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,

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* 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.

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, (1988a,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.

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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}, $\lambda(\text{Mo } K\alpha) = 0.71069$ 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

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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 $-6 \leq h \leq 6$, $0 \leq k \leq 15$, $0 \leq l \leq 21$. 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;

1·3 (3) × 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 (129 parameters). Final $R = 0.030$, $wR = 0.036$, $S = 1.47$, max. $\Delta/\sigma = 0.04$. Final ΔF Fourier synthesis $-0.14 < \Delta\rho < 0.19$ 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 to show the network of hydrogen bonds. 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. The bond lengths and angles of the anion ring are virtually identical to those in both the hydrated (Cromer & Storm, 1991) and anhydrous (Garcia & Lee, 1992) forms of this substituted triazole except as noted below. The nitro group is twisted out of plane by 7·6° and the amino group by 13·8°, more than twice as much as in the hydrated crystal. The C(2)—N(5) bond to the amino group of

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* Corrected structure factor $F^* = F_c/(1 + 0.002\chi F_c^2/\sin2h)^{1/4}$.

† Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54113 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
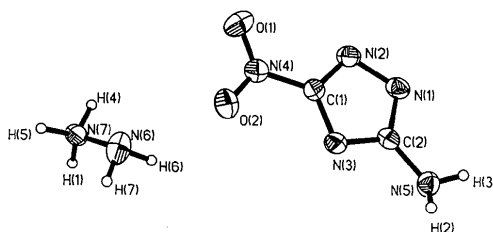

Fig. 1. Drawing of title compound showing the atom-numbering scheme. Thermal ellipsoids are 50% probability with H atoms of arbitrary size.
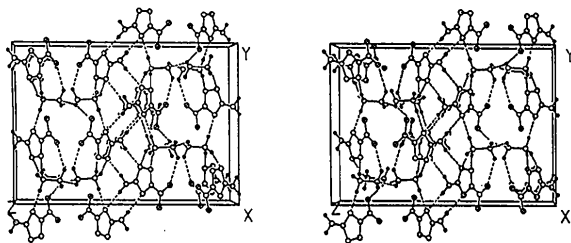

Fig. 2. Stero drawing to show the ribbon structure. Hydrogen bonds are dotted. The view is along the a axis with c vertical and b horizontal. The origin is at the upper left rear.

Table 1. *Positional parameters* (× 10⁴) *and equivalent isotropic thermal parameters* (Å² × 10²) *for the hydrazinium salt of* 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)	−1917 (4)	6114 (2)	3779 (1)	2·6 (1)
C(2)	1145 (4)	6269 (2)	4431 (1)	2·5 (1)
N(1)	0474 (3)	7204 (1)	4239 (1)	3·1 (1)
N(2)	−1584 (3)	7087 (1)	3798 (1)	3·1 (1)
N(3)	−0308 (3)	5548 (1)	4153 (1)	2·8 (1)
N(4)	−3996 (4)	5666 (2)	3414 (1)	3·5 (1)
N(5)	3212 (4)	6072 (1)	4843 (1)	3·2 (1)
O(1)	−5346 (4)	6203 (1)	3042 (1)	5·2 (1)
O(2)	−4338 (3)	4760 (1)	3501 (1)	4·8 (1)
N(6)	−3862 (4)	3549 (2)	2090 (1)	4·6 (1)
N(7)	−4344 (4)	3492 (1)	1320 (1)	3·0 (1)

Table 2. *Bond distances* (Å) *and angles* (°) *involving* C, N *and* O, *and hydrogen-bond geometry* (Å, °) *in the hydrazinium salt of* 3-amino-5-nitro-1,2,4-triazole

C(1)—N(2)	1·309 (3)	C(2)—N(5)	1·376 (3)
C(1)—N(3)	1·341 (3)	N(1)—N(2)	1·386 (3)
C(1)—N(4)	1·438 (3)	N(4)—O(1)	1·230 (3)
C(2)—N(1)	1·344 (3)	N(4)—O(2)	1·231 (3)
C(2)—N(3)	1·341 (3)	N(6)—N(7)	1·450 (3)

N(2)—C(1)—N(3)	117·0 (2)	C(2)—N(1)—N(2)	105·5 (2)
N(2)—C(1)—N(4)	122·2 (2)	C(1)—N(2)—N(1)	103·7 (2)
N(3)—C(1)—N(4)	120·9 (2)	C(1)—N(3)—C(2)	100·0 (2)
N(1)—C(2)—N(3)	113·9 (2)	C(1)—N(4)—O(1)	118·8 (2)
N(1)—C(2)—N(5)	122·8 (2)	C(1)—N(4)—O(2)	117·5 (2)
N(3)—C(2)—N(5)	123·2 (2)	O(1)—N(4)—O(2)	123·7 (2)

X—H···Y	d(X—Y)	d(H···Y)	∠X—H···Y
N(5)—H(3)···N(1ⁱ)	3·105 (3)	2·22 (2)	172 (2)
N(5)—H(2)···N(3ⁱⁱ)	3·247 (3)	2·49 (3)	150 (2)
N(6)—H(6)···O(2ⁱⁱⁱ)	3·121 (3)	2·36 (4)	139 (3)
N(6)—H(6)···N(6ⁱⁱⁱ)	3·092 (3)	2·36 (4)	136 (3)
N(6)—H(7)···O(1ⁱᵛ)	3·163 (4)	2·40 (4)	137 (3)
N(7)—H(1)···N(1ᵛ)	2·891 (3)	2·00 (3)	162 (2)
N(7)—H(4)···N(3ᵛⁱ)	2·921 (3)	1·98 (3)	173 (3)
N(7)—H(5)···N(2ⁱᵛ)	2·893 (3)	1·99 (3)	169 (2)

Symmetry code: (i) $\frac{1}{2}+x$, $\frac{3}{2}-y$, $1-z$; (ii) $-x$, $1-y$, $1-z$; (iii) $\frac{1}{2}+x$, y, $\frac{1}{2}-z$; (iv) $-1-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$; (v) $-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$; (vi) $-\frac{1}{2}+x$, y, $\frac{1}{2}-z$.

the anion is 0·035–0·040 Å longer and the N(1)—N(2) bond is 0·013–0·020 Å longer than in the neutral molecules. These longer bonds could be caused by a repulsion between the amino lone pair and the electron-rich ring and by repulsion between the lone electron pair on N(2) and the formal negative charge on N(1). The two H atoms of the amino group on the ring form H bonds to two different triazole anions. One of the H atoms on N(6) bonds to an anion and the other one to another cation. The three H atoms on N(7) form shorter, nearly linear hydrogen bonds to three different triazole anions. These stronger bonds are between the electronegative ring N atoms and the positive end of the hydrazinium cation. In spite of this hydrogen-bond network the compound slowly decomposes by loss of hydrazine at room temperature.

Related literature. See Cromer, Hall, Lee & Ryan (1988) for further triazole and small explosive molecule references.

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Structure of 4,5-Di-*O*-acetyl-6-*S*-acetyl-2,3-di-*S*-ethyl-2,3,6-trithio-D-allose Diethyl Dithioacetal

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Abstract. $C_{20}H_{36}O_5S_5$, $M_r = 516.82$, monoclinic, $P2_1$, $a = 8.670$ (1), $b = 17.149$ (1), $c = 9.224$ (1) Å, $\beta = 95.88$ (1)°, $V = 1364.2$ (3) Å^3, $Z = 2$, $D_m = 1.26$, $D_x = 1.27$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 40.8$ cm^{-1} $F(000) = 552.0$, $T = 296$ K, final $R = 0.066$, $wR = 0.065$ for 3478 observed reflections. The purpose of the structure determination was to establish the position of the substituents and the molecular conformation of the title compound (2) obtained by acid catalyzed ethanethiolysis of 3,5-di-*O*-acetyl-6-*S*-acetyl-1,2-isopropylidene-6-thio-α-D-glucofuranose (1). Since the product (2) was planned as a starting synthone in a sequence of reactions, its stereochemistry was of essential importance to us.

(1)   (2)
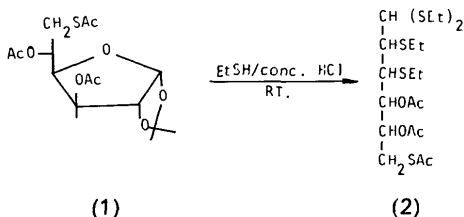
Experimental. The compound (2) was isolated as the only reaction product in almost quantitative yield. A colourless plate-like crystal, $0.16 \times 0.24 \times 0.44$ mm, was mounted on an Enraf–Nonius CAD-4 diffractometer, and data collected with monochromated Cu $K\alpha$ radiation and $\omega/2\theta$ scans. 4650 reflections were measured ($-10 \le h \le 10$; $-20 \le k \le 20$; $0 \le l \le 10$) to $2\theta_{max} = 60°$. Three check reflections showed no significant intensity variation. 4285 unique reflections ($R_{int} = 0.019$) with $I > 3\sigma(I)$ were used for structure solution. Data were corrected for Lorentz and polarization effects, as well as for absorption (*DIFABS*; Walker & Stuart, 1983), maximum and minimum transmission 1.00 and 0.83. Cell constants were refined from $\pm \theta$ values of 25 reflections in the range 11–21°. An extinction correction was not necessary. Structure solution was by direct methods (Sheldrick, 1986), and refinement was by the full-matrix least-squares method based on F; $w = 1/\sigma^2$ (Sheldrick, 1976). Final $R = 0.066$, $wR = 0.065$ for 3478 observations and 272 variables in the last cycle. All non-H atoms were anisotropically refined; H