Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
6136 measured reflections
1692 independent reflections
1220 reflections with $I > 3\sigma(I)$
$R_{\text{int}} = 0.044$
$\theta_{\text{max}} = 30.0°$
$h = -25 \rightarrow 25$
$k = -5 \rightarrow 5$
$l = -22 \rightarrow 22$
3 standard reflections
frequency: 120 min
intensity decay: 1.6%

Refinement

Refinement on F
$R = 0.043$
$wR = 0.071$
$S = 1.232$
1220 reflections
95 parameters
H atoms: see below
$w = 1/[\sigma^2(F) + (0.045F)^2]$
$(\Delta/\sigma)_{\text{max}} = 0.018$
$\Delta\rho_{\text{max}} = 0.143$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.095$ e Å^{-3}
Extinction correction: Stout & Jensen (1968), formula 17.16
Extinction coefficient: $3.976(6) \times 10^{-6}$
Scattering factors from *SDP/PDP* (Enraf–Nonius, 1985)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O1	0.41131 (6)	−0.1925 (4)	0.15292 (7)	0.0545 (3)
O2	0.31355 (7)	0.0426 (4)	0.08584 (7)	0.0601 (3)
N1	0.31503 (6)	0.0694 (3)	0.34356 (6)	0.0311 (2)
N2	0.32041 (6)	0.0516 (4)	0.21382 (6)	0.0342 (3)
N3	0.35095 (7)	−0.0373 (4)	0.14993 (7)	0.0378 (3)
C2	0.35668 (7)	−0.0279 (3)	0.28721 (7)	0.0281 (3)
C3	0.42603 (7)	−0.1823 (4)	0.31444 (8)	0.0339 (3)
C4	0.44784 (8)	−0.2285 (4)	0.39414 (9)	0.0391 (3)
C5	0.40256 (8)	−0.1269 (4)	0.44970 (9)	0.0412 (4)
C6	0.33611 (8)	0.0230 (5)	0.42173 (8)	0.0375 (3)

Table 2. *Selected geometric parameters (Å, °)*

O1—N3	1.239 (2)	N2—C2	1.360 (2)
O2—N3	1.237 (2)	C2—C3	1.407 (2)
N1—C2	1.360 (2)	C3—C4	1.370 (2)
N1—C6	1.343 (2)	C4—C5	1.399 (2)
N2—N3	1.336 (2)	C5—C6	1.358 (2)
N3—N2—C2	119.4 (1)	O2—N3—N2	114.7 (1)
O1—N3—O2	121.4 (1)	N1—C2—N2	110.1 (1)
O1—N3—N2	123.9 (1)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
N1—HN1···N2$^{\text{i}}$	0.95 (2)	1.95 (2)	2.890 (3)	175.7 (18)
C3—H3···O1	0.95	2.18	2.728 (2)	115.4
C4—H4···O1$^{\text{ii}}$	0.95	2.56	3.291 (2)	134.3
C5—H5···O1$^{\text{iii}}$	0.95	2.60	3.509 (2)	160.3
C6—H6···O2$^{\text{iv}}$	0.95	2.54	3.300 (2)	137.5
C6—H6···O2$^{\text{i}}$	0.95	2.55	3.161 (2)	122.1

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

The H atoms bonded to C atoms were constrained to idealized positions, while the amino H atom was located from a difference Fourier map and further included in the refinement. All H atoms were assigned isotropic U values of 0.0506 Å^2.

Data collection: *CAD*-4 *Users Manual* (Enraf–Nonius, 1988). Data reduction: *SDP/PDP* (Enraf–Nonius, 1985). Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *SDP/PDP*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *KAPPA* (Macíček, 1992).

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

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Abstract

The bond lengths in the five-membered ring of the title compound, $C_3H_3F_3N_4$, (1), are equal within three standard deviations to those in 5-amino-3-nitro-1*H*-1,2,4-triazole. The amino group in (1) has a trigonal-

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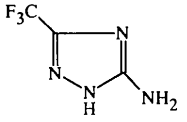
pyramidal configuration. Molecules of (1) form layers in the *ab* plane *via* intermolecular hydrogen bonds of the type N—H···N.

Comment

5-Amino-1,2,4-triazole and its derivatives are starting materials for the synthesis of many heterocycles (Desenko, 1995). Knowledge of the molecular structure of these compounds is very important for understanding their reactivity under condensation reaction conditions. Comparison of the bond lengths in (1) with those in the 3,5-diamino-, 5-amino- and 3-nitro-5-amino- derivatives of 1*H*-1,2,4-triazole revealed that the nature of the substituent in the 3-position of the triazole ring influenced the N(1)—N(2) bond length. The presence of the electron-donating amino group in this position leads to an elongation to 1.398 Å (Starova *et al.*, 1980), while this bond becomes shorter [1.371 Å in (1)] when an electron-withdrawing group such as nitro or trifluoromethyl is present in this position. The bond lengths in the five-membered ring of (1) lie within three standard deviations of those found in 5-amino-3-nitro-1*H*-1,2,4-triazole (Garcia & Lee, 1992).



(1)

The amino group in (1) has a trigonal-pyramidal configuration. The N atom is displaced out of the plane of its three neighbouring atoms by 0.20 (2) Å. A similar configuration was observed in other 5-amino derivatives of 1*H*-1,2,4-triazole (Garcia & Lee, 1992; Starova *et al.*, 1978, 1980).

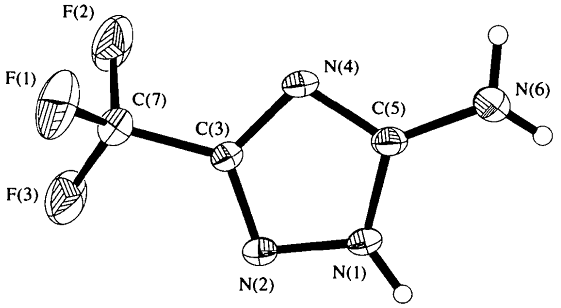


Fig. 1. A view of (1) in which the non-H atoms are shown with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as circles of arbitrary radii for clarity. The structure is projected on to the *ab* plane.

Molecules of (1) form layers due to intermolecular H(1)···N(4^{i}) hydrogen bonds [H···N 1.96 (4) Å and N(1)—H(1)···N(4^{i}) 158 (3)°; symmetry code: (i) $-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$] and shortened intermolecular contacts, H(6*A*)···N(2^{ii}) 2.48 (4) Å [symmetry code: (ii) $-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$; the van der Waals radii sum is 2.66 Å (Zefirov & Zorky, 1989)]. These layers are parallel to the *ab* plane. There are possible intermolecular contacts [F(1)···F(1^{iii}) 2.742 (3) Å; the van der Waals sum is 2.80 Å; symmetry code: (iii) $1-x$, $1-y$, $1-z$] between molecules belonging to neighbouring layers.

Experimental

Crystals of (1) were obtained by slow diffusion of toluene into a solution of (1) in ethyl acetate.

Crystal data

$C_3H_3F_3N_4$
$M_r = 152.09$
Monoclinic
$P2_1/c$
$a = 8.629\ (3)$ Å
$b = 9.599\ (3)$ Å
$c = 6.739\ (3)$ Å
$\beta = 90.29\ (3)°$
$V = 558.1\ (4)$ Å^3
$Z = 4$
$D_x = 1.810$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 10–11°$
$\mu = 0.193$ mm^{-1}
$T = 153\ (2)$ K
Plate
0.40 × 0.30 × 0.20 mm
Colourless

Data collection

Siemens *P*3/*PC* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4111 measured reflections
1960 independent reflections
1538 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\rm max} = 32.97°$
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -10 \rightarrow 9$
2 standard reflections every 98 reflections intensity decay: 5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.312$
$S = 1.059$
1901 reflections
103 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0864P)^2 + 0.5663P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.52$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.38$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

F(1)—C(7)	1.327 (3)	N(2)—C(3)	1.311 (2)
F(2)—C(7)	1.335 (3)	N(4)—C(5)	1.335 (2)
F(3)—C(7)	1.313 (3)	N(4)—C(3)	1.351 (2)
N(1)—C(5)	1.343 (2)	N(6)—C(5)	1.357 (2)
N(1)—N(2)	1.371 (2)	C(3)—C(7)	1.493 (3)
C(5)—N(1)—N(2)	110.13 (14)	N(4)—C(5)—N(1)	109.62 (15)
C(3)—N(2)—N(1)	101.41 (13)	N(4)—C(5)—N(6)	125.6 (2)
C(5)—N(4)—C(3)	102.40 (14)	N(1)—C(5)—N(6)	124.7 (2)
N(2)—C(3)—N(4)	116.4 (2)		

H atoms were found from ΔF synthesis, then refined freely with individual isotropic displacement parameters.

Data collection: *P*3 (Siemens, 1989). Cell refinement: *P*3. Data reduction: *PROFIT* (Strel'tsov & Zavodnik, 1989). Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1994). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

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