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Bis(melaminium) terephthalate dihydrate

Jian Zhang, Yao Kang, Yi-Hang Wen, Zhao-Ji Li, Ye-Yan Qin and Yuan-Gen Yao*

The State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, The Chinese Academy of Sciences, Fuzhou, Fujian 350002, People's Republic of China

Correspondence e-mail: yyg@ms.fjirsm.ac.cn

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Key indicators

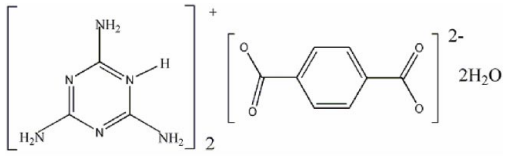
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.067
wR factor = 0.174
Data-to-parameter ratio = 11.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The crystal structure of the title melaminium salt, bis(2,4,6-triamino-1,3,5-triazin-1-ium) terephthalate dihydrate, $2C_3H_7N_6^+\cdot C_8H_4O_4^{2-}\cdot 2H_2O$, is composed of monoprotonated melaminium cations, terephthalate dianions and water molecules. The protonated melaminium cation and the terephthalate anion are almost coplanar, the latter possessing a twofold axis of symmetry. The crystal structure involves extensive N—H···N, N—H···O and O—H···O hydrogen bonding.

Comment

We present here the crystal structure of monoprotonated bis(melaminium) terephthalate dihydrate, (I). The cation has a structure very similar to that reported for related compounds. These include: $C_3H_7N_6^+\cdot CH_3COO^-\cdot CH_3COOH\cdot H_2O$ (Perpétuo & Janczak, 2002), $6C_3H_7N_6^+\cdot HPO_4^{2-}\cdot 4H_2PO_4^-\cdot 4H_2O$ (Janczak & Perpétuo, 2002), $C_3H_7N_6^+\cdot C_8H_5O_4^-$ (Janczak & Perpétuo, 2001*a*), $2C_3H_7N_6^+.SO_4^{2-}\cdot 2H_2O$ (Janczak & Perpétuo, 2001*d*), $C_3H_7N_6^+\cdot N_3O_4^-$ (Tanbug *et al.*, 1999), $C_3H_7N_6^+\cdot NO_3^-$ (Tanbug *et al.*, 1999), $C_3H_7N_6^+\cdot C_4H_3N_2O_3^-$ (Zerkowski *et al.*, 1994), $C_3H_7N_6^+\cdot Cl^-\cdot 0.5H_2O$ (Janczak & Perpétuo, 2001*c*). In addition to these monoprotonated melaminium salts, the diprotonated cation salt has also been structurally characterized in the following compounds: $C_3H_8N_6^{2+}\cdot 2C_6H_5O_4S^-\cdot 2H_2O$ (Janczak & Perpétuo, 2001*b*) and $C_3H_8N_6^{2+}\cdot 2ClO_4^-\cdot H_2O$ (Martin & Pinkerton, 1995). A search of the Cambridge Structural Database (Version 5.25; Allen, 2002) confirmed that the geometry of the terephthalate anion is normal. A twofold symmetry axis passes through the centre of the anion, bisecting the C5—C8^{v} and C8—C5^{v} bonds [symmetry code: (v) $1-x, 2-y, 1-z$].



(I)

There is extensive hydrogen bonding in the structure (Fig. 2). The melaminium residues in (I) are involved in six hydrogen bonds with the anions, water and neighbouring cations. The water molecule forms two hydrogen bonds with the terephthalate (Table 1).

Experimental

A mixture of melamine (0.126 g, 1 mmol), terephthalic acid (0.166 g, 1 mmol) and water (10 ml) was sealed in a 15 ml Teflon-lined stainless steel reactor and heated to 433 K for 60 h. Colorless crystals of the title compound suitable for X-ray analysis were obtained.

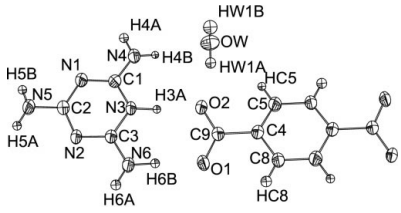


Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme of the asymmetric unit.

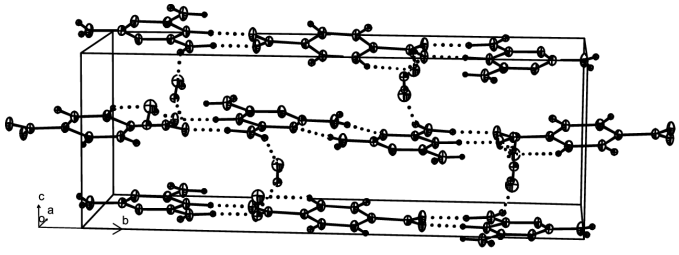


Figure 2
The packing arrangement in the unit cell, showing the hydrogen-bonding interactions as dashed lines.

Crystal data

$2C_3H_7N_6^+\cdot C_8H_4O_4^{2-}\cdot 2H_2O$
M_r = 454.44
Monoclinic, $P2_1/c$
a = 6.809 (4) Å
b = 20.164 (10) Å
c = 7.062 (6) Å
β = 93.33 (5)°
V = 967.9 (11) Å^3
Z = 2
D_x = 1.559 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1132 reflections
θ = 2.0–25.1°
μ = 0.13 mm^{-1}
T = 293 (2) K
Prism, colorless
0.20 × 0.16 × 0.15 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.975, T_{max} = 0.981
3095 measured reflections
1691 independent reflections
1215 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 25.1°
$h = -8 \rightarrow 7$
$k = -22 \rightarrow 23$
$l = -3 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.174
S = 1.08
1691 reflections
154 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0718P)^2 + 1.0433P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.170
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.009 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3*A*···O2	0.86	1.86	2.717 (4)	179
N6—H6*A*···O*W*^{i}	0.86	2.18	2.959 (4)	151
N6—H6*B*···O1	0.86	1.95	2.811 (4)	180
N5—H5*A*···N2^{ii}	0.86	2.20	3.055 (4)	175
N4—H4*A*···O1^{iii}	0.86	2.07	2.889 (4)	159
N4—H4*B*···O*W*	0.86	2.22	3.056 (5)	163
O*W*—H*W*1*B*···O2	0.87 (5)	1.88 (5)	2.738 (4)	168 (4)
O*W*—H*W*1*A*···O1^{iv}	0.84 (6)	2.13 (6)	2.889 (5)	151 (6)

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

H atoms were located in a difference Fourier synthesis. The H atoms of the water molecule were refined freely. Other H atoms were refined in the riding-model approximation, with C—H = 0.93 Å and N—H = 0.86 Å; U_{iso}(H) = 1.2U_{eq} of the carrier atom.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART* and *SAINT* (Siemens, 1994); data reduction: *SAINT* and *XPREP* in *SHELXTL* (Siemens, 1994); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Janczak, J. & Perpétuo, G. J. (2001*a*). *Acta Cryst.* C**57**, 123–125.
Janczak, J. & Perpétuo, G. J. (2001*b*). *Acta Cryst.* C**57**, 873–875.
Janczak, J. & Perpétuo, G. J. (2001*c*). *Acta Cryst.* C**57**, 1120–1122.
Janczak, J. & Perpétuo, G. J. (2001*d*). *Acta Cryst.* C**57**, 1431–1433.
Janczak, J. & Perpétuo, G. J. (2002). *Acta Cryst.* C**58**, o455–o459.
Martin, A. & Pinkerton, A. A. (1995). *Acta Cryst.* C**51**, 2174–2177.
Perpétuo, G. J. & Janczak, J. (2002). *Acta Cryst.* C**58**, o112–o114.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Siemens (1994). *SAINT* and *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Tanbug, R., Kirschbaum, K. & Pinkerton, A. A. (1999). *J. Chem. Crystallogr.* **29**, 45–55.
Zerkowski, J. A., MacDonald, J. C. & Whitesides, G. M. (1994). *Chem. Mater.* **6**, 1250–1257.