N(7)—C(8). Its value is 104·2 (3)° in the neutral guanine molecule and 108·0 (2)° in the protonated molecule.

Del Bene (1983) optimized the geometry of both the neutral and the protonated guanine molecules and calculated the protonation energies for four different protonation sites. Del Bene came to the same conclusions as Taylor & Kennard (1982), *i.e.* the most favourable site is N(7) with C(5)—N(7)—C(18) angles of 104·0 and 109·1°, respectively.

All bond distances and angles presented in this work are in agreement with the above results, *i.e.* the value of the angle C(5)—N(7)—C(8) is 108·3 (1)°.

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

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Abstract. $C_2H_3N_5O_2.H_2O$, $M_r = 147·09$, monoclinic, $P2_1/c$, $a = 11·129$ (3), $b = 15·227$ (3), $c = 7·130$ (2) Å, $\beta = 101·29$ (2)°, $V = 1184·88$ Å^3, $Z = 8$, $D_x = 1·649$ g cm^{-3}, $\lambda(\text{Mo } K\alpha_1) = 0·70926$ Å, $\mu = 1·4$ cm^{-1}, $F(000) = 608$, room temperature, final $R = 0·028$ for the 989 observed reflections with $I > 3\sigma(I)$ from 1540 independent reflections. There are two formula units in the asymmetric unit. The geometries of the two molecules are very similar. Rings are planar within 0·006 Å. All H atoms take part in hydrogen bonds.

Experimental. Pale straw-colored crystals of the title compound were prepared according to Hartman & Silloway (1955) and grown from water. Selected crystal *ca* 0·20 × 0·15 × 0·10 mm, 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 $\frac{1}{6}$ of scan, graphite-monochromated Mo $K\alpha$ radiation. Unit cell from 25 reflections with $10 < \theta < 18°$. No absorption corrections. $(\sin \theta)/\lambda_{max} = 0·540$ Å^{-1}. Index range $-12 \le h \le 12$, $0 \le k \le 16$, $-7 \le l \le 7$, 3070 reflections measured and averaged to yield 1540 unique reflections of which 989 were observed with $I > 3\sigma(I)$, $R_{int} = 0·012$. Standard reflections ($\bar{1}18$ and $40\bar{4}$) showed no significant variation. Least squares

minimized $\Sigma w(\Delta F)^2$ with $w = [\sigma_c^2(F) + 0·015F^2]^{-1}$, $\sigma_c^2(F)$ based on counting statistics. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971). Scale factor, extinction parameter [2·9 (3) × 10^{-6} mm; Larson, 1969], positional parameters, anisotropic thermal parameters for C, N, O, and isotropic thermal parameters for H atoms were refined (222 parameters). Final $R = 0·028$, $wR = 0·030$, $S = 2·0$, max. $\Delta/\sigma = 0·0004$. Final ΔF Fourier synthesis $-0·17 < \Delta\rho < 0·13$ 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). Calculations on CDC-7600 using the Los Alamos Crystal Structure System developed primarily by A. C. Larson.

Fig. 1. is an *ORTEP* drawing (Johnson, 1965) to show the atom-numbering scheme of molecule (1). Final parameters are given in Table 1.‡ Bond lengths and angles are given in Table 2. Nitro-group parameters are in the range of values found in other compounds. In molecule (1) the nitro group is twisted out of plane by 4·3 (1)° and the amino group by 2·1 (8)°. In molecule (2) these twists are 2·2 (1)

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‡ Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54111 (11 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. *Final least-squares positional parameters for C, N and O ($\times 10^4$) and equivalent isotropic U values ($\times 10^2$) in 3-amino-5-nitro-1,2,4-triazole hydrate*

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

|	x	y	z	$U_{eq}(\text{Å}^2)$
Molecule (1)				
C(1)	4772 (2)	0574 (2)	2256 (4)	3·1 (3)
C(2)	5639 (3)	−0594 (2)	3158 (4)	3·1 (3)
N(1)	4532 (2)	−0765 (2)	2034 (3)	3·3 (3)
N(2)	3946 (2)	0009 (1)	1449 (3)	3·4 (3)
N(3)	5820 (2)	0273 (1)	3317 (3)	3·0 (3)
N(4)	4529 (2)	1502 (2)	1988 (4)	4·0 (3)
N(5)	6413 (3)	−1208 (2)	4015 (4)	4·3 (3)
O(1)	3592 (2)	1732 (1)	0913 (3)	6·5 (3)
O(2)	5266 (2)	2016 (1)	2858 (4)	6·3 (3)
Molecule (2)				
C(3)	0310 (2)	1228 (2)	2644 (4)	3·2 (3)
C(4)	−0613 (3)	0062 (2)	1917 (4)	3·1 (3)
N(6)	0473 (2)	−0100 (2)	3074 (3)	3·4 (3)
N(7)	1090 (2)	0668 (2)	3566 (3)	3·6 (3)
N(8)	−0740 (2)	0926 (1)	1604 (3)	3·3 (3)
N(9)	0601 (3)	2152 (2)	2742 (4)	4·4 (3)
N(10)	−1418 (3)	−0558 (2)	1182 (4)	4·3 (3)
O(3)	1560 (2)	2388 (2)	3746 (3)	6·7 (3)
O(4)	−0126 (2)	2659 (1)	1806 (3)	6·3 (3)
Water 1				
O(5)	7425 (2)	3121 (2)	3992 (3)	4·5 (3)
Water 2				
O(6)	7798 (2)	3646 (1)	0367 (4)	4·1 (3)
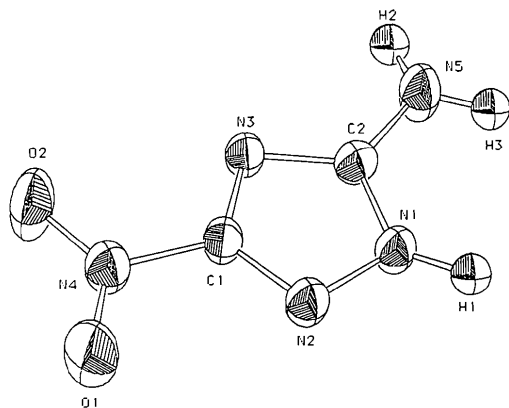
Fig. 1. *ORTEP* (Johnson, 1965) drawing of molecule (1) to show atom-numbering scheme. Thermal ellipsoids are 30% probability. H atoms are of arbitary size.

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

	Molecule (1)	Molecule (2)
C(1)—N(2)	1·307 (3)	1·300 (3)
C(1)—N(3)	1·340 (3)	1·337 (3)
C(1)—N(4)	1·445 (3)	1·443 (4)
C(2)—N(1)	1·356 (3)	1·347 (3)
C(2)—N(3)	1·338 (3)	1·337 (3)
C(2)—N(5)	1·335 (4)	1·336 (4)
N(1)—N(2)	1·373 (3)	1·366 (3)
N(4)—O(1)	1·218 (3)	1·217 (3)
N(4)—O(2)	1·213 (3)	1·218 (3)
N(2)—C(1)—N(3)	118·9 (2)	118·7 (3)
N(2)—C(1)—N(4)	119·2 (2)	119·6 (3)
N(3)—C(1)—N(4)	121·9 (2)	121·7 (3)
N(1)—C(2)—N(3)	110·1 (2)	109·6 (3)
N(1)—C(2)—N(5)	124·4 (3)	124·3 (3)
N(3)—C(2)—N(5)	125·4 (3)	126·1 (3)
C(2)—N(1)—N(2)	109·7 (2)	110·2 (2)
C(1)—N(2)—N(1)	100·4 (2)	100·3 (2)
C(1)—N(3)—C(2)	100·9 (2)	101·1 (2)
C(1)—N(4)—O(1)	118·7 (2)	118·6 (3)
C(1)—N(4)—O(2)	118·2 (2)	118·2 (2)
O(1)—N(4)—O(2)	123·1 (3)	123·2 (3)

X—H⋯Y	d(X—Y)	d(H⋯Y)	∠X—H⋯Y
N(1)—H(1)⋯O(5')	2·744 (3)	1·86 (3)	152 (3)
N(5)—H(2)⋯O(1')	3·137 (3)	2·31 (3)	157 (3)
N(5)—H(3)⋯N(7'')	3·082 (4)	2·21 (3)	172 (3)
N(6)—H(4)⋯O(6')	2·784 (3)	1·97 (3)	157 (2)
N(10)—H(5)⋯N(2''')	3·173 (4)	2·41 (3)	174 (3)
N(10)—H(6)⋯O(3'')	3·133 (4)	2·22 (4)	152 (2)
O(5)—H(7)⋯N(8')	2·873 (3)	2·00 (4)	172 (3)
O(5)—H(8)⋯O(6'')	2·865 (3)	2·03 (4)	165 (3)
O(6)—H(9)⋯N(3''')	2·904 (3)	2·12 (3)	177 (3)
O(6)—H(10)⋯O(5'')	2·813 (4)	1·91 (4)	177 (3)

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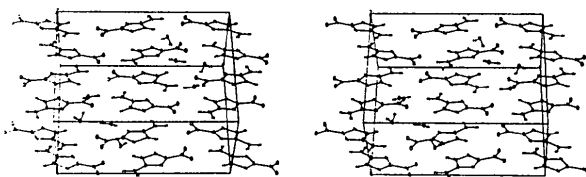

Fig. 2. Stereo drawing of 3-amino-5-nitro-1,2,4-triazole hydrate. Hydrogen bonds are dotted. Origin is at the lower, left rear, with y horizontal and x vertical.

and 1·7 (6)°. The ring bonds are very similar to those in the anhydrous crystal of this molecule (Garcia & Lee, 1991) and in the hydrazinium salt (Garcia, Lee & Storm, 1991). The ring normals of molecules (1) and (2) are at an angle of 4·2 (1)°. Hydrogen bonds are given in Table 2. Both molecules, as well as the water O atoms, lie close to the 20$\bar{2}$ planes. The molecules in the planes are linked by N—H⋯N, N—H⋯O(nitrate) and N—H⋯O(water)—H⋯N hydrogen bonds. The layers are linked by hydrogen bonds between water molecules. Fig. 2 is a stereo drawing showing these hydrogen-bonded layers.

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

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N,N',N'',N'''-Bis(m-xylyl)bis(4,13-diaza-1,7,10-trioxacyclopentadecane): a Multidentate Macrocyclic Complexing Agent Containing Two 15-Crown-5 Rings

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Abstract. $C_{36}H_{56}N_4O_6$, $M_r = 640.84$, orthorhombic, $Pbca$, $a = 22.460$ (9), $b = 17.520$ (8), $c = 9.04$ (1) Å, $V = 3557.2$ Å^3, $Z = 4$, $D_x = 1.20$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 0.76$ cm^{-1}, room temperature, $R = 0.073$ for 970 reflections with $F > 6\sigma(F)$. The molecule has symmetry $\bar{1}$ (i). In the crystal the central cavity of the whole molecule, bounded by two 15-membered rings, is nearly closed and could not accommodate a cation; a pair of C atoms come within 2·3 Å of the inversion centre. All the O atoms point towards the centres of their 15-membered rings. Substantial conformational change must occur when this multidentate chelating agent forms a complex with a cation.

Experimental. The title compound was prepared in 30% yield from α,α-dibromo-m-xylene and 1,10-di-aza-4,7,13-trioxacyclopentadecane, in a solution of K_2CO_3 and MeCN, and recrystallized from dichloromethane and heptane. Crystals were provided by Professor S. Mageswaren and Professor I. O. Sutherland, Liverpool University.

Crystal 0·28 × 0·28 × 0·12 mm, preliminary oscillation and Weissenberg photographs gave unit cell and space group. Stoe Stadi-2 diffractometer, graphite monochromator, a and b parameters refined from 12 reflections with $14 \le 2\theta \le 24°$, Layers $l = 0–8$, with h 0–24, k 0–18, $2\theta_{max} = 50°$, recorded using ω scans, one standard reflection per layer, no significant variations in intensity. 2791 measured reflections, 2335 independent reflections, R_{int} 0·02. Lorentz and polarization corrections. Structure solved by direct methods (*SHELX*86, Sheldrick, 1986) and difference Fourier maps. Least-squares refinement, on F, using 970 reflections with $F > 6\sigma(F)$, since data was rather weak. Most H atoms located in difference Fourier maps; all H atoms, placed at calculated positions, included in F with $U = 0.05$ Å^2, but not refined. Anisotropic vibration

Table 1. *Fractional atomic coordinates with e.s.d.'s in parentheses*

$$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	U_{eq}(Å^2)
N(1)	0·1328 (3)	0·1506 (4)	0·1782 (6)	0·0471 (46)
N(2)	−0·1172 (3)	0·0286 (3)	0·2508 (7)	0·0408 (44)
O(1)	0·0155 (3)	0·2276 (3)	0·1560 (7)	0·0597 (41)
O(2)	−0·1060 (2)	0·1998 (3)	0·1466 (6)	0·0581 (41)
O(3)	0·0446 (3)	0·0435 (3)	0·3086 (6)	0·0435 (39)
C(1)	0·1026 (3)	0·1999 (4)	0·2855 (8)	0·0480 (58)
C(2)	0·0658 (4)	0·2615 (5)	0·2194 (10)	0·0706 (71)
C(3)	−0·0254 (4)	0·2783 (5)	0·0952 (11)	0·0517 (69)
C(4)	−0·0758 (4)	0·2347 (5)	0·0284 (9)	0·0578 (67)
C(5)	−0·1457 (4)	0·1415 (5)	0·0992 (10)	0·0715 (75)
C(6)	−0·1611 (3)	0·0884 (4)	0·2247 (9)	0·0499 (58)
C(7)	−0·0609 (3)	0·0564 (4)	0·3148 (8)	0·0508 (60)
C(8)	−0·0094 (4)	0·0135 (5)	0·2541 (10)	0·0663 (75)
C(9)	0·0942 (4)	0·0199 (4)	0·2228 (10)	0·0543 (67)
C(10)	0·1446 (3)	0·0742 (4)	0·2392 (9)	0·0457 (61)
C(11)	0·1866 (3)	0·1866 (4)	0·1197 (8)	0·0325 (50)
C(12)	0·2052 (2)	0·1537 (2)	−0·0262 (5)	0·0389 (21)
C(13)	0·2619 (2)	0·1716 (2)	−0·0788 (5)	0·0463 (22)
C(14)	0·2807 (2)	0·1440 (2)	−0·2158 (5)	0·0535 (24)
C(15)	0·2427 (2)	0·0985 (2)	−0·3003 (5)	0·0508 (23)
C(16)	0·1860 (2)	0·0805 (2)	−0·2477 (5)	0·0413 (20)
C(17)	0·1673 (2)	0·1081 (2)	−0·1107 (5)	0·0402 (22)
C(18)	−0·1411 (3)	−0·0315 (4)	0·3399 (8)	0·0778 (65)

Table 2. *Selected molecular geometry* (*in* Å *and* °)

Mean bond lengths

C—C	(not aromatic)	1·490 (6)
C—N		1·459 (12)
C—O		1·407 (17)

Torsion angles

N(1)—C(1)—C(2)—O(1)	71·4 (8)
N(2)—C(7)—C(8)—O(3)	176·3 (6)
O(1)—C(3)—C(4)—O(2)	67·1 (8)
O(2)—C(5)—C(6)—N(2)	−82·9 (8)
O(3)—C(9)—C(10)—N(1)	−65·7 (8)
C(1)—C(2)—O(1)—C(3)	176·9 (7)
C(3)—C(4)—O(2)—C(5)	−165·2 (7)
C(4)—C(3)—O(1)—C(2)	178·6 (7)
C(5)—C(6)—N(2)—C(7)	70·8 (8)
C(6)—C(5)—O(2)—C(4)	160·7 (7)
C(7)—C(8)—O(3)—C(9)	−162·9 (6)
C(8)—C(7)—N(2)—C(6)	−143·6 (7)
C(9)—C(10)—N(1)—C(1)	85·8 (8)
C(10)—C(9)—O(3)—C(8)	156·6 (7)
C(10)—N(1)—C(1)—C(2)	−153·9 (7)