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4-Amino-3,5-dimethyl-4*H*-1,2,4-triazole

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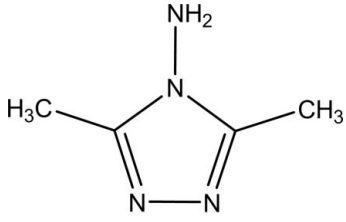
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.143; data-to-parameter ratio = 16.5.

In the title compound, $C_4H_8N_4$, intermolecular N—H···N hydrogen bonds involving the amino groups and triazole N atoms form a two-dimensional sheet.

Related literature

For background, see: Desenko (1995); For further synthetic details, see: Van Albada *et al.* (1984). For related literature, see: Allen *et al.* (1987); Ding *et al.* (2004); Steel (2005); Van Diemen *et al.* (1991); Yi *et al.* (2004).



Experimental

Crystal data

$C_4H_8N_4$
M_r = 112.14
Monoclinic, $P2_1/c$
a = 5.8423 (12) Å
b = 7.7540 (16) Å
c = 12.846 (3) Å
β = 96.91 (3)°
V = 577.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.30 × 0.30 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer
Absorption correction: none
5941 measured reflections
1333 independent reflections
1101 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.142
S = 1.12
1333 reflections
81 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4D···N1^{i}	0.91 (2)	2.25 (2)	3.145 (2)	170 (2)
N)—H4E···N2^{ii}	0.96 (2)	2.20 (2)	3.086 (2)	154 (2)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); 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: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2093).

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supporting information

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4-Amino-3,5-dimethyl-4*H*-1,2,4-triazole

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S1. Comment

N-containing heterocyclic aromatic compounds are extensively used as bridging ligands in coordination and metallosupramolecular chemistry (Steel, 2005). For its strong σ-donor and weak π-acceptor properties, 1,2,4-triazole and its derivatives possess several coordination modes through three N donor atoms coordinating to metal ions (Van Diemen *et al.*, 1991;Yi *et al.*,2004; Ding *et al.*, 2004). We herein report the crystal structure of the title compound (I). In the molecule of (I), (Fig. 1), the bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987). The H atoms of the amino group form hydrogen bonds with the N atoms of neighbouring triazole rings. The geometric parameters of the N—H···N (Spek, 2003) hydrogen-bonding interactions are given in Table 1, and a two dimensional sheet is formed by these intermolecular hydrogen bonds (Fig. 2).

S2. Experimental

A 80% aqueous solution of 2.6 mol of hydrazine hydrate was added slowly to 2.0 mol of acetic acid. The mixture was heated slowly and kept at 493 K for about 3 h. When the mixture was cooled, colourless block shape crystals 4-amino-3,5-dimethyl-4*H*-1,2,4-triazole were isolated.

S3. Refinement

Methyl H atoms were included in calculated positions and treated in the subsequent refinement as riding atoms, with C—H = 0.96Å and U_{iso}(H) = 1.5U_{eq}(C). Atoms H4D and H4E, which are involved in hydrogen-bonding interactions, were located in a difference Fourier map and refined freely with isotropic displacement parameters.

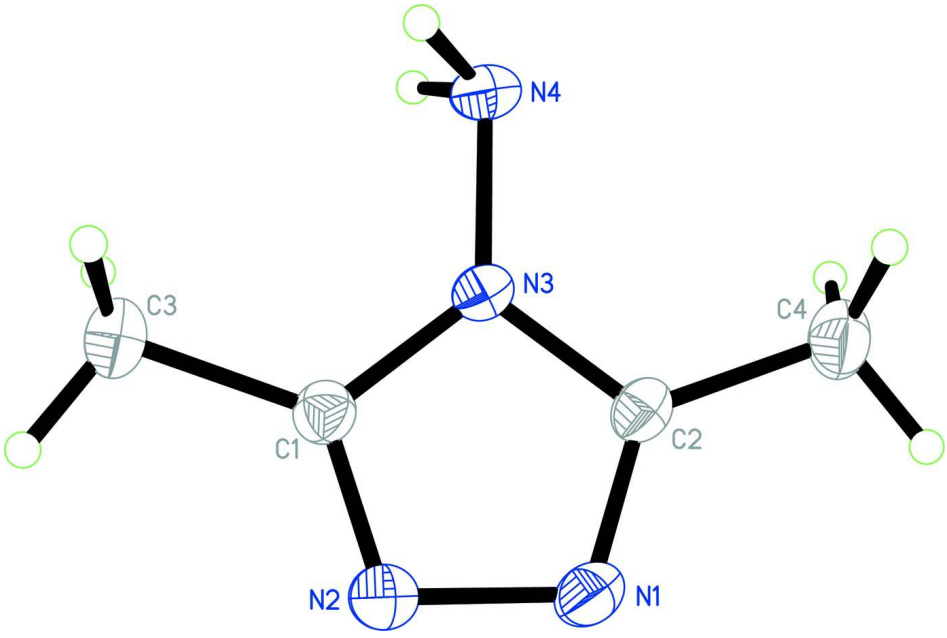

Figure 1

The structure of the title compound with ellipsoids drawn with 30% displacement probability.

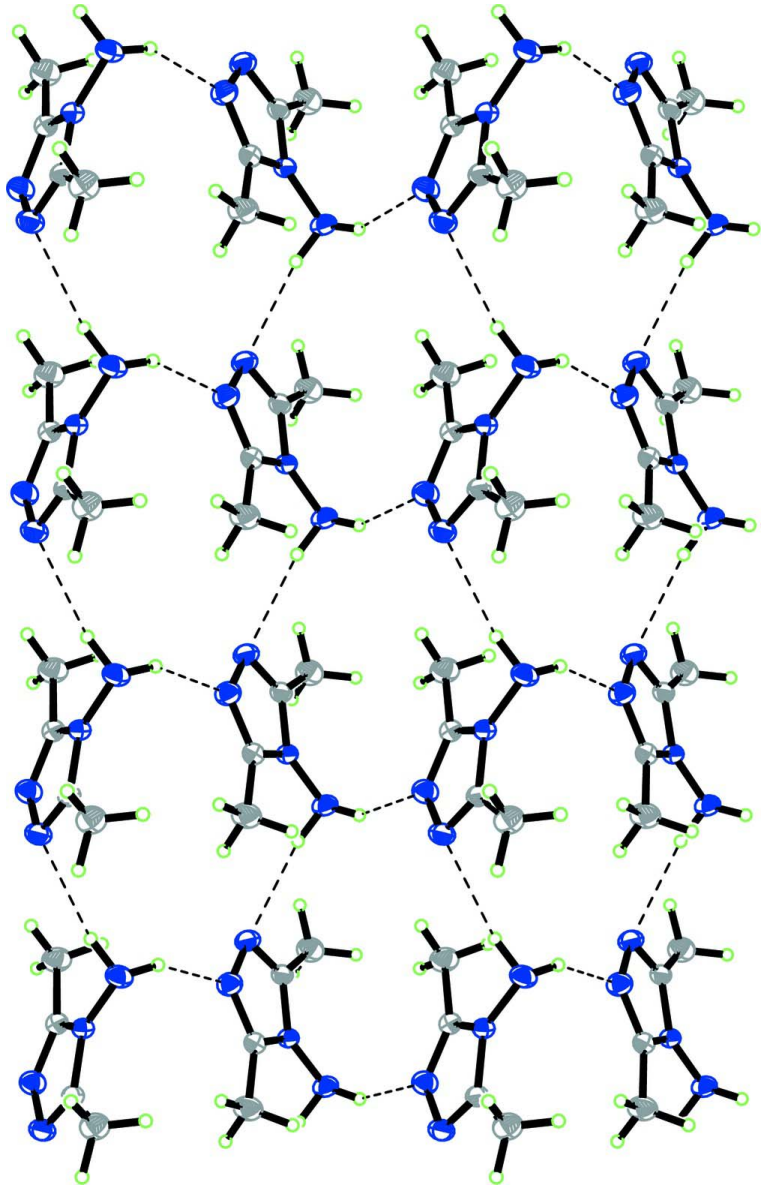

Figure 2

Two dimensional sheet formed by intermolecular hydrogen bonds in the title compound, with the hydrogen bonds shown as dashed lines.

4-Amino-3,5-dimethyl-4*H*-1,2,4-triazole

Crystal data

$C_4H_8N_4$
$M_r = 112.14$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.8423\ (12)$ Å
$b = 7.7540\ (16)$ Å
$c = 12.846\ (3)$ Å
$\beta = 96.91\ (3)°$
$V = 577.7\ (2)$ Å^3
$Z = 4$
$F(000) = 240$
$D_x = 1.289$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5363 reflections
$\theta = 3.1–27.5°$
$\mu = 0.09$ mm^{-1}

T = 293 K
Block, colourless

0.30 × 0.30 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
5941 measured reflections
1333 independent reflections

1101 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 27.5°, θ_{min} = 3.1°
$h = -7 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.142
S = 1.12
1333 reflections
81 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.068P)^2 + 0.1583P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 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.

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_{iso}*/U_{eq}
N3	1.0528 (2)	0.17414 (16)	0.13102 (10)	0.0297 (3)
N4	1.2366 (2)	0.2703 (2)	0.09833 (12)	0.0396 (4)
N2	0.8357 (3)	0.02909 (19)	0.22608 (11)	0.0408 (4)
N1	0.7072 (2)	0.06903 (19)	0.13019 (12)	0.0406 (4)
C2	0.8418 (3)	0.1557 (2)	0.07503 (12)	0.0322 (4)
C1	1.0418 (3)	0.0933 (2)	0.22443 (13)	0.0323 (4)
C3	1.2408 (3)	0.0804 (3)	0.30796 (15)	0.0481 (5)
H3A	1.1950	0.0182	0.3667	0.072*
H3B	1.2906	0.1941	0.3299	0.072*
H3C	1.3651	0.0206	0.2812	0.072*
C4	0.7788 (3)	0.2255 (3)	−0.03203 (14)	0.0467 (5)
H4A	0.6213	0.1969	−0.0559	0.070*
H4B	0.8775	0.1762	−0.0787	0.070*
H4C	0.7968	0.3486	−0.0308	0.070*

H4D	1.362 (4)	0.200 (3)	0.1041 (18)	0.060 (6)*
H4E	1.263 (4)	0.361 (3)	0.1488 (19)	0.064 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N3	0.0243 (7)	0.0317 (7)	0.0332 (7)	−0.0002 (5)	0.0036 (5)	−0.0007 (5)
N4	0.0288 (8)	0.0462 (9)	0.0446 (9)	−0.0059 (7)	0.0082 (6)	0.0035 (7)
N2	0.0359 (8)	0.0444 (8)	0.0421 (9)	−0.0032 (6)	0.0042 (6)	0.0060 (6)
N1	0.0296 (7)	0.0453 (8)	0.0458 (9)	−0.0045 (6)	0.0003 (6)	0.0014 (7)
C2	0.0272 (8)	0.0332 (8)	0.0353 (8)	0.0019 (6)	0.0006 (6)	−0.0042 (6)
C1	0.0301 (8)	0.0323 (8)	0.0342 (9)	0.0017 (6)	0.0029 (6)	0.0009 (6)
C3	0.0413 (10)	0.0579 (11)	0.0426 (10)	0.0024 (9)	−0.0060 (8)	0.0093 (9)
C4	0.0457 (11)	0.0541 (11)	0.0378 (10)	0.0022 (9)	−0.0050 (8)	0.0020 (8)

Geometric parameters (Å, º)

N3—C2	1.358 (2)	C2—C4	1.482 (2)
N3—C1	1.362 (2)	C1—C3	1.487 (2)
N3—N4	1.4123 (18)	C3—H3A	0.9600
N4—H4D	0.91 (2)	C3—H3B	0.9600
N4—H4E	0.95 (2)	C3—H3C	0.9600
N2—C1	1.305 (2)	C4—H4A	0.9600
N2—N1	1.398 (2)	C4—H4B	0.9600
N1—C2	1.306 (2)	C4—H4C	0.9600
C2—N3—C1	106.40 (13)	N3—C1—C3	123.42 (15)
C2—N3—N4	124.91 (14)	C1—C3—H3A	109.5
C1—N3—N4	128.54 (13)	C1—C3—H3B	109.5
N3—N4—H4D	106.9 (14)	H3A—C3—H3B	109.5
N3—N4—H4E	104.6 (13)	C1—C3—H3C	109.5
H4D—N4—H4E	109 (2)	H3A—C3—H3C	109.5
C1—N2—N1	107.45 (14)	H3B—C3—H3C	109.5
C2—N1—N2	107.31 (13)	C2—C4—H4A	109.5
N1—C2—N3	109.51 (14)	C2—C4—H4B	109.5
N1—C2—C4	126.35 (15)	H4A—C4—H4B	109.5
N3—C2—C4	124.14 (15)	C2—C4—H4C	109.5
N2—C1—N3	109.34 (14)	H4A—C4—H4C	109.5
N2—C1—C3	127.23 (16)	H4B—C4—H4C	109.5
C1—N2—N1—C2	−0.04 (18)	N1—N2—C1—N3	0.12 (18)
N2—N1—C2—N3	−0.05 (18)	N1—N2—C1—C3	−178.51 (17)
N2—N1—C2—C4	−179.82 (16)	C2—N3—C1—N2	−0.15 (18)
C1—N3—C2—N1	0.12 (18)	N4—N3—C1—N2	175.53 (15)
N4—N3—C2—N1	−175.76 (14)	C2—N3—C1—C3	178.54 (16)
C1—N3—C2—C4	179.90 (15)	N4—N3—C1—C3	−5.8 (3)
N4—N3—C2—C4	4.0 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*D*···N1^{i}	0.91 (2)	2.25 (2)	3.145 (2)	170 (2)
N)—H4*E*···N2^{ii}	0.96 (2)	2.20 (2)	3.086 (2)	154 (2)

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+2, *y*+1/2, −*z*+1/2.