of aromatic rings bridged by an ethynyl spacer see Prince, Evans, Fronczek & Gandour (1992), and references therein.

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Structure of the 3-Amino-1,2,4-triazolium Salt of 3-Nitro-1,2,4-triazol-5-one

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Abstract. $C_2H_5N_4^+.C_2HN_4O_3^-$, $M_r = 214.2$, monoclinic, $P2_1/c$, $a = 6.539\ (2)$, $b = 19.063\ (8)$, $c = 6.749\ (4)$ Å, $\beta = 94.31\ (4)°$, $V = 838.9\ (6)$ Å^3, $Z = 4$, $D_m = 1.716$, $D_x = 1.700$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7069 Å, $\mu = 1.6$ cm^{-1}, $F(000) = 440$, room temperature, final $R = 0.047$ for 1679 unique observed reflections. The amine groups are planar with the five-membered ring of 1,2,4-triazole, the dihedral angles between cations and anions are 172.9° and the nitro groups are rotated 4.2° out of the plane of the triazolone. The proton is linked not at the amine group but at the 4-position of 3-amino-1,2,4-triazole, and the charge of the anions is mainly concentrated at the N_4 atom of 3-nitro-1,2,4-triazol-5-one. All H atoms except those on the C atom are involved in hydrogen bonds.

Experimental. The title compound (3ATNTO) was prepared by adding 3-amino-1,2,4-triazole to 3-nitro-1,2,4-triazol-5-one (NTO) dissolved in water. Yellow needle crystals for X-ray diffraction were crystallized from water solution. Dimensions 0.4 × 0.2 × 0.2 mm, automated Nicolet *R3m* diffractometer, Mo $K\alpha$ radiation monochromated by a graphite crystal, room temperature, θ–2θ scan, scan range $[2\theta(\alpha_1) - 1°] - [2\theta(\alpha_2) + 1°]$, variable scan speed, 7–29° min^{-1}. 20 centered reflections ($12 < 2\theta < 27°$) used for determining lattice parameters. No absorption corrections. Max. $(\sin\theta)/\lambda = 0.60$ Å^{-1}. Index range $0 < h < 8$, $0 < k < 23$, $-9 < l < 9$. 1679 reflections were collected of which 1446 were observed [$I/\sigma(I) > 2$]. Standard reflections $4\bar{2}\bar{2}$ and $2\bar{8}1$ showed no significant variation. The structure was solved by direct methods with the program *SHELXTL* (Sheldrick, 1981). H atoms were located in difference maps. 160 parameters were refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for H atoms; a maximum of 103 parameters refined each least-squares cycle with a subset of coordinates in each cycle. $R = 0.047$, unit weight, $(\Delta/\sigma)_{max} = 0.16 \times 10^{-4}$. Final difference Fourier synthesis $-0.34 < \Delta\rho < 0.84$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Atomic coordinates and isotropic thermal parameters are given in Table 1.* Bond lengths and angles

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54976 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$)

	x	y	z	U
Anion				
C(1)	5074 (4)	−470 (1)	−2622 (4)	27 (1)*
C(2)	7308 (4)	231 (1)	−1401 (4)	28 (1)*
N(1)	7996 (3)	−447 (1)	−1238 (3)	31 (1)*
N(2)	6545 (3)	−904 (1)	−2024 (3)	31 (1)*
N(3)	5386 (3)	216 (1)	−2324 (3)	29 (1)*
N(4)	3165 (3)	−739 (1)	−3550 (3)	33 (1)*
O(1)	8302 (3)	761 (1)	−785 (3)	38 (1)*
O(2)	1786 (3)	−325 (1)	−3978 (3)	46 (1)*
O(3)	3055 (3)	−1374 (1)	−3867 (3)	52 (1)*
H(1)	10825 (49)	607 (16)	543 (43)	59 (9)
Cation				
C(3)	4419 (4)	2058 (1)	−2380 (4)	31 (1)*
C(4)	1398 (4)	1692 (2)	−3510 (4)	39 (1)*
N(5)	1245 (4)	2367 (1)	−3588 (4)	41 (1)*
N(6)	3179 (4)	2593 (1)	−2872 (4)	35 (1)*
N(7)	3301 (3)	1475 (1)	−2781 (3)	34 (1)*
N(8)	6345 (4)	2076 (1)	−1641 (4)	46 (1)*
H(2)	3522 (59)	3010 (19)	−2864 (51)	73 (12)
H(3)	3782 (52)	1023 (16)	−2658 (45)	66 (10)
H(4)	429 (48)	1348 (16)	−4006 (43)	46 (9)
H(5)	7081 (61)	2454 (21)	−1333 (53)	78 (12)
H(6)	6930 (59)	1752 (20)	−1130 (52)	66 (12)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and angles* (°)

Anion		Cation	
C(1)—N(2)	1.309 (3)	C(3)—N(6)	1.329 (3)
C(1)—N(3)	1.337 (3)	C(3)—N(7)	1.346 (3)
C(1)—N(4)	1.448 (3)	C(3)—N(8)	1.320 (4)
C(2)—N(1)	1.370 (3)	C(4)—N(5)	1.291 (4)
C(2)—N(3)	1.360 (3)	C(4)—N(7)	1.367 (4)
C(2)—O(1)	1.256 (3)	C(4)—H(4)	0.956 (31)
N(4)—O(2)	1.217 (3)	N(5)—N(6)	1.388 (3)
N(4)—O(3)	1.229 (3)	N(6)—H(2)	0.827 (36)
N(1)—N(2)	1.365 (3)	N(7)—H(3)	0.918 (32)
N(1)—H(1)	0.924 (30)	N(8)—H(5)	0.883 (38)
		N(8)—H(6)	0.792 (37)
N(2)—C(1)—N(3)	118.1 (2)	N(6)—C(3)—N(7)	105.7 (2)
N(2)—C(1)—N(4)	120.0 (2)	N(6)—C(3)—N(8)	128.4 (2)
N(3)—C(1)—N(4)	121.9 (2)	N(7)—C(3)—N(8)	125.9 (2)
N(1)—C(2)—N(3)	107.7 (2)	N(5)—C(4)—N(7)	112.5 (2)
N(1)—C(2)—O(1)	125.0 (2)	N(5)—C(4)—H(4)	128.6 (18)
N(3)—C(2)—O(1)	127.3 (2)	N(7)—C(4)—H(4)	118.7 (18)
C(2)—N(1)—N(2)	110.8 (2)	C(4)—N(5)—N(6)	103.2 (2)
C(2)—N(1)—H(1)	127.6 (2)	C(3)—N(6)—N(5)	111.9 (2)
N(2)—N(1)—H(1)	121.0 (2)	N(5)—N(6)—H(2)	122.7 (26)
C(1)—N(2)—N(1)	101.0 (2)	C(3)—N(6)—H(2)	125.2 (26)
C(1)—N(3)—C(2)	102.4 (2)	C(3)—N(7)—C(4)	106.8 (2)
C(1)—N(4)—O(2)	118.2 (2)	C(3)—N(7)—H(3)	125.4 (21)
C(1)—N(4)—O(3)	117.5 (2)	C(4)—N(7)—H(3)	127.7 (21)
O(2)—N(4)—O(3)	124.3 (2)	C(3)—N(8)—H(5)	126.8 (26)
		C(3)—N(8)—H(6)	124.0 (28)
		H(5)—N(8)—H(6)	107.3 (37)

X—H···Y	X···Y (Å)	H···Y (Å)	X—H···Y (°)
N(1)—H(1)···O(1)	2.330 (3)	1.84 (3)	111 (2)
N(6)—H(2)···N(2)^i	2.874 (3)	2.07 (3)	163 (2)
N(7)—H(3)···N(3)	2.767 (3)	1.87 (3)	166 (2)
N(8)—H(5)···O(3)^i	2.997 (3)	2.24 (4)	144 (3)
N(8)—H(6)···O(1)	2.854 (3)	2.10 (4)	160 (3)

Symmetry code: (i) $-x, -y, -1-z$.

are listed in Table 2. Fig. 1 is an *ORTEP* (Johnson, 1965) drawing of the 3AT cation and the NTO anion and shows the atom-numbering scheme. A stereo-view of the structure is shown in Fig. 2.

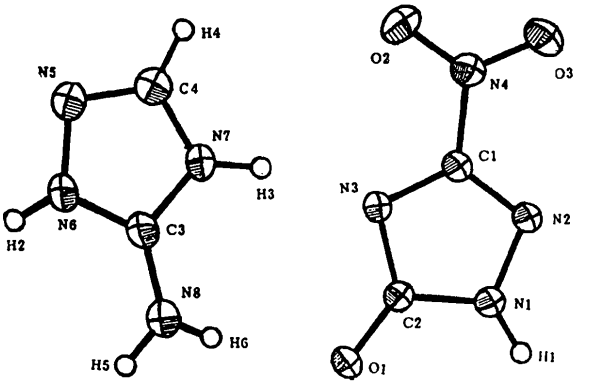

Fig. 1. Thermal-ellipsoid plot of the asymmetric unit, showing the atom-numbering scheme.

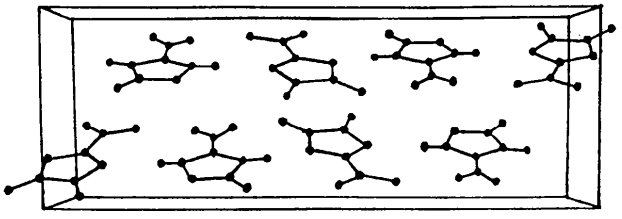

Fig. 2. A packing view of the structure with the *a* axis out of the page.

Related literature. NTO is a good insensitive explosive (Lee & Coburn, 1985) and an ideal ingredient of composite explosives (Becuwe & Delclos, 1987). Its salts, including many inorganic salts (Pevzner, 1966; Chen & Li, 1988) and some organic salts (Lee & Stinecipher, 1989; Chen, Ou & Li, 1988), are currently being studied. Structures of the 3-nitro-1,2,4-triazol-5-one anion include those of the ethyldiammonium salt (Cromer, Hall, Lee & Ryan, 1988*a*), the lead salt (Chen, Ou, Li, Fan & Chui, 1990), the diaminoguanidinium salt (Cromer, Hall, Lee & Ryan, 1988*b*) and the ammonium salt (Li, Chen, Ou & Zhou, 1990). No structure of the 3-amino-1,2,4-triazole cation has been reported in the literature. The structure of 3-amino-1,2-4-triazole itself was given by Makarskii, Starova, Frank-Kamenetskaya, Lopyrev & Voronkov (1977) and Starova, Frank-Kamenetskaya, Makarskii & Lopyrev (1978). The only example, with the proton attached not at the side-chain group but at the five-membered ring of the substituted 1,2,4,-triazoles, is 3-azido-1,2,4-triazole (Pevzner, Martynova & Timofeeva, 1974).

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