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Dimethylammonium 2-amino-5-nitroterephthalate hemihydrate

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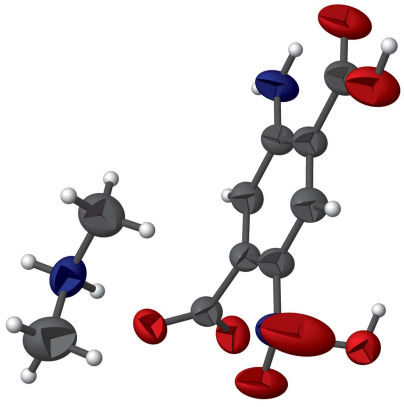
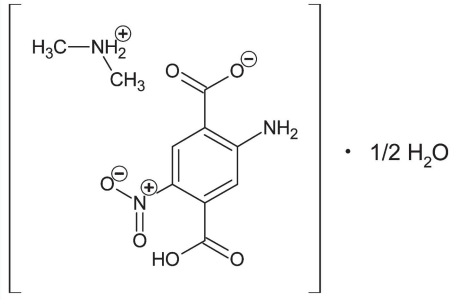
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound, $C_2H_8N^+\cdot C_8H_5N_2O_6^-\cdot 0.5H_2O$, comprises a monodeprotonated 2-amino-5-nitroterephthalate anion and a dimethylammonium counter-ion on general positions and a water molecule that lies on a twofold rotation axis. Extensive hydrogen bonding is observed between the carboxylate group and the dimethylammonium ion, the water molecule and the carboxylic acid group, as well as between the amino group, the water molecule and the carboxylic acid group (N—H···O and O—H···O hydrogen bonds are involved).

3D view



Chemical scheme



Structure description

The title compound comprises a mono-deprotonated benzene 1,4-dicarboxylic acid with an NH_2 and an NO_2 group at the 2- and 5-positions, respectively (Fig. 1). Charge balance is accomplished with a dimethylammonium counter-cation. Extensive N—H···O and O—H···O hydrogen bonding between the cation, the anion and the water molecule leads to a three-dimensional structure (Table 1, Fig. 2).

Functionalized terephthalate ions are of special interest for the synthesis of porous coordination polymers in order to tune host–guest interactions within functionalized pore surfaces (Biswas *et al.*, 2011). A compound with the same cation but the non-functionalized terephthalate ion, which does not contain an additional water molecule, has been reported (Zhao *et al.*, 2007). Extensive hydrogen bonding is also observed in this structure. Thus, the terephthalate ions are linked to each other by O—H···O hydrogen bonds, forming a one-dimensional polymeric chain. In addition, the terephthalate anions and dimethylammonium cations are connected into a three-dimensional structure by N—H···O hydrogen-bonds (Zhao *et al.*, 2007).

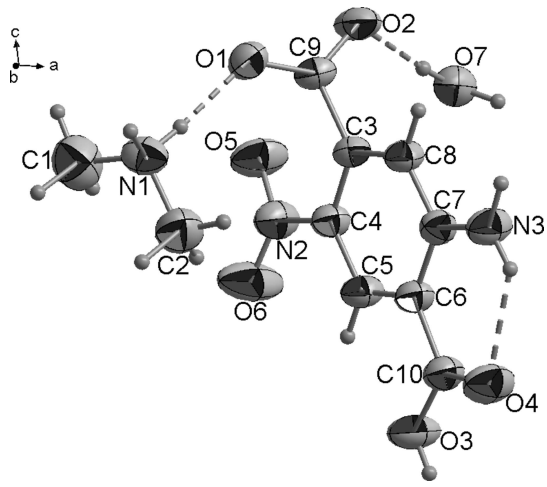


Figure 1
The components of the title compound (the atom O7 lies on a twofold axis) showing the atom labelling with displacement ellipsoids drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

Synthesis and crystallization

Dimethylammonium 2-amino-5-nitroterephtalate hemi hydrate was obtained from 10 mg 2-amino-5-nitro-terephthalic acid (0.18 mmol), 0.256 ml DMF and 0.244 ml H_2O. A 2 ml teflon-lined steel autoclave was used for the synthesis. The reactor was heated to 150°C for 24 h then cooled down to room temperature over 12 h, to give crystals suitable for X-ray data collection.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

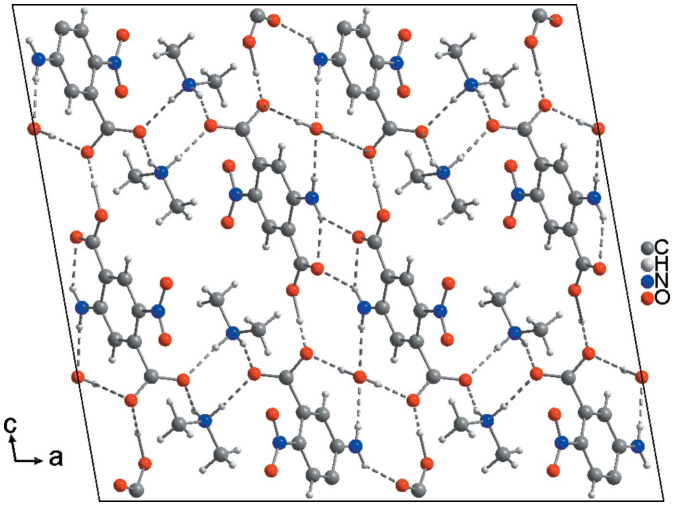


Figure 2
Crystal packing of the title compound viewed along the crystallographic *b* axis. Hydrogen bonds are shown as dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H3*B*···O1	0.89	1.87	2.761 (4)	175
N1—H3*A*···O1^{i}	0.89	2.01	2.880 (4)	164
N3—H6*B*···O4	0.86	2.11	2.736 (4)	130
N3—H6*B*···O4^{ii}	0.86	2.32	3.035 (4)	141
N3—H6*A*···O7^{iii}	0.86	2.10	2.959 (3)	173
O3—H9···O2^{iv}	0.82	1.80	2.614 (3)	169
O7—H7···O2^{v}	0.89 (4)	1.85 (4)	2.743 (3)	176 (4)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$2C_2H_8N^+\cdot 2C_8H_5N_2O_6^-\cdot H_2O$
M_r	560.48
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	293
a, *b*, *c* (Å)	21.494 (4), 6.4300 (13), 19.193 (4)
β (°)	99.98 (3)
V (Å^3)	2612.5 (9)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.12
Crystal size (mm)	0.21 × 0.12 × 0.05
Data collection	
Diffractometer	Stoe *IPDS1* diffractometer
Absorption correction	Numerical (*X-SHAPE* and *X-RED*; Stoe, 2008)
T_{min}, T_{max}	0.96, 0.98
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8870, 2305, 1218
R_{int}	0.087
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.051, 0.139, 1.00
No. of reflections	2305
No. of parameters	191
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.17, −0.23

Computer programs: *X-AREA* (Stoe, 2008), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg, 1999), *publCIF* (Westrip, 2010).

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x160048 [doi:10.1107/S2414314616000481]

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Crystal data

$2C_2H_8N^+·2C_8H_5N_2O_6^-·H_2O$
M_r = 560.48
Monoclinic, *C*2/*c*
a = 21.494 (4) Å
b = 6.4300 (13) Å
c = 19.193 (4) Å
β = 99.98 (3)°
V = 2612.5 (9) Å^3
Z = 4
F(000) = 1176
D_x = 1.425 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 9323 reflections
θ = 2.1–25°
μ = 0.12 mm^{-1}
T = 293 K
Needle, brown
0.21 × 0.12 × 0.05 mm

Data collection

Stoe IPDS-1 diffractometer
Radiation source: fine-focus sealed tube
Phi scan
Absorption correction: numerical (*X-SHAPE* and *X-RED*; Stoe, 2008)
T_{min} = 0.96, T_{max} = 0.98
8870 measured reflections
2305 independent reflections
1218 reflections with $I > 2\sigma(I)$
R_{int} = 0.087
θ_{max} = 25.0°, θ_{min} = 2.2°
h = −25→25
k = −7→7
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.139
S = 1.00
2305 reflections
191 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.060P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1647 (2)	0.5874 (7)	0.6545 (3)	0.0857 (13)
H1A	0.1554	0.6218	0.7003	0.103*
H1B	0.1810	0.7079	0.6343	0.103*
H1C	0.1268	0.5418	0.6243	0.103*
C2	0.2293 (2)	0.3567 (7)	0.5941 (2)	0.0754 (12)
H2A	0.2602	0.2476	0.6026	0.091*
H2B	0.1926	0.3071	0.5628	0.091*
H2C	0.2468	0.4732	0.5728	0.091*
N1	0.21164 (14)	0.4215 (5)	0.66167 (15)	0.0564 (7)
H3A	0.1965	0.3117	0.6815	0.091 (14)*
H3B	0.2462	0.4640	0.6906	0.085 (14)*
C3	0.38888 (14)	0.5454 (5)	0.67852 (15)	0.0394 (7)
C4	0.37102 (14)	0.6636 (5)	0.61645 (15)	0.0432 (7)
C5	0.38628 (15)	0.5967 (5)	0.55256 (15)	0.0482 (8)
H5	0.3741	0.6770	0.5122	0.064 (10)*
C6	0.41882 (14)	0.4154 (5)	0.54760 (15)	0.0432 (7)
C7	0.43852 (14)	0.2946 (5)	0.61005 (15)	0.0438 (8)
C8	0.42165 (15)	0.3674 (5)	0.67438 (15)	0.0452 (8)
H8	0.4337	0.2893	0.7153	0.057 (10)*
C9	0.36954 (16)	0.5969 (5)	0.74900 (15)	0.0439 (7)
C10	0.43132 (15)	0.3464 (6)	0.47806 (16)	0.0498 (8)
N2	0.33726 (14)	0.8558 (5)	0.61761 (15)	0.0572 (8)
N3	0.47084 (14)	0.1170 (5)	0.61051 (16)	0.0613 (8)
H6A	0.4812	0.0484	0.6493	0.077 (13)*
H6B	0.4813	0.0712	0.5721	0.076 (13)*
O1	0.31397 (11)	0.5663 (4)	0.75635 (12)	0.0555 (6)
O2	0.41281 (11)	0.6591 (4)	0.79741 (11)	0.0609 (7)
O3	0.40789 (14)	0.4728 (4)	0.42515 (13)	0.0717 (8)
H9	0.4136	0.4220	0.3876	0.17 (3)*
O4	0.45861 (13)	0.1878 (5)	0.46819 (12)	0.0748 (8)
O5	0.33347 (14)	0.9371 (4)	0.67470 (13)	0.0740 (8)
O6	0.31376 (18)	0.9364 (5)	0.56135 (15)	0.1048 (13)
O7	0.5000	0.9096 (6)	0.7500	0.0556 (9)
H7	0.529 (2)	0.826 (7)	0.737 (2)	0.100 (16)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.089 (3)	0.071 (3)	0.095 (3)	0.013 (2)	0.009 (2)	−0.005 (2)
C2	0.076 (3)	0.085 (3)	0.066 (3)	−0.001 (2)	0.014 (2)	−0.010 (2)
N1	0.0667 (19)	0.0524 (19)	0.0493 (16)	−0.0100 (15)	0.0074 (15)	0.0073 (14)
C3	0.0457 (17)	0.0415 (18)	0.0313 (15)	−0.0016 (15)	0.0076 (13)	−0.0029 (12)
C4	0.0538 (18)	0.0402 (19)	0.0362 (16)	0.0059 (15)	0.0092 (13)	0.0002 (13)
C5	0.063 (2)	0.048 (2)	0.0341 (17)	0.0029 (16)	0.0104 (14)	0.0042 (14)
C6	0.0530 (18)	0.044 (2)	0.0338 (16)	0.0026 (15)	0.0096 (13)	−0.0021 (13)

C7	0.0474 (18)	0.044 (2)	0.0402 (18)	0.0049 (15)	0.0076 (14)	−0.0007 (13)
C8	0.0552 (19)	0.048 (2)	0.0331 (16)	0.0017 (16)	0.0096 (13)	0.0048 (14)
C9	0.057 (2)	0.043 (2)	0.0333 (16)	−0.0007 (15)	0.0129 (14)	0.0009 (13)
C10	0.058 (2)	0.054 (2)	0.0387 (17)	0.0031 (17)	0.0117 (15)	−0.0022 (16)
N2	0.075 (2)	0.0490 (19)	0.0479 (17)	0.0158 (15)	0.0123 (14)	0.0025 (14)
N3	0.082 (2)	0.058 (2)	0.0458 (17)	0.0266 (16)	0.0178 (14)	0.0051 (14)
O1	0.0532 (14)	0.0672 (17)	0.0488 (13)	−0.0041 (11)	0.0169 (10)	−0.0068 (11)
O2	0.0691 (16)	0.0805 (19)	0.0338 (11)	−0.0170 (13)	0.0105 (11)	−0.0075 (11)
O3	0.106 (2)	0.0723 (19)	0.0376 (13)	0.0244 (15)	0.0160 (14)	0.0045 (12)
O4	0.101 (2)	0.075 (2)	0.0511 (15)	0.0311 (17)	0.0185 (13)	−0.0058 (13)
O5	0.118 (2)	0.0561 (17)	0.0507 (16)	0.0237 (15)	0.0234 (14)	−0.0048 (12)
O6	0.165 (3)	0.096 (2)	0.0505 (16)	0.075 (2)	0.0096 (18)	0.0136 (16)
O7	0.059 (2)	0.056 (2)	0.052 (2)	0.000	0.0088 (16)	0.000

Geometric parameters (Å, º)

C1—N1	1.458 (5)	C5—H5	0.9300
C1—H1A	0.9600	C6—C7	1.430 (4)
C1—H1B	0.9600	C6—C10	1.475 (4)
C1—H1C	0.9600	C7—N3	1.336 (4)
C2—N1	1.474 (4)	C7—C8	1.425 (4)
C2—H2A	0.9600	C8—H8	0.9300
C2—H2B	0.9600	C9—O1	1.243 (4)
C2—H2C	0.9600	C9—O2	1.261 (4)
N1—H3A	0.8900	C10—O4	1.208 (4)
N1—H3B	0.8900	C10—O3	1.330 (4)
C3—C8	1.354 (4)	N2—O6	1.225 (4)
C3—C4	1.409 (4)	N2—O5	1.229 (3)
C3—C9	1.519 (4)	N3—H6A	0.8600
C4—C5	1.391 (4)	N3—H6B	0.8600
C4—N2	1.435 (4)	O3—H9	0.8200
C5—C6	1.371 (4)	O7—H7	0.89 (4)
N1—C1—H1A	109.5	C6—C5—H5	119.2
N1—C1—H1B	109.5	C4—C5—H5	119.2
H1A—C1—H1B	109.5	C5—C6—C7	119.0 (3)
N1—C1—H1C	109.5	C5—C6—C10	119.6 (3)
H1A—C1—H1C	109.5	C7—C6—C10	121.4 (3)
H1B—C1—H1C	109.5	N3—C7—C8	119.0 (3)
N1—C2—H2A	109.5	N3—C7—C6	123.3 (3)
N1—C2—H2B	109.5	C8—C7—C6	117.7 (3)
H2A—C2—H2B	109.5	C3—C8—C7	122.8 (3)
N1—C2—H2C	109.5	C3—C8—H8	118.6
H2A—C2—H2C	109.5	C7—C8—H8	118.6
H2B—C2—H2C	109.5	O1—C9—O2	124.6 (3)
C1—N1—C2	113.8 (3)	O1—C9—C3	118.9 (3)
C1—N1—H3A	108.8	O2—C9—C3	116.4 (3)
C2—N1—H3A	108.8	O4—C10—O3	121.8 (3)

C1—N1—H3B	108.8	O4—C10—C6	124.5 (3)
C2—N1—H3B	108.8	O3—C10—C6	113.7 (3)
H3A—N1—H3B	107.7	O6—N2—O5	121.7 (3)
C8—C3—C4	118.3 (3)	O6—N2—C4	118.9 (3)
C8—C3—C9	117.5 (3)	O5—N2—C4	119.5 (3)
C4—C3—C9	124.0 (3)	C7—N3—H6A	120.0
C5—C4—C3	120.5 (3)	C7—N3—H6B	120.0
C5—C4—N2	118.5 (3)	H6A—N3—H6B	120.0
C3—C4—N2	121.0 (3)	C10—O3—H9	109.5
C6—C5—C4	121.7 (3)		

Hydrogen-bond geometry (Å, º)

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