

Melaminium iodide monohydrate

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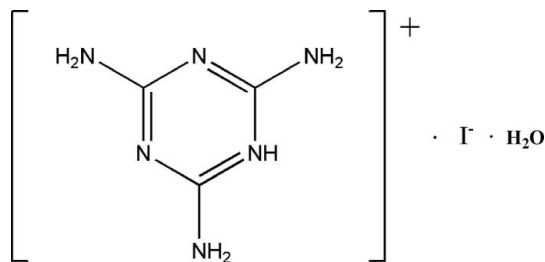
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{N}-\text{C}) = 0.004$ Å; R factor = 0.026; wR factor = 0.067; data-to-parameter ratio = 18.9.

In the title melaminium salt, 2,4,6-triamino-1,3,5-triazin-1-ium iodide monohydrate, $\text{C}_3\text{H}_7\text{N}_6^+\text{I}^-\cdot\text{H}_2\text{O}$, the components are linked *via* $\text{N}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{N}$, $\text{O}-\text{H}\cdots\text{I}$ and $\text{N}-\text{H}\cdots\text{I}$ hydrogen bonds. All of the H atoms of the melaminium cation are involved in hydrogen bonds. The melaminium cations are interconnected by four $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds, forming ribbons along [111]. The water molecules connected by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds also form part of these ribbons. The ribbons are interconnected by other hydrogen bonds ($\text{O}-\text{H}\cdots\text{I}$ and $\text{N}-\text{H}\cdots\text{I}$), as well as by $\pi-\pi$ interactions [centroid-centroid distance = 3.6597 (17) Å].

Related literature

For similar singly protonated melaminium salts, see: Janczak *et al.* (2001); Athikomrattanukul *et al.* (2007). For ferroelectric materials, see: Fu *et al.* (2009); Hang *et al.* (2009). For impedance studies, see: Uthrakumar *et al.* (2008).



Experimental

Crystal data

 $\text{C}_3\text{H}_7\text{N}_6^+\text{I}^-\cdot\text{H}_2\text{O}$ $M_r = 272.06$ Triclinic, $P\bar{1}$ $a = 6.0655$ (12) Å $b = 7.0370$ (14) Å $c = 11.413$ (2) Å $\alpha = 104.02$ (3)° $\beta = 93.95$ (3)° $\gamma = 109.08$ (3)° $V = 440.80$ (19) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 3.59$ mm⁻¹
 $T = 293$ K

0.40 × 0.30 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan
(*CrystalClear*; Rigaku, 2005)
 $T_{\text{min}} = 0.285$, $T_{\text{max}} = 0.487$

4551 measured reflections
2006 independent reflections
1896 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.067$
 $S = 1.11$
2006 reflections
106 parameters
3 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.47$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.61$ e Å⁻³

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2A}\cdots\text{O1}$	0.86	1.87	2.724 (4)	172
$\text{N4}-\text{H4A}\cdots\text{I1}^{\text{i}}$	0.86	2.95	3.764 (3)	159
$\text{N4}-\text{H4B}\cdots\text{I1}^{\text{ii}}$	0.86	3.20	3.758 (3)	125
$\text{N6}-\text{H6A}\cdots\text{I1}^{\text{iii}}$	0.86	2.88	3.647 (3)	149
$\text{N6}-\text{H6B}\cdots\text{N1}^{\text{iv}}$	0.86	2.15	3.009 (3)	173
$\text{O1}-\text{H1A}\cdots\text{I1}^{\text{v}}$	0.83 (2)	3.13 (4)	3.760 (4)	134 (5)
$\text{O1}-\text{H1A}\cdots\text{I1}^{\text{vi}}$	0.83 (2)	3.39 (5)	3.778 (3)	112 (4)
$\text{O1}-\text{H1B}\cdots\text{I1}$	0.82 (2)	3.00 (3)	3.732 (4)	150 (5)
$\text{N5}-\text{H5A}\cdots\text{N3}^{\text{vii}}$	0.86	2.15	3.013 (4)	177
$\text{N5}-\text{H5B}\cdots\text{I1}^{\text{v}}$	0.86	2.97	3.698 (3)	143

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

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).

The authors are grateful to the starter fund of Southeast University for financial support to purchase the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FB2196).

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supplementary materials

Acta Cryst. (2010). E66, o1415 [doi:10.1107/S160053681001785X]

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Comment

The melamine molecule and its organic and inorganic complexes or salts can develop supramolecular structures *via* multiple hydrogen-bonding systems by self-assembly of components which contain abundant hydrogen-bonding sites (Janczak *et al.*, 2001; Athikomrattanakul *et al.*, 2007). The present study is a part of systematic investigation of ferroelectric materials (Fu *et al.*, 2009; Hang *et al.*, 2009) that include metal-organic coordination compounds with organic ligands or are related to the structures with both organic and inorganic building fragments.

The compound was characterized by the X-ray powder diffraction (XRPD) at room temperature. The pattern calculated from the single-crystal X-ray data was in a good agreement with the observed at to the peak positions but with different peak intensities.

The structure is composed of the melaminium cations, iodide anions and the water molecules (Fig. 1). The melaminium cation is protonated at only one melamine site. The six-membered melaminium ring exhibits distortions from the regular hexagonal form. The internal C—N—C angle at the protonated N atom (119.5 (2)°) is greater than the other two C—N—C angles of the ring (115.5 (2)°) and the internal N—C—N angles involving the unprotonated ring N atoms (126.1 (2)°) are obviously larger than those containing protonated and unprotonated N atoms (121.4 (2)°).

Fig. 2 shows a view down the *b* axis. The melaminium cations are interconnected by four N—H...N hydrogen bonds, forming ribbons parallel to (1 1 1). The water molecules connected by N—H...O hydrogen bonds (Tab. 1) form also a part of these ribbons. The ribbons are interconnected by other hydrogen bonds that involve I⁻ as well as by π -electron ring - π -electron ring interactions with the distance between the centroids of the neighbour melaminium rings (1-*x*, 1-*y*, 1-*z*) equal to 3.6597 (17) Å. The hydrogen bonds are summarized in Tab. 1. It is of interest that water oxygens despite of being quite close to each other are not interconnected by the hydrogen bond. The distance between the water oxygens is 2.9643 (40) Å [Symmetry code: 1-*x*, 1-*y*, -*z*]. The H atom of the protonated ring N atom (H2a) is donated to the water molecule, being involved in a strong N—H...O hydrogen bond. The other amine H atoms are involved in N—H...I and N—H...N hydrogen bonds. I⁻ anions that take part in the electrostatic equilibrium with the melaminium cations are also involved in O—H...I hydrogen bonds.

Experimental

Melamine (0.252 g, 0.002 mol) was dissolved in 25 ml of water at 323 K. 0.569 g of 45% (weight concentration) solution of HI was added to the solution. (This amount corresponded to about 0.002 mol of pure HI.) The temperature was maintained at 323 K for one hour while stirring the mixture. Then the solution was let to cool down to room temperature. After several days, the title salt, C₃H₇N₆⁺.I⁻.H₂O, crystallized from the solution. The crystals were colourless, prismatic and of the average size about 0.2×0.3×0.4 mm.

Refinement

All the hydrogens were discernible in the difference electron density maps. The positions of the H atoms of the melamine cations were refined using a riding model with $N-H = 0.86 \text{ \AA}$ and $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(N)$. The coordinates of the water hydrogens have been refined under restraints $0.82 (2)\text{\AA}$; $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(O)$. (The constrained and the restrained values fit well to the trial refinement with the freely refined hydrogen parameters.)

Dielectric studies (capacitance and dielectric loss measurements) were performed on powder samples which have been pressed into tablets with conducting carbon glue deposited on their faces. The automatic impedance TongHui2828 Analyzer has been used (Uthrakumar *et al.*, 2008). In the measured temperature range from 80 to 450 K (m.p. > 470 K), the temperature dependence of the relative permittivity at 1 MHz varied smoothly from 3.9 to 5.2 in the title compound. No dielectric anomaly has been observed.

Figures

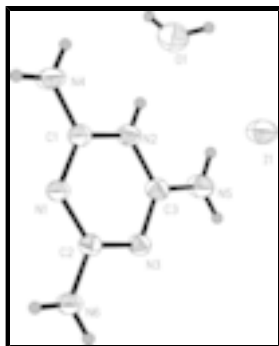


Fig. 1. The title molecules with the atomic numbering scheme. The displacement ellipsoids are drawn at the 30% probability level.

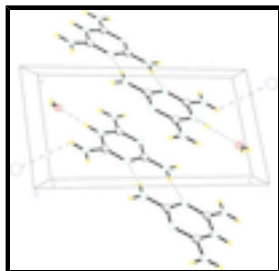


Fig. 2. A view of the structure along the *b* axis. The dashed lines depict the hydrogen bonds.

2,4,6-triamino-1,3,5-triazin-1-ium iodide monohydrate

Crystal data

$C_3H_7N_6^+ \cdot I^- \cdot H_2O$

$M_r = 272.06$

Triclinic, $P\bar{1}$

Hall symbol: $-P 1$

$a = 6.0655 (12) \text{ \AA}$

$b = 7.0370 (14) \text{ \AA}$

$c = 11.413 (2) \text{ \AA}$

$Z = 2$

$F(000) = 260$

$D_x = 2.050 \text{ Mg m}^{-3}$

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

Cell parameters from 4510 reflections

$\theta = 3.2\text{--}27.5^\circ$

$\mu = 3.59 \text{ mm}^{-1}$

$\alpha = 104.02 (3)^\circ$
 $\beta = 93.95 (3)^\circ$
 $\gamma = 109.08 (3)^\circ$
 $V = 440.80 (19) \text{ \AA}^3$

$T = 293 \text{ K}$
 Prism, colourless
 $0.40 \times 0.30 \times 0.20 \text{ mm}$

Data collection

Rigaku SCXmini diffractometer
 Radiation source: fine-focus sealed tube
 graphite
 Detector resolution: $13.6612 \text{ pixels mm}^{-1}$
 ω scans
 Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.285$, $T_{\max} = 0.487$
 4551 measured reflections

2006 independent reflections
 1896 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$
 $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.2^\circ$
 $h = -7 \rightarrow 7$
 $k = -9 \rightarrow 9$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.067$
 $S = 1.11$
 2006 reflections
 106 parameters
 3 restraints
 30 constraints

Primary atom site location: structure-invariant direct methods
 Secondary atom site location: difference Fourier map
 Hydrogen site location: difference Fourier map
 H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0285P)^2 + 0.1083P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.008$
 $\Delta\rho_{\max} = 0.47 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.60 \text{ e \AA}^{-3}$

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	$U_{\text{iso}}^*/U_{\text{eq}}$
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supplementary materials

N1	0.1770 (4)	0.3970 (4)	0.3846 (2)	0.0392 (5)
N2	0.4296 (4)	0.2565 (4)	0.2773 (2)	0.0433 (5)
H2A	0.5039	0.2463	0.2159	0.052*
N3	0.3428 (4)	0.1720 (4)	0.4600 (2)	0.0402 (5)
N4	0.2758 (6)	0.4725 (5)	0.2059 (3)	0.0567 (7)
H4A	0.1912	0.5504	0.2121	0.068*
H4B	0.3504	0.4576	0.1449	0.068*
N6	0.1007 (5)	0.3160 (4)	0.5631 (2)	0.0510 (6)
H6A	0.1161	0.2533	0.6175	0.061*
H6B	0.0144	0.3925	0.5713	0.061*
O1	0.6721 (6)	0.1912 (5)	0.0884 (3)	0.0817 (9)
H1A	0.707 (10)	0.091 (5)	0.051 (5)	0.123*
H1B	0.731 (10)	0.301 (5)	0.070 (5)	0.123*
C1	0.2911 (5)	0.3752 (4)	0.2904 (3)	0.0401 (6)
C2	0.2097 (5)	0.2947 (4)	0.4671 (2)	0.0375 (5)
C3	0.4499 (5)	0.1533 (4)	0.3622 (3)	0.0414 (6)
I1	0.89281 (4)	0.76920 (3)	0.134131 (16)	0.05182 (10)
N5	0.5833 (5)	0.0365 (5)	0.3468 (3)	0.0562 (7)
H5A	0.6010	-0.0276	0.4000	0.067*
H5B	0.6525	0.0246	0.2835	0.067*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0500 (13)	0.0449 (12)	0.0330 (11)	0.0251 (10)	0.0130 (10)	0.0164 (10)
N2	0.0505 (13)	0.0533 (13)	0.0369 (12)	0.0275 (11)	0.0152 (10)	0.0170 (11)
N3	0.0489 (12)	0.0450 (12)	0.0339 (12)	0.0243 (10)	0.0076 (10)	0.0133 (10)
N4	0.0751 (18)	0.0785 (19)	0.0448 (15)	0.0473 (16)	0.0294 (14)	0.0350 (14)
N6	0.0732 (17)	0.0630 (16)	0.0401 (13)	0.0427 (14)	0.0239 (12)	0.0264 (12)
O1	0.093 (2)	0.086 (2)	0.080 (2)	0.0379 (18)	0.0513 (17)	0.0311 (18)
C1	0.0445 (14)	0.0427 (14)	0.0369 (14)	0.0182 (12)	0.0088 (11)	0.0136 (11)
C2	0.0415 (13)	0.0382 (13)	0.0330 (13)	0.0148 (11)	0.0054 (11)	0.0095 (11)
C3	0.0424 (14)	0.0453 (14)	0.0375 (14)	0.0186 (12)	0.0035 (11)	0.0099 (12)
I1	0.06273 (16)	0.06633 (16)	0.03972 (14)	0.03312 (12)	0.01791 (10)	0.02193 (11)
N5	0.0674 (17)	0.0751 (18)	0.0485 (16)	0.0474 (15)	0.0192 (13)	0.0240 (14)

Geometric parameters (\AA , $^\circ$)

N1—C1	1.322 (3)	N4—H4B	0.8600
N1—C2	1.357 (3)	N6—C2	1.321 (4)
N2—C1	1.357 (3)	N6—H6A	0.8600
N2—C3	1.366 (4)	N6—H6B	0.8600
N2—H2A	0.8600	O1—H1A	0.831 (18)
N3—C3	1.330 (4)	O1—H1B	0.822 (18)
N3—C2	1.354 (3)	C3—N5	1.321 (4)
N4—C1	1.325 (4)	N5—H5A	0.8600
N4—H4A	0.8600	N5—H5B	0.8600
C1—N1—C2	115.6 (2)	N1—C1—N4	120.5 (3)

C1—N2—C3	119.5 (2)	N1—C1—N2	121.9 (2)
C1—N2—H2A	120.3	N4—C1—N2	117.6 (3)
C3—N2—H2A	120.3	N6—C2—N3	117.0 (2)
C3—N3—C2	115.5 (2)	N6—C2—N1	117.0 (2)
C1—N4—H4A	120.0	N3—C2—N1	126.1 (2)
C1—N4—H4B	120.0	N5—C3—N3	120.1 (3)
H4A—N4—H4B	120.0	N5—C3—N2	118.5 (3)
C2—N6—H6A	120.0	N3—C3—N2	121.4 (2)
C2—N6—H6B	120.0	C3—N5—H5A	120.0
H6A—N6—H6B	120.0	C3—N5—H5B	120.0
H1A—O1—H1B	115 (3)	H5A—N5—H5B	120.0

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2A \cdots O1	0.86	1.87	2.724 (4)	172
N4—H4A \cdots I1 ⁱ	0.86	2.95	3.764 (3)	159
N4—H4B \cdots I1 ⁱⁱ	0.86	3.20	3.758 (3)	125
N6—H6A \cdots I1 ⁱⁱⁱ	0.86	2.88	3.647 (3)	149
N6—H6B \cdots N1 ^{iv}	0.86	2.15	3.009 (3)	173
O1—H1A \cdots I1 ^v	0.83 (2)	3.13 (4)	3.760 (4)	134 (5)
O1—H1A \cdots I1 ^{vi}	0.83 (2)	3.39 (5)	3.778 (3)	112 (4)
O1—H1B \cdots I1	0.82 (2)	3.00 (3)	3.732 (4)	150 (5)
N5—H5A \cdots N3 ^{vii}	0.86	2.15	3.013 (4)	177
N5—H5B \cdots I1 ^v	0.86	2.97	3.698 (3)	143

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Fig. 1

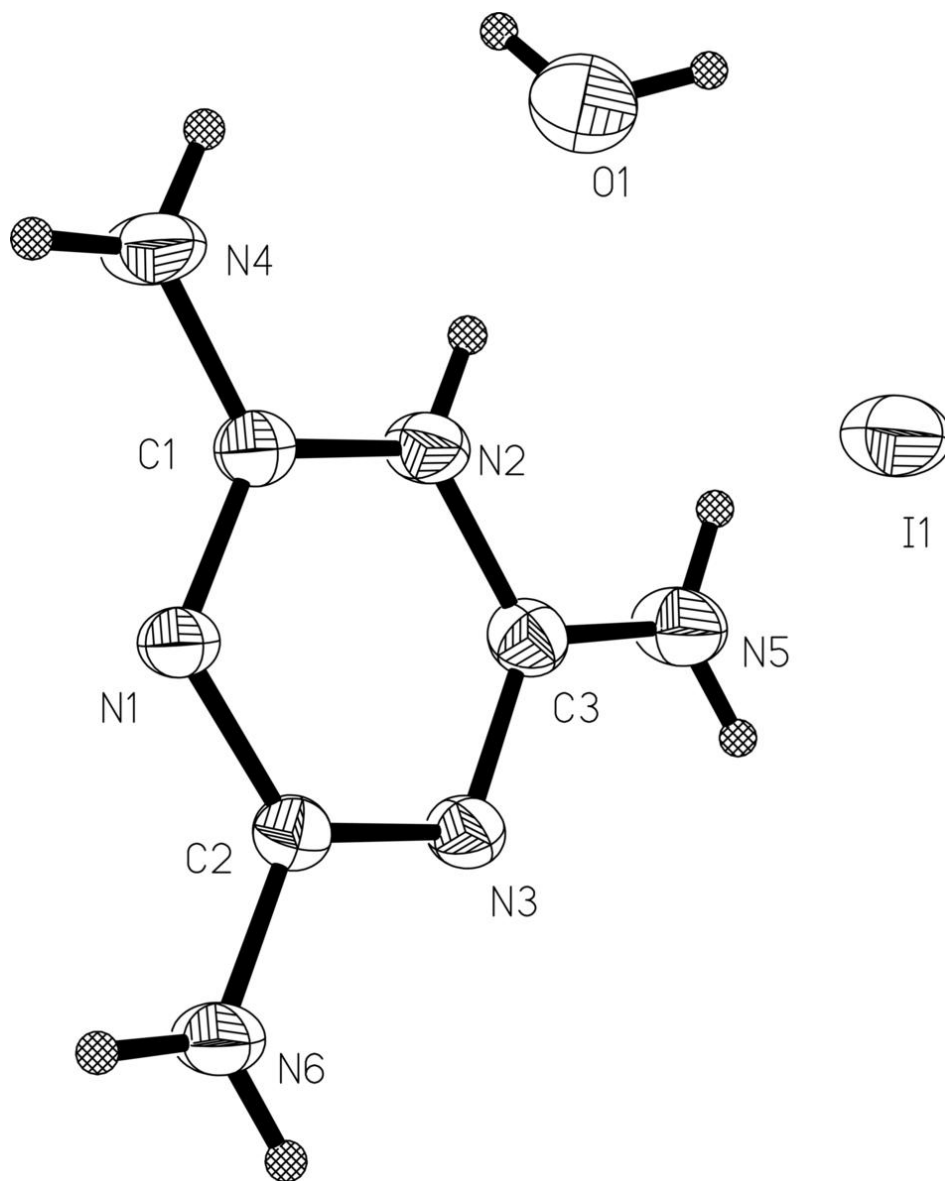


Fig. 2

