0092 (9)	0.0069 (8)
C2	0.0321 (12)	0.0225 (11)	0.0299 (12)	0.0021 (9)	0.0057 (10)	−0.0010 (9)
O1	0.0824 (15)	0.0496 (11)	0.0354 (10)	0.0243 (10)	0.0225 (10)	0.0014 (9)
O3	0.0760 (14)	0.0357 (10)	0.0470 (11)	0.0214 (10)	0.0261 (10)	0.0177 (8)
C4	0.0365 (13)	0.0251 (11)	0.0269 (12)	0.0028 (9)	0.0025 (10)	0.0010 (9)
C1	0.0283 (11)	0.0236 (11)	0.0275 (11)	−0.0002 (9)	0.0038 (9)	0.0026 (9)
C5	0.0323 (12)	0.0258 (11)	0.0313 (12)	0.0017 (9)	0.0029 (10)	−0.0025 (9)
C6	0.0345 (12)	0.0204 (10)	0.0348 (12)	0.0054 (9)	0.0027 (10)	0.0003 (9)
O2	0.0920 (16)	0.0371 (11)	0.0573 (13)	0.0270 (11)	0.0282 (12)	0.0011 (9)
C3	0.0317 (12)	0.0209 (10)	0.0293 (12)	0.0016 (9)	0.0021 (9)	0.0008 (9)
C7	0.0421 (14)	0.0308 (13)	0.0343 (13)	0.0059 (11)	0.0063 (11)	−0.0055 (10)
O5	0.0385 (10)	0.0427 (10)	0.0604 (12)	−0.0026 (8)	0.0199 (9)	0.0066 (9)
O6	0.0436 (10)	0.0346 (9)	0.0562 (12)	0.0130 (8)	0.0149 (9)	0.0030 (8)
O7	0.0483 (11)	0.0269 (9)	0.0643 (12)	−0.0019 (8)	0.0128 (9)	0.0126 (8)
N2	0.0363 (11)	0.0263 (10)	0.0377 (11)	−0.0009 (9)	0.0063 (9)	−0.0016 (8)
C8	0.0379 (13)	0.0246 (11)	0.0290 (12)	0.0042 (10)	0.0049 (10)	−0.0001 (9)
O9	0.0356 (10)	0.0465 (11)	0.0460 (11)	0.0073 (9)	0.0106 (9)	−0.0011 (9)
O8	0.0748 (16)	0.0582 (14)	0.0742 (17)	0.0179 (12)	0.0371 (14)	0.0138 (12)

Geometric parameters (Å, º)

O4—C8	1.279 (3)	C4—H4A	0.9300
O4—H4	0.8200	C1—C6	1.371 (3)
N1—C1	1.464 (3)	C5—C6	1.395 (3)
N1—H1A	0.8900	C5—C7	1.496 (3)
N1—H1B	0.8900	C6—H6	0.9300
N1—H1C	0.8900	O2—C7	1.205 (3)
C2—C1	1.383 (3)	C3—C8	1.488 (3)
C2—C3	1.384 (3)	O5—N2	1.249 (2)
C2—H2	0.9300	O6—N2	1.256 (2)
O1—C7	1.309 (3)	O7—N2	1.240 (2)
O1—H1	0.8200	O9—H9A	0.88 (4)
O3—C8	1.243 (3)	O9—H9B	0.84 (5)
C4—C5	1.392 (3)	O8—H8A	0.97 (4)
C4—C3	1.393 (3)	O8—H8B	0.85 (5)

C8—O4—H4	109.5	C6—C5—C7	118.5 (2)
C1—N1—H1A	109.5	C1—C6—C5	119.2 (2)
C1—N1—H1B	109.5	C1—C6—H6	120.4
H1A—N1—H1B	109.5	C5—C6—H6	120.4
C1—N1—H1C	109.5	C2—C3—C4	120.3 (2)
H1A—N1—H1C	109.5	C2—C3—C8	119.59 (19)
H1B—N1—H1C	109.5	C4—C3—C8	120.1 (2)
C1—C2—C3	119.0 (2)	O2—C7—O1	124.5 (2)
C1—C2—H2	120.5	O2—C7—C5	122.6 (2)
C3—C2—H2	120.5	O1—C7—C5	112.9 (2)
C7—O1—H1	109.5	O7—N2—O5	120.9 (2)
C5—C4—C3	119.7 (2)	O7—N2—O6	119.8 (2)
C5—C4—H4A	120.2	O5—N2—O6	119.32 (19)
C3—C4—H4A	120.2	O3—C8—O4	123.7 (2)
C6—C1—C2	121.8 (2)	O3—C8—C3	120.5 (2)
C6—C1—N1	119.39 (19)	O4—C8—C3	115.8 (2)
C2—C1—N1	118.78 (19)	H9A—O9—H9B	96 (4)
C4—C5—C6	119.9 (2)	H8A—O8—H8B	103 (4)
C4—C5—C7	121.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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