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2,4-Diamino-6-methyl-1,3,5-triazine ethanol solvate

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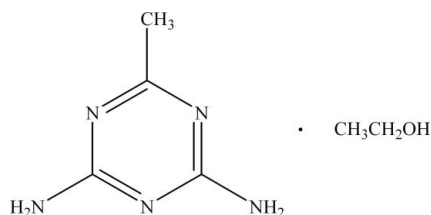
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Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.043; wR factor = 0.119; data-to-parameter ratio = 14.0.

The crystal structure of the title compound, $\text{C}_4\text{H}_7\text{N}_5 \cdot \text{C}_2\text{H}_6\text{O}$, is determined by extensive hydrogen bonding. A sequence of dimeric associations, formed by $\text{N}-\text{H}(\text{amino}) \cdots \text{N}(\text{ring})$, connects the triazine rings into a molecular tape. Molecules are linked into a supramolecular structure by $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds. The asymmetric unit consists of two formula units.

Related literature

For general background, see: Sebenik *et al.* (1989); Tashiro & Oiwa (1981).



Experimental

Crystal data

 $\text{C}_4\text{H}_7\text{N}_5 \cdot \text{C}_2\text{H}_6\text{O}$ $M_r = 171.21$ Triclinic, $P\bar{1}$ $a = 8.3860$ (6) Å $b = 9.1514$ (6) Å $c = 11.9104$ (9) Å $\alpha = 88.703$ (1)° $\beta = 87.614$ (2)° $\gamma = 76.668$ (2)° $V = 888.56$ (11) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.09$ mm⁻¹ $T = 273$ (2) K $0.34 \times 0.26 \times 0.21$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2005)

 $T_{\min} = 0.975$, $T_{\max} = 0.985$

7627 measured reflections

3111 independent reflections

2619 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.017$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.042$ $wR(F^2) = 0.118$ $S = 1.07$

3111 reflections

223 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N4}-\text{H4B} \cdots \text{N7}^i$	0.86	2.11	2.9666 (18)	171
$\text{N5}-\text{H5D} \cdots \text{N8}^{\text{ii}}$	0.86	2.19	3.0132 (19)	159
$\text{N5}-\text{H5E} \cdots \text{O2}^{\text{iii}}$	0.86	2.29	3.0071 (19)	142
$\text{N10}-\text{H10A} \cdots \text{O2}^{\text{iv}}$	0.86	2.10	2.9337 (18)	163
$\text{O2}-\text{H2} \cdots \text{O1}^{\text{v}}$	0.82	1.90	2.7185 (18)	174

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2061).

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supplementary materials

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2,4-Diamino-6-methyl-1,3,5-triazine ethanol solvate

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Comment

Triazine compounds are used in pharmaceutical industry as coupling agents for the synthesis of peptides and as side chain of antibiotics, as well as in formulating bactericides and fungicides. 2,4-Diamino-6-methyl-1,3,5-triazine (acetoguanamine) is used as an intermediate for pharmaceuticals and as a modifier and flexibilizer of formaldehyde resins (Sebenik *et al.*, 1989, Tashiro *et al.*, 1981).

The crystal structure of the title compound (Fig. 1) consists of triazine and solvate ethanol molecule. The amino groups are coplanar with the ring plane, the dihedral angle between the triazine ring (C2,N2,C4,N1,C3,N3) and the ring (C8,N7,C9,N8,C10,N6) is 12.73 (7)°. A lot of hydrogen bonds are observed (Table 1), each NH₂ group acts as a donor in hydrogen bond with the ring nitrogen atoms of neighboring molecules, these contacts and the cross-linking interactions stabilize the crystal packing.

Experimental

2,4-diamino-6-methyl-1,3,5-triazine (0.625 g, 0.05 mol) was added to a stirred solvent of ethanol (100 ml) at 50°C for 3 h. After cooling to room temperature, the mixture was filtered. The filtrate was set aside for one week to obtain colorless crystals.

Refinement

Water H atoms were located in a difference Fourier map and refined as riding in their as-found positions relative to O atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$. All other H atoms were placed in calculated positions and refined as riding, with C—H = 0.93–0.97 Å, N—H = 0.86 Å, and $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{eq}}(\text{C},\text{N})$.

Figures

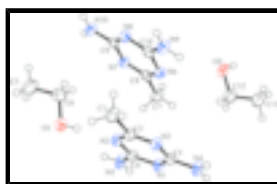


Fig. 1. The molecular structure of (I) showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

2,4-Diamino-6-methyl-1,3,5-triazine ethanol solvate

Crystal data

C₄H₇N₅·C₂H₆O

Z = 4

supplementary materials

$M_r = 171.21$	$F_{000} = 368.0$
Triclinic, $P\bar{1}$	$D_x = 1.280 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 8.3860 (6) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 9.1514 (6) \text{ \AA}$	Cell parameters from 3111 reflections
$c = 11.9104 (9) \text{ \AA}$	$\theta = 1.7\text{--}25.0^\circ$
$\alpha = 88.703 (1)^\circ$	$\mu = 0.09 \text{ mm}^{-1}$
$\beta = 87.614 (2)^\circ$	$T = 273 (2) \text{ K}$
$\gamma = 76.668 (2)^\circ$	Block, colorless
$V = 888.56 (11) \text{ \AA}^3$	$0.34 \times 0.26 \times 0.21 \text{ mm}$

Data collection

Bruker CCD area-detector diffractometer	3111 independent reflections
Radiation source: fine-focus sealed tube	2619 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.017$
$T = 273(2) \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.7^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.975$, $T_{\text{max}} = 0.985$	$k = -10 \rightarrow 10$
7627 measured reflections	$l = -12 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters constrained
$wR(F^2) = 0.118$	$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.2811P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
3111 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
223 parameters	$\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$
	Extinction correction: none

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.

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 $F^2 > 2\sigma(F^2)$ is used only for calculat-

ing 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0454 (2)	0.41963 (19)	0.15635 (15)	0.0388 (4)
H1A	-0.0653	0.4778	0.1527	0.058*
H1B	0.0892	0.3946	0.0817	0.058*
H1C	0.1110	0.4770	0.1920	0.058*
C2	0.04756 (19)	0.27874 (17)	0.22268 (13)	0.0290 (4)
C3	-0.07229 (18)	0.14537 (17)	0.34835 (13)	0.0258 (3)
C4	0.18337 (18)	0.04532 (17)	0.27722 (13)	0.0268 (3)
C5	0.6644 (3)	0.2978 (3)	0.0124 (2)	0.0651 (6)
H5A	0.7394	0.2012	0.0089	0.098*
H5B	0.7212	0.3716	0.0348	0.098*
H5C	0.6200	0.3244	-0.0602	0.098*
C6	0.5286 (2)	0.2917 (2)	0.09584 (17)	0.0486 (5)
H6A	0.5730	0.2628	0.1691	0.058*
H6B	0.4548	0.3900	0.1019	0.058*
C7	0.3737 (2)	0.18852 (18)	0.49621 (15)	0.0342 (4)
H7A	0.4172	0.1132	0.4412	0.051*
H7B	0.2683	0.1765	0.5241	0.051*
H7C	0.4469	0.1780	0.5572	0.051*
C8	0.35597 (18)	0.34072 (16)	0.44340 (12)	0.0254 (3)
C9	0.47170 (18)	0.50943 (16)	0.34763 (12)	0.0244 (3)
C10	0.20147 (18)	0.56977 (16)	0.39979 (12)	0.0255 (3)
N1	0.05944 (15)	0.02890 (14)	0.34824 (11)	0.0279 (3)
N2	0.18531 (16)	0.17023 (15)	0.21443 (11)	0.0299 (3)
N3	-0.08554 (15)	0.27267 (14)	0.28518 (11)	0.0284 (3)
N4	-0.20112 (16)	0.13534 (15)	0.41484 (11)	0.0323 (3)
H4A	-0.1984	0.0563	0.4559	0.039*
H4B	-0.2871	0.2079	0.4167	0.039*
N6	0.20797 (15)	0.43256 (14)	0.44771 (11)	0.0273 (3)
N7	0.49186 (15)	0.37128 (13)	0.39648 (10)	0.0268 (3)
N8	0.32886 (15)	0.61343 (14)	0.34805 (11)	0.0274 (3)
N9	0.05530 (15)	0.66659 (15)	0.40412 (12)	0.0336 (3)
H9A	0.0442	0.7546	0.3742	0.040*
H9B	-0.0276	0.6409	0.4368	0.040*
N10	0.60412 (15)	0.54140 (15)	0.29698 (11)	0.0309 (3)
H10A	0.5979	0.6276	0.2650	0.037*
H10B	0.6959	0.4759	0.2962	0.037*
O1	0.44204 (16)	0.18580 (16)	0.06094 (10)	0.0475 (4)
H1	0.3607	0.1897	0.1024	0.071*
C11	0.0989 (3)	0.1326 (3)	0.89654 (19)	0.0599 (6)
H11A	0.1324	0.1124	0.9725	0.090*
H11B	0.0321	0.0655	0.8769	0.090*
H11C	0.0370	0.2344	0.8900	0.090*

supplementary materials

C12	0.2460 (2)	0.1100 (2)	0.81971 (17)	0.0495 (5)
H12A	0.3036	0.0053	0.8237	0.059*
H12B	0.2103	0.1310	0.7434	0.059*
N5	0.31467 (16)	-0.06868 (15)	0.26948 (12)	0.0353 (3)
H5D	0.3180	-0.1490	0.3091	0.042*
H5E	0.3961	-0.0619	0.2249	0.042*
O2	0.35702 (16)	0.19983 (16)	0.84251 (11)	0.0461 (3)
H2	0.3769	0.1931	0.9095	0.069*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0392 (10)	0.0326 (9)	0.0423 (10)	-0.0051 (7)	0.0017 (8)	0.0095 (8)
C2	0.0299 (8)	0.0272 (8)	0.0294 (8)	-0.0059 (7)	-0.0023 (6)	0.0018 (6)
C3	0.0241 (8)	0.0242 (8)	0.0282 (8)	-0.0041 (6)	-0.0011 (6)	-0.0001 (6)
C4	0.0250 (8)	0.0267 (8)	0.0281 (8)	-0.0051 (6)	-0.0004 (6)	0.0010 (6)
C5	0.0531 (13)	0.0762 (16)	0.0747 (16)	-0.0344 (12)	0.0045 (11)	0.0046 (12)
C6	0.0485 (11)	0.0488 (11)	0.0516 (12)	-0.0174 (9)	0.0003 (9)	-0.0063 (9)
C7	0.0298 (9)	0.0250 (8)	0.0454 (10)	-0.0028 (7)	0.0032 (7)	0.0040 (7)
C8	0.0248 (8)	0.0233 (8)	0.0269 (8)	-0.0032 (6)	0.0004 (6)	-0.0019 (6)
C9	0.0251 (8)	0.0230 (7)	0.0242 (8)	-0.0037 (6)	0.0001 (6)	-0.0011 (6)
C10	0.0237 (8)	0.0244 (8)	0.0270 (8)	-0.0028 (6)	-0.0012 (6)	-0.0002 (6)
N1	0.0247 (7)	0.0251 (7)	0.0313 (7)	-0.0019 (5)	0.0038 (5)	0.0036 (5)
N2	0.0293 (7)	0.0285 (7)	0.0311 (7)	-0.0062 (6)	0.0035 (5)	0.0031 (6)
N3	0.0257 (7)	0.0258 (7)	0.0321 (7)	-0.0030 (5)	0.0003 (5)	0.0039 (5)
N4	0.0258 (7)	0.0250 (7)	0.0419 (8)	0.0007 (5)	0.0089 (6)	0.0058 (6)
N6	0.0237 (7)	0.0234 (7)	0.0334 (7)	-0.0032 (5)	0.0008 (5)	0.0007 (5)
N7	0.0241 (7)	0.0228 (7)	0.0312 (7)	-0.0013 (5)	0.0016 (5)	0.0015 (5)
N8	0.0251 (7)	0.0239 (7)	0.0309 (7)	-0.0016 (5)	0.0009 (5)	0.0026 (5)
N9	0.0223 (7)	0.0269 (7)	0.0478 (9)	0.0005 (5)	0.0038 (6)	0.0078 (6)
N10	0.0237 (7)	0.0253 (7)	0.0411 (8)	-0.0017 (5)	0.0039 (6)	0.0054 (6)
O1	0.0477 (8)	0.0614 (9)	0.0398 (7)	-0.0274 (7)	0.0112 (6)	-0.0083 (6)
C11	0.0513 (12)	0.0746 (15)	0.0606 (14)	-0.0303 (11)	0.0095 (10)	-0.0034 (11)
C12	0.0453 (11)	0.0601 (13)	0.0463 (11)	-0.0190 (10)	0.0023 (9)	-0.0044 (9)
N5	0.0260 (7)	0.0298 (7)	0.0455 (8)	0.0005 (6)	0.0105 (6)	0.0072 (6)
O2	0.0461 (8)	0.0581 (8)	0.0391 (7)	-0.0241 (6)	0.0010 (6)	0.0112 (6)

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

C1—C2	1.494 (2)	C8—N7	1.3327 (19)
C1—H1A	0.9600	C9—N10	1.3300 (19)
C1—H1B	0.9600	C9—N8	1.3471 (19)
C1—H1C	0.9600	C9—N7	1.3571 (19)
C2—N3	1.327 (2)	C10—N9	1.3364 (19)
C2—N2	1.340 (2)	C10—N8	1.3459 (19)
C3—N4	1.332 (2)	C10—N6	1.3580 (19)
C3—N1	1.3466 (19)	N4—H4A	0.8600
C3—N3	1.3576 (19)	N4—H4B	0.8600
C4—N5	1.332 (2)	N9—H9A	0.8600

C4—N1	1.346 (2)	N9—H9B	0.8600
C4—N2	1.355 (2)	N10—H10A	0.8600
C5—C6	1.490 (3)	N10—H10B	0.8600
C5—H5A	0.9600	O1—H1	0.8200
C5—H5B	0.9600	C11—C12	1.482 (3)
C5—H5C	0.9600	C11—H11A	0.9600
C6—O1	1.417 (2)	C11—H11B	0.9600
C6—H6A	0.9700	C11—H11C	0.9600
C6—H6B	0.9700	C12—O2	1.415 (2)
C7—C8	1.494 (2)	C12—H12A	0.9700
C7—H7A	0.9600	C12—H12B	0.9700
C7—H7B	0.9600	N5—H5D	0.8600
C7—H7C	0.9600	N5—H5E	0.8600
C8—N6	1.3285 (19)	O2—H2	0.8200
C2—C1—H1A	109.5	N10—C9—N7	116.38 (13)
C2—C1—H1B	109.5	N8—C9—N7	124.41 (13)
H1A—C1—H1B	109.5	N9—C10—N8	118.57 (13)
C2—C1—H1C	109.5	N9—C10—N6	116.26 (13)
H1A—C1—H1C	109.5	N8—C10—N6	125.17 (13)
H1B—C1—H1C	109.5	C4—N1—C3	114.56 (13)
N3—C2—N2	125.90 (14)	C2—N2—C4	114.89 (13)
N3—C2—C1	117.42 (14)	C2—N3—C3	114.50 (13)
N2—C2—C1	116.68 (14)	C3—N4—H4A	120.0
N4—C3—N1	117.82 (13)	C3—N4—H4B	120.0
N4—C3—N3	116.89 (13)	H4A—N4—H4B	120.0
N1—C3—N3	125.28 (13)	C8—N6—C10	114.35 (12)
N5—C4—N1	117.59 (14)	C8—N7—C9	114.99 (12)
N5—C4—N2	117.68 (13)	C10—N8—C9	114.81 (12)
N1—C4—N2	124.72 (14)	C10—N9—H9A	120.0
C6—C5—H5A	109.5	C10—N9—H9B	120.0
C6—C5—H5B	109.5	H9A—N9—H9B	120.0
H5A—C5—H5B	109.5	C9—N10—H10A	120.0
C6—C5—H5C	109.5	C9—N10—H10B	120.0
H5A—C5—H5C	109.5	H10A—N10—H10B	120.0
H5B—C5—H5C	109.5	C6—O1—H1	109.5
O1—C6—C5	109.36 (17)	C12—C11—H11A	109.5
O1—C6—H6A	109.8	C12—C11—H11B	109.5
C5—C6—H6A	109.8	H11A—C11—H11B	109.5
O1—C6—H6B	109.8	C12—C11—H11C	109.5
C5—C6—H6B	109.8	H11A—C11—H11C	109.5
H6A—C6—H6B	108.3	H11B—C11—H11C	109.5
C8—C7—H7A	109.5	O2—C12—C11	114.80 (17)
C8—C7—H7B	109.5	O2—C12—H12A	108.6
H7A—C7—H7B	109.5	C11—C12—H12A	108.6
C8—C7—H7C	109.5	O2—C12—H12B	108.6
H7A—C7—H7C	109.5	C11—C12—H12B	108.6
H7B—C7—H7C	109.5	H12A—C12—H12B	107.5
N6—C8—N7	126.22 (13)	C4—N5—H5D	120.0
N6—C8—C7	117.53 (13)	C4—N5—H5E	120.0

supplementary materials

N7—C8—C7	116.25 (13)	H5D—N5—H5E	120.0
N10—C9—N8	119.21 (13)	C12—O2—H2	109.5
N5—C4—N1—C3	-177.19 (14)	N7—C8—N6—C10	-0.8 (2)
N2—C4—N1—C3	3.9 (2)	C7—C8—N6—C10	178.93 (13)
N4—C3—N1—C4	178.53 (14)	N9—C10—N6—C8	-178.84 (13)
N3—C3—N1—C4	-0.9 (2)	N8—C10—N6—C8	2.1 (2)
N3—C2—N2—C4	-0.2 (2)	N6—C8—N7—C9	-1.2 (2)
C1—C2—N2—C4	178.97 (14)	C7—C8—N7—C9	179.03 (13)
N5—C4—N2—C2	177.66 (14)	N10—C9—N7—C8	-177.94 (13)
N1—C4—N2—C2	-3.4 (2)	N8—C9—N7—C8	2.4 (2)
N2—C2—N3—C3	2.9 (2)	N9—C10—N8—C9	179.85 (13)
C1—C2—N3—C3	-176.34 (14)	N6—C10—N8—C9	-1.1 (2)
N4—C3—N3—C2	178.33 (14)	N10—C9—N8—C10	179.00 (13)
N1—C3—N3—C2	-2.3 (2)	N7—C9—N8—C10	-1.3 (2)

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N4—H4B \cdots N7 ⁱ	0.86	2.11	2.9666 (18)	171
N5—H5D \cdots N8 ⁱⁱ	0.86	2.19	3.0132 (19)	159
N5—H5E \cdots O2 ⁱⁱⁱ	0.86	2.29	3.0071 (19)	142
N10—H10A \cdots O2 ^{iv}	0.86	2.10	2.9337 (18)	163
O2—H2 \cdots O1 ^v	0.82	1.90	2.7185 (18)	174

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

Fig. 1

