Table 2. *Selected bond distances* (Å), *bond angles* (°), *torsion angles* (°) *and hydrogen bonds*

	(I)	(II)		(I)	(II)
O(1)—C(9)	1·239 (4)	1·224 (5)	C(5)—C(6)	1·383 (7)	1·380 (6)
N(1)—C(7)	1·481 (4)	1·485 (5)	C(6)—C(7)	1·512 (6)	1·530 (6)
N(1)—C(8)	1·482 (5)	1·463 (5)	C(7)—C(17)	1·516 (7)	1·520 (6)
N(1)—C(9)	1·323 (4)	1·335 (5)	C(9)—C(10)	1·507 (4)	1·513 (5)
N(2)—C(14)	1·333 (6)	1·350 (7)	C(10)—C(11)	1·389 (5)	1·391 (6)
N(2)—C(15)	1·349 (5)	1·341 (6)	C(10)—C(15)	1·376 (5)	1·377 (5)
C(1)—C(2)	1·401 (9)	1·389 (8)	C(11)—C(12)	1·495 (6)	1·495 (7)
C(1)—C(6)	1·373 (6)	1·377 (6)	C(11)—C(13)	1·398 (6)	1·387 (6)
C(2)—C(3)	1·334 (11)	1·380 (9)	C(13)—C(14)	1·356 (7)	1·351 (7)
C(3)—C(4)	1·337 (10)	1·364 (9)	C(15)—C(16)	1·490 (6)	1·495 (7)
C(4)—C(5)	1·391 (10)	1·384 (8)			

	(I)	(II)		(I)	(II)
C(7)—N(1)—C(8)	116·9 (3)	117·7 (3)	N(1)—C(7)—C(17)	109·2 (3)	109·1 (4)
C(7)—N(1)—C(9)	119·1 (3)	118·1 (3)	C(6)—C(7)—C(17)	115·8 (3)	115·6 (3)
C(8)—N(1)—C(9)	123·6 (3)	123·6 (3)	O(1)—C(9)—N(1)	124·0 (3)	124·1 (4)
C(14)—N(2)—C(15)	122·6 (4)	122·6 (4)	O(1)—C(9)—C(10)	116·6 (3)	116·9 (3)
C(2)—C(1)—C(6)	120·1 (5)	120·7 (5)	N(1)—C(9)—C(10)	119·3 (3)	119·1 (3)
C(1)—C(2)—C(3)	121·8 (5)	120·0 (5)	C(9)—C(10)—C(11)	119·7 (3)	119·3 (3)
C(2)—C(3)—C(4)	118·3 (7)	120·0 (5)	C(9)—C(10)—C(15)	119·1 (3)	119·5 (3)
C(3)—C(4)—C(5)	122·3 (7)	119·5 (5)	C(11)—C(10)—C(15)	120·9 (3)	120·5 (3)
C(4)—C(5)—C(6)	119·7 (6)	121·7 (5)	C(10)—C(11)—C(12)	121·4 (4)	121·3 (4)
C(1)—C(6)—C(5)	117·5 (4)	118·1 (4)	C(10)—C(11)—C(13)	117·9 (4)	117·6 (4)
C(1)—C(6)—C(7)	123·9 (4)	122·0 (3)	C(12)—C(11)—C(13)	120·7 (4)	121·0 (4)
C(5)—C(6)—C(7)	118·5 (4)	119·8 (3)	C(11)—C(13)—C(14)	119·6 (4)	121·2 (5)
N(1)—C(7)—C(6)	111·0 (3)	110·9 (3)	N(2)—C(15)—C(10)	118·2 (4)	118·8 (4)
			N(2)—C(14)—C(13)	120·7 (4)	119·2 (4)
			N(2)—C(15)—C(16)	117·4 (4)	117·0 (4)
			C(10)—C(15)—C(16)	124·4 (4)	124·2 (4)

	(I)	(II)
C(9)—N(1)—C(7)—C(6)	−134·1 (3)	−130·5 (3)
C(9)—N(1)—C(7)—C(17)	97·0 (4)	101·1 (4)
C(7)—N(1)—C(9)—O(1)	6·7 (6)	6·2 (6)
C(8)—N(1)—C(9)—O(1)	179·4 (7)	176·8 (4)
C(8)—N(1)—C(9)—C(10)	0·8 (6)	−2·1 (5)
C(1)—C(6)—C(7)—N(1)	−119·4 (5)	−135·8 (3)
O(1)—C(9)—C(10)—C(15)	88·6 (4)	90·8 (5)

Hydrogen-bond distances (Å) and angles (°); H atoms on calculated positions

N—H···Cl	Symmetry operation	N···Cl	N—H	H···Cl	N—H···Cl
N(2′)—H(201)···Cl(1)	(1−x, 0·5+y, 1−z)	3·021 (4)	0·98	2·10	157
N(2″)—H(202)···Cl(2)	(1−x, 0·5+y, −z)	3·037 (4)	0·98	2·06	175

(′) Indicates molecule I; (″) indicates molecule II.

Buck, Smeets, Kanters & Spek, 1988). The title compound represents a diastereorotamer of the previously published structure of 3-[*N*-methyl-*N*-(*R*)-α-methylbenzyl]carbamoyl-1,2,4-trimethylpyridinium iodide (Kanters, van der Steen, Bastiaansen & de Graaf, 1986). The carbonyl groups of these rotamers have an opposite orientation with respect to the pyridinium rings. The carbonyl and N—CH_3 groups are oriented *anti* in the title compound (Fig. 1) and *syn* in the diastereorotamer.

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Structure of the 1,3-Diaminoguanidinium Salt of 3-Nitro-1,2,4-triazol-5-one, $CH_8N_5^+.C_2HN_4O_3^-$[*]

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Abstract. $M_r = 219{\cdot}16$, triclinic, $P\bar{1}$, $a = 6{\cdot}735$ (2), $b = 6{\cdot}753$ (2), $c = 9{\cdot}844$ (2) Å, $\alpha = 88{\cdot}29$ (2), $\beta = 77{\cdot}17$ (2), $\gamma = 86{\cdot}50$ (2)°, $V = 435{\cdot}7$ Å^3, $Z = 2$, $D_x = 1{\cdot}671$ g cm^{-3}, λ(Mo $K\alpha_1$) = 0·70927 Å, $\mu = 1{\cdot}35$ cm^{-1}, $F(000) = 228$, room temperature, final $R = 0{\cdot}031$ for 1088 observed reflections [$I > 2\sigma(I)$] out of 1516 independent reflections. There is one formula unit in the asymmetric unit. The triazole ring is planar within 0·003 Å and the entire anion is planar within 0·04 Å. The nitro group is rotated 2·9 (2)° out of the

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Table 1. *Final least-squares parameters for the* C, N *and* O *atoms in the* 1,3-*diaminoguanidinium salt of* 3-*nitro*-1,2,4-*triazol*-5-*one* (*DAGNTO*) (*positional parameters* × 10^4, *equivalent isotropic* U × 10^2)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum U_{ij}.$$

	x	y	z	$U_{\rm eq}$(Å^2)
NTO anion				
C(1)	5092 (3)	2427 (3)	4150 (2)	2·5 (2)
C(2)	2478 (3)	3588 (3)	5521 (2)	2·7 (2)
N(1)	2244 (3)	3712 (3)	4176 (2)	3·1 (2)
N(2)	3944 (2)	2962 (3)	3275 (1)	3·1 (2)
N(3)	4351 (2)	2730 (2)	5506 (1)	2·8 (2)
N(4)	7142 (2)	1576 (2)	3646 (2)	3·3 (2)
O(1)	1162 (2)	4195 (2)	6576 (1)	3·5 (1)
O(2)	7739 (2)	1285 (3)	2398 (1)	5·0 (2)
O(3)	8174 (2)	1192 (2)	4508 (1)	4·6 (2)
1,3-Diaminoguanidinium cation				
C(3)	4907 (3)	2462 (3)	9065 (2)	2·9 (2)
N(5)	8126 (3)	1204 (4)	7895 (2)	4·2 (2)
N(6)	6165 (3)	2038 (3)	7865 (2)	3·7 (2)
N(7)	5562 (3)	2287 (3)	10233 (2)	3·9 (2)
N(8)	2985 (3)	3060 (3)	9084 (2)	4·0 (2)
N(9)	1605 (4)	3451 (5)	10350 (2)	5·5 (3)

Table 2. *Bond lengths* (Å) *and angles* (°) *for* C, N *and* O *atoms in the* 1,3-*diaminoguanidinium salt of* 3-*nitro*-1,2,4-*triazol*-5-*one* (*DAGNTO*)

C(1)—N(2)	1·309 (2)	N(2)—C(1)—N(3)	118·6 (2)
C(1)—N(3)	1·334 (2)	N(2)—C(1)—N(4)	120·4 (2)
C(1)—N(4)	1·449 (2)	N(3)—C(1)—N(4)	121·0 (2)
C(2)—N(1)	1·367 (2)	N(1)—C(2)—N(3)	107·7 (2)
C(2)—N(3)	1·353 (2)	N(1)—C(2)—O(1)	125·3 (2)
C(2)—O(1)	1·267 (2)	N(3)—C(2)—O(1)	127·0 (2)
N(1)—N(2)	1·363 (2)	C(2)—N(1)—N(2)	111·2 (2)
N(4)—O(2)	1·222 (2)	C(1)—N(2)—N(1)	100·3 (1)
N(4)—O(3)	1·224 (2)	C(1)—N(3)—C(2)	102·2 (1)
C(3)—N(6)	1·320 (2)	C(1)—N(4)—O(2)	118·7 (2)
C(3)—N(7)	1·320 (2)	C(1)—N(4)—O(3)	117·3 (2)
C(3)—N(8)	1·329 (2)	O(2)—N(4)—O(3)	124·0 (2)
N(5)—N(6)	1·410 (2)	N(6)—C(3)—N(7)	120·0 (2)
N(8)—N(9)	1·401 (3)	N(6)—C(3)—N(8)	119·5 (2)
		N(7)—C(3)—N(8)	120·6 (2)
		C(3)—N(6)—N(5)	117·9 (2)
		C(3)—N(8)—N(9)	120·4 (2)

Hydrogen bonds

X—H···Y	Symmetry operation on Y	$d(X-Y)$ (Å)	$d(\mathrm{H}\cdots Y)$ (Å)	$\angle X$—H···Y (°)
N(1)—H(1)···O(1)	$-x, 1-y, 1-z$	2·847 (2)	1·98 (2)	173 (2)
N(5)—H(2)···O(1)	$1+x, y, z$	3·019 (3)	2·19 (3)	172 (2)
N(6)—H(4)···N(3)	x, y, z	2·869 (2)	1·98 (2)	170 (2)
N(7)—H(5)···N(2)	$x, y, 1+z$	2·989 (2)	2·17 (2)	155 (2)
N(8)—H(7)···O(1)	x, y, z	3·051 (2)	2·21 (2)	168 (2)

ring plane. The remaining H on the anion is clearly in the 1 position. With the exception of the H atoms on the two terminal amino groups, the cation is approximately planar (within 0·09 Å) and coplanar with the anion.

Experimental. Title compound (DAGNTO) prepared by passing a solution of 1,3-diaminoguanidine hydrochloride through a column of Amberlite IRA-400 (OH form) to release the free DAG base and mixing it immediately with an equimolar aqueous solution of 3-nitro-1,2,4-triazol-5-one (NTO). Pale straw-colored crystals for X-ray diffraction crystallized from water. Selected crystal *ca* 0·20 × 0·20 × 0·06 mm. CAD-4 diffractometer, θ–2θ scan. Scan range $(0{\cdot}8 + 0{\cdot}34\tan\theta)^\circ$. Scan speed 1·2 to 5·5° min^{-1}. Background first and last 1/6 of scan. Graphite-monochromated Mo $K\alpha$ radiation. Unit cell, 25 reflections $10 < \theta < 20^\circ$. No absorption corrections. Max. $(\sin\theta)/\lambda = 0{\cdot}540$ Å^{-1}. Index range $-7 \le h \le 7$, $-8 \le k \le 8$, $-11 \le l \le 11$, 3032 reflections measured and averaged to yield 1516 unique reflections of which 1088 were observed with $I > 2\sigma(I)$, $R_{\rm int} = 0{\cdot}013$. Standard reflections 200 and 020 showed no significant variation. Least squares minimized $\sum w(\Delta F)^2$ with $w = [\sigma_c^2(F) + 0{\cdot}015F^2]^{-1}$, $\sigma_c^2(F)$ based on counting statistics. Structure was solved by *RANTAN* (Yao Jia-Xing, 1983) which is part of the *TEXRAY* structure analysis codes.* Scale factor, isotropic type-II extinction parameter [= 2·7 (2) × 10^{-6} mm] (Larson, 1969), positional parameters, anisotropic thermal parameters for C, N, O, and isotropic thermal parameters for H were refined. Final $R = 0{\cdot}031$, $wR = 0{\cdot}030$, $S = 1{\cdot}8$. Max. $\Delta/\sigma = 0{\cdot}004$. Final ΔF Fourier synthesis $-0{\cdot}18 < \Delta\rho < 0{\cdot}19$ 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). Calculations on CRAY-1 using the Los Alamos Crystal Structure System developed primarily by A. C. Larson.†

Fig. 1 is an *ORTEP* (Johnson, 1965) drawing of the two ionic groups and shows the atom-numbering

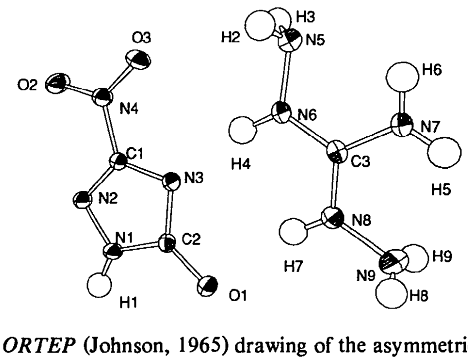

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the asymmetric unit to show atom-numbering scheme. Thermal ellipsoids are 30% probability. H atoms are arbitrarily sized.

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* Molecular Structure Corporation, 3304 Longmire Drive, College Station, TX 77840, USA.

† Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51255 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

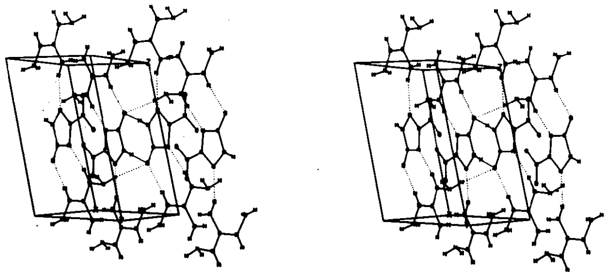

Fig. 2. Stereodrawing of the structure. The origin is at the lower right. Hydrogen bonds are dotted.

scheme. Final parameters are given in Table 1. Bond lengths and angles are given in Table 2. The anion H atom and four of the H atoms on the cation are involved in hydrogen bonds. A stereodrawing of the structure is shown in Fig. 2.

Related literature. The anion geometry is virtually identical to that found in the ethylenediammonium salt of NTO (Cromer, Hall, Lee & Ryan, 1988). The cation geometry is close to that found in the 1,2,3-triaminoguanidinium ion (Bracuti, Troup & Extine, 1986) and in the guanidinium ion (Baldwin, Denner, Egan & Markwell, 1986). The H atoms on the central amino group are directed to the lone-pair regions of the terminal amino groups.

NTO is a good, insensitive explosive (Lee & Coburn, 1985) and DAGNTO has about the same impact sensitivity. See Federov & Sheffield (1975) for a description of the impact sensitivity test. It is thought that extensive hydrogen bonding can contribute to impact insensitivity.

See Cromer *et al.* (1988) for further triazole and small explosive molecule references.

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Structures of Three *N*-Methylated 4-Hydroxyproline Derivatives

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Abstract. (I) $[C_6H_{12}NO_3][Cl]$, $M_r = 181{\cdot}6$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}099\,(1)$, $b = 10{\cdot}651\,(2)$, $c = 13{\cdot}692\,(1)$ Å, $V = 889\,(2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}37\,(1)$, $D_x = 1{\cdot}357$ Mg m^{-3}, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}341$ mm^{-1}, $F(000) = 384$, $T = 295\,(2)$ K, $R = 0{\cdot}053$ for 2027 reflections with $I \geq 2{\cdot}5(I)$; (II) *trans*-$[C_7H_{14}NO_3][Cl]$, $M_r = 195{\cdot}6$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}607\,(2)$, $b = 11{\cdot}079\,(2)$, $c = 12{\cdot}362\,(2)$ Å, $V = 905\,(2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}42\,(1)$, $D_x = 1{\cdot}436$ Mg m^{-3}, $\mu = 0{\cdot}338$ mm^{-1}, $F(000) = 416$, $R = 0{\cdot}035$ for 1835 reflections; (III) *cis*-$[C_7H_{14}NO_3][Cl]$, $M_r = 195{\cdot}6$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}031\,(2)$, $b = 10{\cdot}797\,(2)$, $c = 12{\cdot}708\,(1)$ Å, $V = 965\,(2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}34$, $D_x = 1{\cdot}346$ Mg m^{-3}, $\mu = 0{\cdot}317$ mm^{-1}, $F(000) = 416$, $R = 0{\cdot}051$ for 949 reflections. The crystal structures of (I) *N*-methyl- and (II) *N,N'*-dimethyl-4-hydroxy-L-proline and (III) *N,N'*-dimethyl-4-hydroxy-D-proline have been determined as their hydrochlorides. A *trans*

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