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Structure of 3-Amino-5-nitro-1,2,4-triazole*

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Abstract. $C_2H_3N_5O_2$, $M_r = 129{\cdot}1$, monoclinic, $C2/c$, $a = 14{\cdot}199\ (4)$, $b = 4{\cdot}844\ (1)$, $c = 14{\cdot}258\ (4)$ Å, $\beta = 105{\cdot}98\ (1)°$, $V = 942{\cdot}7\ (4)$ Å^3, $Z = 8$, $D_x = 1{\cdot}819$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 0{\cdot}149$ mm^{-1}, $F(000) = 528$, room temperature, final $R = 0{\cdot}028$ for 705 observed reflections [$F > 5\sigma(F)$] out of 834 independent reflections. The ring geometry is very similar to other substituted 1,2,4-triazoles and is planar within 0·002 Å. The molecules are joined by N—H···N hydrogen bonds to form flat ribbons. Adjacent ribbons are joined by N—H···O hydrogen bonds.

Experimental. Yellow crystals of the title compound were prepared according to Lee & Storm (1990) and grown from an ethanol and chloroform mixture. Selected crystal *ca* 0·5 × 0·3 × 0·05 mm. Siemens *R3m/V* diffractometer, θ–2θ scan. Scan range 1·2° + α_1,α_2 separation. Scan speed 2·5–15° min^{-1}. Background stationary crystal and counter beginning and end of scan, each 25% of total scan time. Graphite-monochromated Mo $K\alpha$ radiation. Unit cell, 20 reflections $7 < 2\theta < 25°$. No absorption corrections. $\sin\theta/\lambda_{max} = 0.60$ Å^{-1}. Index rnage $-16 \le h \le 16$, $0 \le k \le 5$, $-16 \le l \le 16$. 3774 reflections measured and averaged to yield 834 unique reflections of which 705 were observed with $F > 5\sigma(F)$, $R_{int} = 0{\cdot}028$. Standard reflections 222 and $\bar{5}15$ showed no significant variation. Least squares minimized $\sum w(\Delta F)^2$ with $w = [\sigma_c^2(F) + 0{\cdot}0003F^2]^{-1}$, $\sigma_c^2(F)$ based on counting statistics. All structure solution, refinement and graphics software was from the *SHELXTL-Plus*

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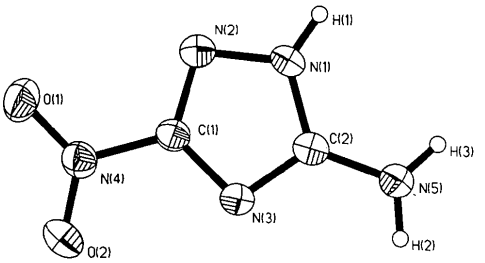

Fig. 1. Drawing of the unique molecule showing the atom-numbering scheme. Thermal ellipsoids are 50% probability with H atoms arbitrary.

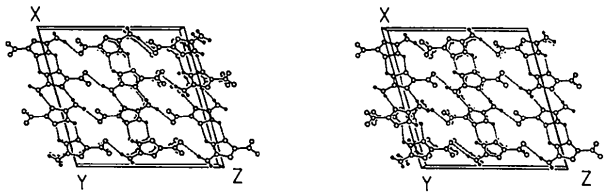

Fig. 2. Stero drawing to show the ribbon structure. Hydrogen bonds are dotted. The view is along the *b* axis with *c* vertical and *a* horizontal. The origin is at the upper left rear.

Table 1. *Positional parameters* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^2) *for* 3-*amino*-5-*nitro*-1,2,4-*triazole*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	8632 (1)	0427 (3)	3942 (1)	2·5 (1)
C(2)	9019 (1)	2133 (3)	5329 (1)	2·6 (1)
N(1)	8356 (1)	0101 (3)	5277 (1)	3·0 (1)
N(2)	8094 (1)	−1049 (3)	4365 (1)	3·1 (1)
N(3)	9209 (1)	2397 (2)	4468 (1)	2·7 (1)
N(4)	8598 (1)	−0020 (3)	2930 (1)	3·0 (1)
N(5)	9444 (1)	3585 (3)	6135 (1)	3·6 (1)
O(1)	8173 (1)	−2077 (2)	2522 (1)	4·6 (1)
O(2)	8999 (1)	1677 (2)	2537 (1)	4·3 (1)

Table 2. *Bond distances* (Å) *and angles* (°) *involving* C, N *and* O *in* 3-*amino*-5-*nitro*-1,2,4-*triazole*

C(1)—N(2)	1·308 (2)	C(2)—N(5)	1·342 (2)
C(1)—N(3)	1·344 (2)	N(1)—N(2)	1·368 (2)
C(1)—N(4)	1·447 (2)	N(4)—O(1)	1·226 (2)
C(2)—N(1)	1·350 (2)	N(4)—O(2)	1·221 (2)
C(2)—N(3)	1·333 (2)		

N(2)—C(1)—N(3)	118·4 (1)	C(2)—N(1)—N(2)	110·6 (1)
N(2)—C(1)—N(4)	120·9 (1)	C(1)—N(2)—N(1)	100·0 (1)
N(3)—C(1)—N(4)	120·7 (1)	C(1)—N(3)—C(2)	101·5 (1)
N(1)—C(2)—N(3)	109·5 (1)	C(1)—N(4)—O(1)	118·8 (2)
N(1)—C(2)—N(5)	124·8 (1)	C(1)—N(4)—O(2)	118·2 (1)
N(3)—C(2)—N(5)	125·6 (1)	O(1)—N(4)—O(2)	124·1 (1)

Hydrogen bonds

X—H···Y	Symmetry operation on Y	$d(X—Y)$	d(H···Y)	$\angle X$—H···Y
N(1)—H(1)···N(2)	$\frac{1}{2}-x, -\frac{1}{2}-y, 1-z$	2·989 (2)	2·355 (8)	132·5 (5)
N(5)—H(2)···N(3)	$2-x, 1-y, 1-z$	3·015 (2)	2·173 (10)	164·2 (9)
N(5)—H(3)···O(1)	$x, -y, \frac{1}{2}+z$	3·109 (2)	2·274 (4)	156·3 (6)

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package (Sheldrick, 1990). Structure solved by direct methods. Scale factor, extinction parameter [χ; 8 (3) $\times 10^{-4}$]* similar to that described by Larson (1969), positional parameters, anisotropic thermal parameters for C, N and O, and isotropic thermal parameters for H were refined (95 parameters). Final $R = 0.027$, $wR = 0.036$, $S = 1.34$. Maximum $\Delta/\sigma = 0.06$. Final ΔF Fourier synthesis $-0.15 < \Delta\rho < 0.17$ e Å^{-3}. Scattering factors f (RHF for C, N, O and SDS for H), f', f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV).†

Fig. 1 is a drawing to show the atom-numbering scheme. Fig. 2 is a stereo drawing showing the ribbons of hydrogen-bonded molecules. Final parameters are given in Table 1. Bond lengths and angles, including hydrogen bonds, are given in Table 2. Nitro-group parameters are in the range of values found in other nitro-substituted rings. In particular, the bond lengths and angles are virtually identical to those in the hydrated form of this molecule (Cromer & Storm, 1991). The nitro group is twisted out of the plane by 10.5° and the amino group by 5.4°, more than twice as much as in the hydrated crystal.

Related literature. See Cromer, Hall, Lee & Ryan, (1988*a*,*b*) for further triazole and small explosive molecule references and Garcia, Lee & Storm (1992) for the structure of the hydrazinium salt of the present compound.

* Corrected structure factor $F^* = F_c/(1 + 0.002\chi F_c^2/\sin 2h)^{1/4}$.

† Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths involving H have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54121 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of the Hydrazinium Salt of 3-Amino-5-nitro-1,2,4-triazole*

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Abstract. Hydrazinium 3-amino-5-nitro-1,2,4-triazolide, $N_2H_5^+.C_2H_2N_5O_2^-$, $M_r = 161.1$, orthorhombic, *Pbca*, $a = 5.392$ (2), $b = 13.316$ (2), $c = 18.483$ (6) Å, $V = 1327.1$ (7) Å^3, $Z = 8$, $D_x = 1.612$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.130$ mm^{-1}, $F(000) = 672$, room temperature, final $R = 0.030$ for 717 observed reflections [$F > 5\sigma(F)$] out of 1157 independent reflections. The ring geometry is very similar to other substituted 1,2,4-triazoles and is planar within 0.003 Å. The structure is held together by an extensive network of hydrogen bonds.

Experimental. Yellow crystals of the title compound were prepared according to Lee & Storm (1990) and grown from an ethanol and chloroform mixture. Selected crystal *ca* $0.4 \times 0.4 \times 0.16$ mm, Enraf–Nonius CAD-4 diffractometer, θ–2θ scan, scan range $(1 + 0.34 \tan\theta)°$, scan speed 1.6 to 5.5° min^{-1}, background first and last $\frac{1}{6}$th of scan, graphite-monochromated Mo $K\alpha$ radiation. Unit cell from 25 reflections with $16 < 2\theta < 26°$. No absorption corrections. $(\sin\theta)/\lambda_{max} = 0.60$ Å^{-1}. Index range $-6 \le h \le 6$, $0 \le k \le 15$, $0 \le l \le 21$. 2513 reflections measured and averaged to yield 1157 unique reflections of which 717 were observed with $F > 5\sigma(F)$, $R_{int} = 0.016$. Standard reflections (152 and 811) showed no significant variation. Least squares minimized $\sum w(\Delta F)^2$ with $w = [\sigma_c^2(F) + 0.0002F^2]^{-1}$, $\sigma_c^2(F)$ based on counting statistics. Structure solution, refinement and graphics software was from the *SHELXTL-Plus* (Sheldrick, 1990) package. Structure solved by direct methods. Scale factor, extinction parameter [χ;

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