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## 3,5-Dicarboxyanilinium nitrate dihydrate

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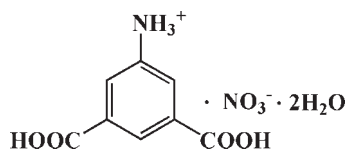
Received 28 January 2010; accepted 9 February 2010

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.054;  $wR$  factor = 0.129; data-to-parameter ratio = 14.5.

In the crystal of the title compound,  $\text{C}_8\text{H}_8\text{NO}_4^+ \cdot \text{NO}_3^- \cdot 2\text{H}_2\text{O}$ , the 5-ammonioisophthalic acid cations, the nitrate anions and the water molecules are linked by  $\text{N}-\text{H} \cdots \text{O}$ ,  $\text{O}-\text{H} \cdots \text{O}$  and  $\text{C}-\text{H} \cdots \text{O}$  hydrogen bonds into a three-dimensional network. The structure is further stabilized by aromatic  $\pi-\pi$  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

$\text{C}_8\text{H}_8\text{NO}_4^+ \cdot \text{NO}_3^- \cdot 2\text{H}_2\text{O}$   
 $M_r = 280.20$   
Monoclinic,  $P2_1/c$   
 $a = 8.3436$  (17) Å  
 $b = 8.6234$  (17) Å  
 $c = 16.862$  (3) Å  
 $\beta = 97.31$  (3)°

$V = 1203.4$  (4) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.14$  mm<sup>-1</sup>  
 $T = 293$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.055$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.129$   
 $S = 1.07$   
2753 reflections

190 parameters  
?  
 $\Delta\rho_{\text{max}} = 0.28$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.24$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

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

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

## References

- Dobson, A. J. & Gerkin, R. E. (1998). *Acta Cryst.* **C54**, 1503–1505.  
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**supplementary materials**

*Acta Cryst.* (2010). E66, o667 [ doi:10.1107/S1600536810005362 ]

### 3,5-Dicarboxyanilinium nitrate dihydrate

W.-X. Liang and Y.-T. Zhu

#### 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-aminocaprylic 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 $\cdots$ O, O—H $\cdots$ O, C—H $\cdots$ O (Table 1) connecting neighbouring water molecules, cations and anions into a three-dimensional network (Fig. 2). The structure is further stabilized by aromatic  $\pi\cdots\pi$  stacking interactions, with centroid-to-centroid separations of 3.827 (2) Å.

#### 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.

#### Refinement

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

#### Figures

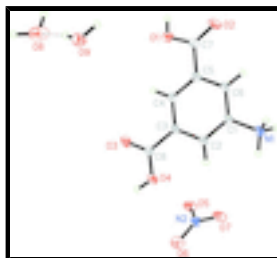


Fig. 1. The asymmetric unit of the title compound, with displacement ellipsoids drawn at the 30% probability level. Intermolecular hydrogen bonds are shown as dashed lines.

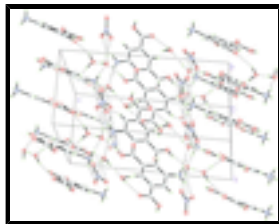


Fig. 2. Packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

## 3,5-Dicarboxyanilinium nitrate dihydrate

### Crystal data

$C_8H_8NO_4^+ \cdot NO_3^- \cdot 2H_2O$

$M_r = 280.20$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.3436 (17) \text{ \AA}$

$b = 8.6234 (17) \text{ \AA}$

$c = 16.862 (3) \text{ \AA}$

$\beta = 97.31 (3)^\circ$

$V = 1203.4 (4) \text{ \AA}^3$

$Z = 4$

$F(000) = 584$

$D_x = 1.547 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1753 reflections

$\theta = 3.1\text{--}27.5^\circ$

$\mu = 0.14 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Prism, colourless

$0.35 \times 0.25 \times 0.10 \text{ mm}$

### Data collection

Rigaku SCXmini  
diffractometer

Radiation source: fine-focus sealed tube  
graphite

Detector resolution:  $13.6612 \text{ pixels mm}^{-1}$

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  $I > 2\sigma(I)$

$R_{\text{int}} = 0.055$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 3.2^\circ$

$h = -10 \rightarrow 10$

$k = -11 \rightarrow 11$

$l = -21 \rightarrow 21$

### 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

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  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.28 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.24 \text{ e \AA}^{-3}$

Extinction correction: SHELXL97 (Sheldrick, 2008)

0 restraints

Extinction coefficient: 0.0014 (1)

Primary atom site location: structure-invariant direct methods

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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)*

## supplementary materials

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H9B                    0.949 (6)                    0.706 (6)                    0.771 (3)                    0.14 (2)\*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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 ( $\text{\AA}$ , $^\circ$ )

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 ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2 $\cdots$ O6 <sup>i</sup>	0.93	2.58	3.324 (3)	138.
O9—H9A $\cdots$ O7 <sup>ii</sup>	0.88 (4)	2.39 (4)	3.048 (3)	133 (3)
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O8—H8B $\cdots$ O3 <sup>iii</sup>	0.85 (5)	2.29 (5)	3.057 (3)	149 (4)
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O4—H4 $\cdots$ O3 <sup>v</sup>	0.82	1.84	2.652 (2)	169.
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N1—H1B $\cdots$ O6 <sup>vii</sup>	0.89	2.00	2.859 (3)	161.
N1—H1B $\cdots$ O5 <sup>vii</sup>	0.89	2.55	3.134 (3)	124.
O9—H9B $\cdots$ O8	0.84 (5)	1.96 (5)	2.796 (3)	171 (5)
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O1—H1 $\cdots$ O9 <sup>viii</sup>	0.82	1.80	2.611 (3)	169.

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

Fig. 1

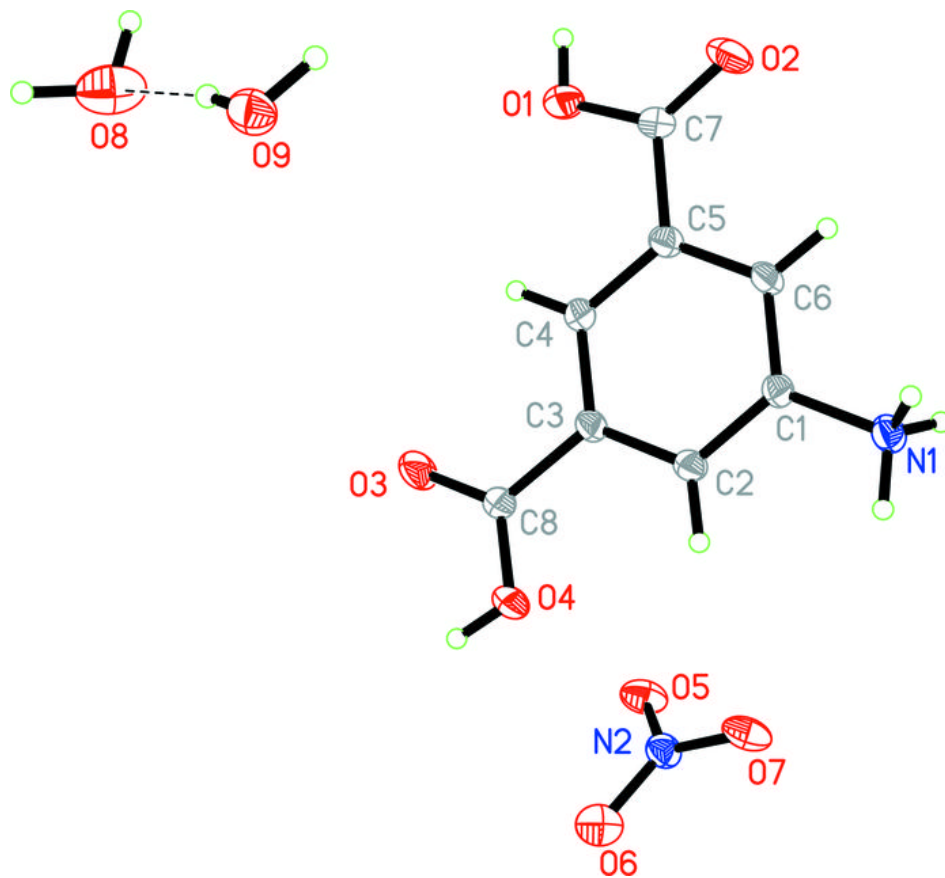




Fig. 2

