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5-Amino-3-methyl-1-phenyl-1*H*-1,2,4-triazole

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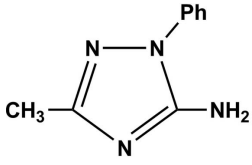
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.051; wR factor = 0.148; data-to-parameter ratio = 31.3.

In the title compound, $C_9H_{10}N_4$, the phenyl and triazole rings make a dihedral angle of 38.80 (2)°. N—H···N hydrogen bonds link the molecules, forming centrosymmetric $R_2^2(8)$ rings; these rings are interconnected through a $C(5)$ chain, building up a zigzag layer parallel to the (100) plane.

Related literature

For related literature, see: Altman & Solomost (1993); Genady & Gabel (2003); Kanazawa *et al.* (1988); Karanik *et al.* (2003); Hashimoto *et al.* (1990); Allouch *et al.* (2004). For a discussion of hydrogen-bond patterns, see: Bernstein *et al.* (1995); Etter *et al.* (1990).



Experimental

Crystal data

$C_9H_{10}N_4$
M_r = 174.21
Monoclinic, $P2_1/c$
a = 8.5110 (5) Å
b = 11.2490 (8) Å
c = 10.1048 (7) Å
β = 101.866 (4)°
V = 946.76 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 296 (7) K
0.49 × 0.14 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.984, T_{max} = 0.997
16028 measured reflections
3882 independent reflections
1997 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.148
S = 0.94
3882 reflections
124 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···N3^{i}	0.886 (8)	2.110 (8)	2.9923 (13)	173.1 (12)
N4—H4B···N2^{ii}	0.917 (8)	2.210 (10)	3.0415 (14)	150.4 (10)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2319).

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supporting information

Acta Cryst. (2008). E**64**, o684 [doi:10.1107/S1600536808005801]

5-Amino-3-methyl-1-phenyl-1*H*-1,2,4-triazole

Fatma Allouch, Fatma Zouari, Fakher Chabchoub and Mansour Salem

S1. Comment

The aminotriazoles are crucial heterocyclic substances which have a great interest thanks to their biological and pharmacological activity (Kanazawa *et al.*, 1988; Hashimoto *et al.*, 1990) such as antitumoral and inhibition of cholesterol activity. In addition they have many applications in agriculture domain (Altman *et al.*, 1993). Aminotriazole are useful binucleophilic agents that lead to polycondensed heterocycles (Genady *et al.*, 2003; Karanik *et al.*, 2003). However, the studies that deal with N^1-phenyl-aminotriazole are very limited (Allouch *et al.*, 2004). Until now only a few reactions were reported concerning the addition-cyclizations of bielectrophile compounds with N^1-phenyl-aminotriazoles. In fact, these later are not well identified, this can be explicable by the existence of the tautomer equilibrium. That is why we have undertaken a crystallographic study.

In this paper, we report the synthesis of 5-amino-3-méthyl N^1-phényl-1,2,4-triazole. The reaction of *N*-phenyl ethyl acetydrazonate with cyanamidegave aminotriazole could lead to structure (I) or its isomer (II) (Scheme). The structure elucidation was achieved by X-ray diffraction, and proved that the reaction occurs cleanly to form 5-amino-3-méthyl N^1-phényl-1,2,4-triazole (I).

In the title compound, the phenyl and the triazole rings remain planar with mean deviations from planarity of 0.0072 and 0.0049Å respectively. However, the two rings are twisted with respect to each other making a dihedral angle of 38.80 (2)° (Fig. 1).

The occurrence of N—H···N hydrogen bonds links the molecules through inversion centre to form $R_2^2(8)$ ring (Etter *et al.*, 1990; Bernstein *et al.*, 1995) and these rings are interconnected through C(5) chain to build up a like zigzag layer developping along the (1 0 0) plane (Table 1, Fig. 2)

S2. Experimental

A mixture containing 3.56 g (0.02 mol) of *N*-phenyl ethyl acetydrazonateand 0.88 g (0.021 mol) of cyanamidein 20 ml of methanol was stirred and heated to reflux for 12 h. The solvent was removed under rotary evaporation. The crude product was washed with ether then recrystallized from methanol to give analytically pure crystals.

S3. Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.96 Å (methyl) and 0.93 Å (aromatic) with U_{iso}(H) = 1.2U_{eq}(Phenyl) or U_{iso}(H) = 1.5U_{eq}(methyl). The methyl was found to be statistically disordered over two positions. H atoms attached to nitrogen were located in difference Fourier maps and included in the subsequent refinement using soft restraints (N—H= 0.90 (1)Å and H···H= 1.59 (2) Å) with U_{iso}(H) = 1.2U_{eq}(N).

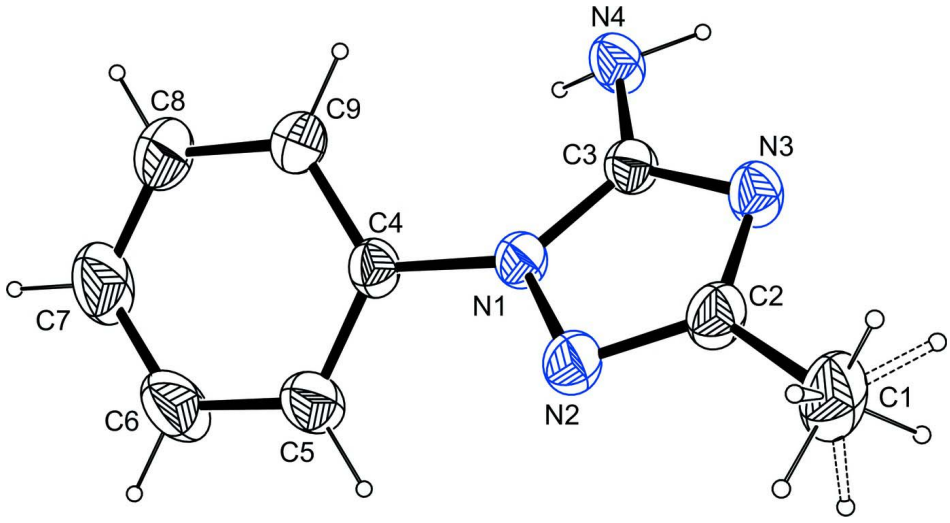

Figure 1

The molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

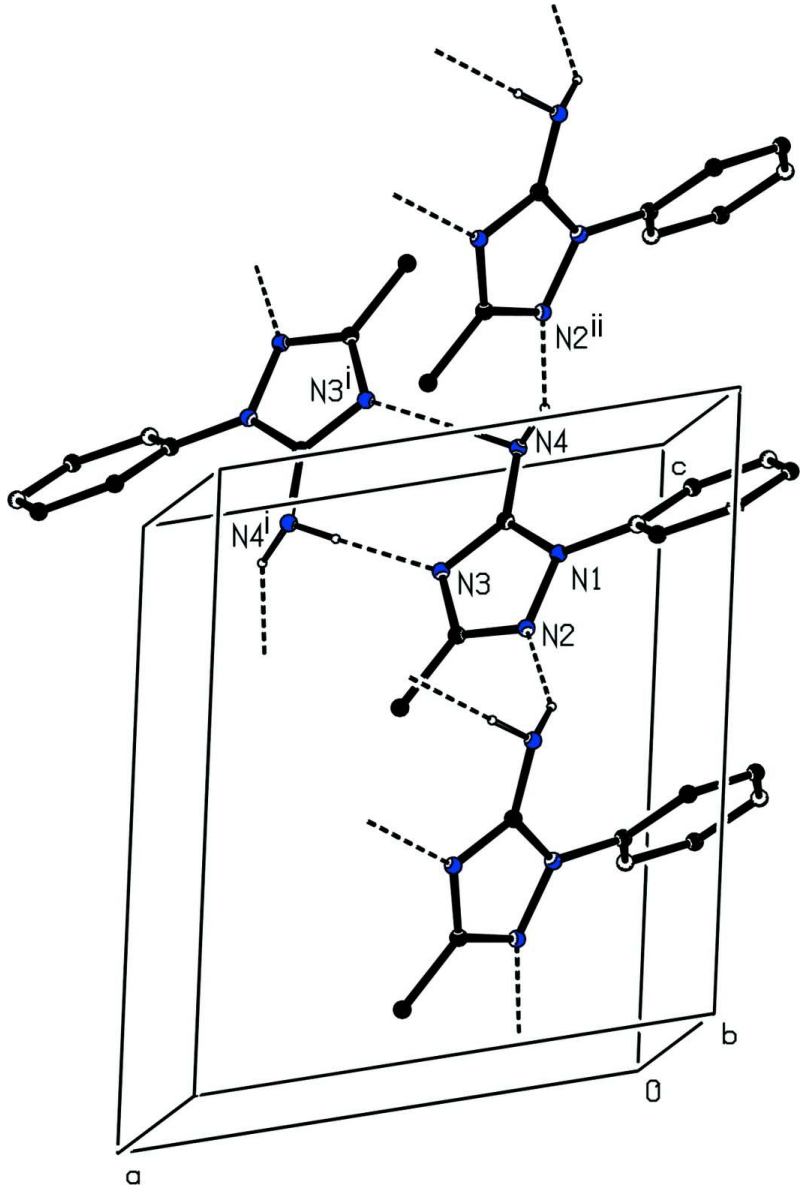

Figure 2

Partial packing view showing the formation of the $R_2^2(8)$ ring and C(5) chains through N—H···N hydrogen bonds drawn as dashed lines. H atoms not involved in hydrogen bondings have been omitted for clarity. [Symmetry codes: (i) -*x* + 1, -*y*, -*z* + 2; (ii) *x*, -*y* + 1/2, *z* + 1/2]

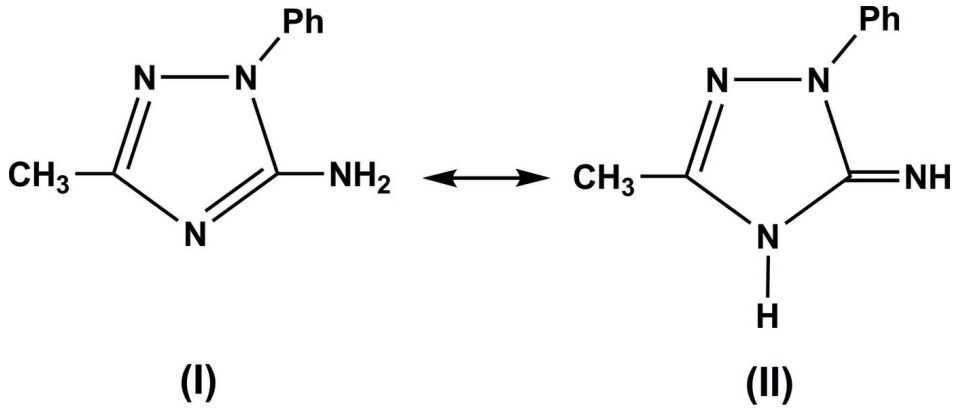

Figure 3

The tautomeric forms of the title compound.

5-Amino-3-methyl-1-phenyl-1*H*-1,2,4-triazole

Crystal data

$C_9H_{10}N_4$
M_r = 174.21
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.5110 (5) Å
b = 11.2490 (8) Å
c = 10.1048 (7) Å
β = 101.866 (4)°
V = 946.76 (11) Å^3
Z = 4
$F(000)$ = 368
D_x = 1.222 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2898 reflections
θ = 2.5–23.3°
μ = 0.08 mm^{-1}
T = 296 K
Prism, colourless
0.49 × 0.14 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.984, T_{max} = 0.997
16028 measured reflections
3882 independent reflections
1997 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 34.4°, θ_{min} = 3.0°
h = −11→13
k = −17→17
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.148
S = 0.94
3882 reflections
124 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.0699P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.22326 (10)	0.20008 (8)	0.83687 (9)	0.0445 (2)	
N2	0.28380 (11)	0.22738 (9)	0.72310 (10)	0.0530 (3)	
N3	0.43302 (11)	0.08529 (9)	0.84645 (9)	0.0500 (3)	
N4	0.29094 (12)	0.06921 (9)	1.02612 (10)	0.0528 (3)	
H4A	0.3772 (12)	0.0290 (10)	1.0662 (12)	0.063*	
H4B	0.2503 (14)	0.1219 (9)	1.0797 (11)	0.063*	
C4	0.08173 (12)	0.25677 (10)	0.85950 (12)	0.0454 (3)	
C3	0.31616 (12)	0.11612 (9)	0.90899 (11)	0.0424 (3)	
C2	0.40669 (14)	0.15538 (11)	0.73467 (12)	0.0535 (3)	
C9	−0.03152 (14)	0.19287 (12)	0.90944 (15)	0.0606 (3)	
H9	−0.0159	0.1124	0.9286	0.073*	
C5	0.05851 (16)	0.37569 (12)	0.82934 (14)	0.0663 (4)	
H5	0.1354	0.4193	0.7968	0.080*	
C1	0.51079 (17)	0.15076 (16)	0.63304 (15)	0.0792 (5)	
H1A	0.5936	0.0925	0.6601	0.119*	0.50
H1B	0.5588	0.2273	0.6270	0.119*	0.50
H1C	0.4471	0.1295	0.5464	0.119*	0.50
H1D	0.4727	0.2070	0.5623	0.119*	0.50
H1E	0.5076	0.0722	0.5953	0.119*	0.50
H1F	0.6192	0.1700	0.6760	0.119*	0.50
C7	−0.19460 (19)	0.36600 (17)	0.89769 (18)	0.0921 (6)	
H7	−0.2890	0.4028	0.9085	0.111*	
C6	−0.0814 (2)	0.42885 (14)	0.84843 (18)	0.0877 (5)	
H6	−0.0991	0.5089	0.8274	0.105*	
C8	−0.16821 (16)	0.24917 (14)	0.93076 (18)	0.0797 (5)	
H8	−0.2428	0.2072	0.9679	0.096*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0431 (4)	0.0488 (5)	0.0457 (5)	0.0065 (4)	0.0189 (4)	0.0059 (4)
N2	0.0492 (5)	0.0649 (6)	0.0499 (6)	0.0084 (4)	0.0218 (5)	0.0126 (5)
N3	0.0474 (5)	0.0574 (6)	0.0487 (6)	0.0104 (4)	0.0181 (4)	0.0014 (4)
N4	0.0584 (6)	0.0544 (6)	0.0507 (6)	0.0157 (5)	0.0228 (5)	0.0088 (5)
C4	0.0414 (5)	0.0509 (6)	0.0457 (6)	0.0077 (4)	0.0130 (5)	0.0019 (5)
C3	0.0439 (5)	0.0418 (6)	0.0437 (6)	0.0034 (4)	0.0140 (5)	−0.0003 (5)

C2	0.0480 (6)	0.0669 (7)	0.0500 (7)	0.0048 (5)	0.0200 (5)	0.0064 (6)
C9	0.0476 (6)	0.0612 (8)	0.0777 (9)	0.0036 (5)	0.0243 (6)	0.0070 (7)
C5	0.0711 (8)	0.0611 (8)	0.0754 (10)	0.0174 (6)	0.0353 (7)	0.0164 (7)
C1	0.0679 (8)	0.1133 (12)	0.0673 (10)	0.0214 (8)	0.0392 (7)	0.0151 (8)
C7	0.0709 (9)	0.1057 (13)	0.1108 (14)	0.0396 (9)	0.0445 (9)	0.0214 (10)
C6	0.0926 (11)	0.0767 (10)	0.1056 (13)	0.0420 (8)	0.0481 (10)	0.0290 (9)
C8	0.0534 (8)	0.0923 (11)	0.1032 (12)	0.0100 (7)	0.0385 (8)	0.0146 (9)

Geometric parameters (Å, º)

N1—C3	1.3459 (14)	C5—C6	1.3811 (18)
N1—N2	1.3874 (11)	C5—H5	0.9300
N1—C4	1.4224 (12)	C1—H1A	0.9600
N2—C2	1.3090 (14)	C1—H1B	0.9600
N3—C3	1.3294 (12)	C1—H1C	0.9600
N3—C2	1.3577 (15)	C1—H1D	0.9600
N4—C3	1.3528 (14)	C1—H1E	0.9600
N4—H4A	0.886 (8)	C1—H1F	0.9600
N4—H4B	0.917 (8)	C7—C8	1.363 (2)
C4—C5	1.3773 (17)	C7—C6	1.369 (2)
C4—C9	1.3785 (15)	C7—H7	0.9300
C2—C1	1.4888 (15)	C6—H6	0.9300
C9—C8	1.3799 (17)	C8—H8	0.9300
C9—H9	0.9300		
C3—N1—N2	109.09 (7)	H1A—C1—H1C	109.5
C3—N1—C4	130.56 (8)	H1B—C1—H1C	109.5
N2—N1—C4	120.33 (9)	C2—C1—H1D	109.5
C2—N2—N1	102.42 (9)	H1A—C1—H1D	141.1
C3—N3—C2	103.43 (9)	H1B—C1—H1D	56.3
C3—N4—H4A	109.5 (8)	H1C—C1—H1D	56.3
C3—N4—H4B	114.3 (8)	C2—C1—H1E	109.5
H4A—N4—H4B	115.9 (11)	H1A—C1—H1E	56.3
C5—C4—C9	120.55 (10)	H1B—C1—H1E	141.1
C5—C4—N1	119.18 (9)	H1C—C1—H1E	56.3
C9—C4—N1	120.27 (10)	H1D—C1—H1E	109.5
N3—C3—N1	109.84 (9)	C2—C1—H1F	109.5
N3—C3—N4	125.66 (10)	H1A—C1—H1F	56.3
N1—C3—N4	124.49 (9)	H1B—C1—H1F	56.3
N2—C2—N3	115.20 (9)	H1C—C1—H1F	141.1
N2—C2—C1	122.54 (11)	H1D—C1—H1F	109.5
N3—C2—C1	122.26 (11)	H1E—C1—H1F	109.5
C4—C9—C8	119.55 (13)	C8—C7—C6	119.63 (12)
C4—C9—H9	120.2	C8—C7—H7	120.2
C8—C9—H9	120.2	C6—C7—H7	120.2
C4—C5—C6	118.57 (12)	C7—C6—C5	121.25 (14)
C4—C5—H5	120.7	C7—C6—H6	119.4
C6—C5—H5	120.7	C5—C6—H6	119.4

C2—C1—H1A	109.5	C7—C8—C9	120.39 (13)
C2—C1—H1B	109.5	C7—C8—H8	119.8
H1A—C1—H1B	109.5	C9—C8—H8	119.8
C2—C1—H1C	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···N3^{i}	0.89 (1)	2.11 (1)	2.9923 (13)	173 (1)
N4—H4*B*···N2^{ii}	0.92 (1)	2.21 (1)	3.0415 (14)	150 (1)

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