SHELX76 (Sheldrick, 1976). The refinement converged at $R = 0{\cdot}056$, $wR = 0{\cdot}048$, $w = 1{\cdot}4632[\sigma^2(F) + 0{\cdot}000086F^2]^{-1}$. 217 refined parameters; average shift/e.s.d. = 0·003; max. shift/e.s.d. < 0·01; max. difference peak, 0·23, min., −0·22 e Å^{-3}.

Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The program packages *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978) were also used. All calculations were carried out on the Dundee University PRIME computer. The atomic numbering is shown in the perspective drawing (Fig. 1). Tables 1 and 2 give the atomic parameters, bond lengths and angles.* Fig. 2 shows the hydrogen bonding between symmetry-related molecules. The chirality of the molecule was not determined.

Related literature. The conformational parameters are in accordance with the IUPAC–IUB Joint Commission on Biochemical Nomenclature (1983) guidelines. The related compounds 2′,3′,5′-tri-*O*-acetyladenosine and its 8-bromo derivative have been studied by Wilson, Tollin & Howie (1986) and Boyd, Low & Tollin (1987). Propeller twists between self-base-paired adenine bases are discussed by Wilson & Tollin (1987).

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* 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 51266 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Absolute Structure of 3-[*N*-Methyl-*N*-(*S*)-α-methylbenzyl]carbamoyl-2,4-dimethylpyridinium Chloride Monohydrate

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Abstract. $C_{17}H_{21}ClN_2O.H_2O$, $M_r = 322{\cdot}83$, monoclinic, $P2_1$, $a = 13{\cdot}667\,(1)$, $b = 8{\cdot}585\,(1)$, $c = 16{\cdot}106\,(4)$ Å, $\beta = 106{\cdot}09\,(1)°$, $V = 1815{\cdot}7\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}181$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54184$ Å, $\mu = 19{\cdot}4$ cm^{-1}, $F(000) = 688$, $T = 295$ K, $R = 0{\cdot}0659$ for 6021 observed reflections with $I > 2{\cdot}5\sigma(I)$. The molecules contain three planar parts, the pyridinium and phenyl rings and the carbamoyl fragment in which the carbonyl and *N*-methyl groups are in *anti* position. The water molecules are disordered over two positions with site occupation factors of 0·52 (1) and 0·48 (1) respectively. The Cl$^-$ ions serve as hydrogen-bond acceptors for the N—H groups of the pyridinium cations and for the disordered water molecules.

Experimental. A colourless plate-shaped crystal, 1·00 × 0·40 × 0·06 mm, was glued on top of a glass fibre and transferred to an Enraf–Nonius CAD-4F diffractometer. Unit-cell parameters and their e.s.d.'s were derived from a least-squares treatment of 12 reflections ($10{\cdot}0 < \theta < 14{\cdot}4°$). 7232 intensity data were collected in 'FLAT' mode within two Friedel-related quadrants of the reflection sphere (hkl and $\bar{h}\bar{k}\bar{l}$ with $0 < h < 16$; $0 < k < 10$; $-19 < l < 19$; $2{\cdot}86 < \theta < 70{\cdot}0°$) using Ni-filtered Cu $K\alpha$ radiation in $\omega/2\theta$ scan mode with $\Delta\omega = (0{\cdot}60 + 0{\cdot}15\tan\theta)°$. Three reference

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reflections [400 (r.m.s.d. = 1·23%); 0$\bar{2}$0 (r.m.s.d. = 0·71%); 004 (r.m.s.d. = 0·67%)] measured every hour showed a consistent linear decay of 1·3% in intensity during the 155 h of X-ray exposure time. Intensity data were corrected for this small decay, for Lp and for absorption (Gaussian integration; corrections from 1·13 to 2·11). Standard deviations of the intensities as obtained by counting statistics were increased according to an analysis of the excess variance of the reference reflections: $\sigma^2(I) = \sigma^2_{\rm cs}(I) + (0{\cdot}0141I)^2$ resulting in 6021 observed reflections with $I > 2{\cdot}5\sigma(I)$ (McCandlish, Stout & Andrews, 1975). Structure solved by direct methods (*SHELXS*86; Sheldrick, 1986), the solution with the best figure of merit revealed all non-H atoms except the disordered water molecules. All non-hydrogen atoms (except those of the water molecules) were refined with anisotropic thermal parameters by blocked full-matrix least-squares techniques, minimizing $\sum w\Delta F^2$, using the *SHELX*76 package (Sheldrick, 1976). The disordered water molecules were located from a difference Fourier map and included in the refinement with isotropic thermal parameters. H atoms were introduced on calculated positions (X—H = 0·98 Å) and refined riding on their carrier atoms, with one common isotropic thermal parameter [U = 0·122 (3) Å^2]. H atoms of the water molecules could not be located. At the final stage of the refinement, weights were introduced resulting in $R = 0{\cdot}0659$ $\{wR = 0{\cdot}0845,\ S = 0{\cdot}75,\ w = 1/[\sigma^2(F) + 0{\cdot}00017F^2],\ (\Delta/\sigma)_{\rm ave} = 0{\cdot}093,\ (\Delta/\sigma)_{\rm max} = 0{\cdot}426\}$ for 6021 reflections and 418 parameters. The alternative absolute configuration was rejected based on the refinement of the inverted model which resulted in the significantly higher R values: $R = 0{\cdot}0776$, $wR = 0{\cdot}0982$. These refinements establish the chirality of C(7) as (S), in accordance with the chirality of the commercially obtained synthetic precursor N(S)-α-methylbenzylamine of the title compound. A final difference Fourier synthesis revealed residual densities between 0·57 and −0·37 e Å^{-3} near Cl atoms.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$ or U(Å^2)
Molecule I				
Cl(1)	0·4471 (1)	0·4136 (2)	0·5657 (1)	0·0627 (3)
O(1)	0·9065 (2)	0·4194 (4)	0·3328 (1)	0·0606 (8)
N(1)	0·7970 (2)	0·4604 (4)	0·2008 (2)	0·0466 (8)
N(2)	0·6493 (2)	0·6074 (4)	0·4084 (2)	0·056 (1)
C(1)	0·8615 (4)	0·5251 (7)	0·0044 (3)	0·087 (2)
C(2)	0·8719 (5)	0·6462 (9)	−0·0508 (3)	0·102 (3)
C(3)	0·8994 (5)	0·7894 (9)	−0·0214 (4)	0·099 (3)
C(4)	0·9126 (6)	0·8181 (9)	0·0626 (5)	0·124 (3)
C(5)	0·9055 (5)	0·7019 (7)	0·1208 (4)	0·097 (2)
C(6)	0·8789 (3)	0·5522 (5)	0·0913 (2)	0·053 (1)
C(7)	0·8763 (2)	0·4269 (6)	0·1566 (2)	0·052 (1)
C(8)	0·6927 (3)	0·4908 (6)	0·1450 (3)	0·066 (1)
C(9)	0·8197 (2)	0·4461 (4)	0·2858 (2)	0·042 (1)
C(10)	0·7361 (2)	0·4588 (4)	0·3299 (2)	0·042 (1)
C(11)	0·6868 (3)	0·3252 (5)	0·3457 (2)	0·049 (1)
C(12)	0·7052 (4)	0·1706 (5)	0·3100 (3)	0·068 (2)
C(13)	0·6199 (3)	0·3391 (5)	0·3971 (3)	0·058 (1)
C(14)	0·6023 (3)	0·4813 (6)	0·4267 (3)	0·061 (1)
C(15)	0·7160 (3)	0·6014 (5)	0·3605 (2)	0·049 (1)
C(16)	0·7636 (4)	0·7506 (5)	0·3448 (3)	0·071 (2)
C(17)	0·8633 (4)	0·2615 (6)	0·1223 (3)	0·075 (2)
Molecule II				
Cl(2)	0·5862 (1)	0·1169 (2)	0·0326 (1)	0·1190 (6)
O(1)	0·1220 (2)	0·3928 (4)	0·2361 (2)	0·064 (1)
N(1)	0·2187 (2)	0·5686 (4)	0·3275 (2)	0·0487 (8)
N(2)	0·3686 (3)	0·4443 (5)	0·1172 (2)	0·065 (1)
C(1)	0·1006 (3)	0·8397 (6)	0·4463 (3)	0·065 (1)
C(2)	0·0725 (4)	0·9953 (7)	0·4451 (4)	0·081 (2)
C(3)	0·0576 (4)	1·0813 (7)	0·3702 (4)	0·085 (2)
C(4)	0·0722 (4)	1·0141 (7)	0·2978 (4)	0·087 (2)
C(5)	0·1000 (4)	0·8588 (6)	0·2997 (3)	0·072 (2)
C(6)	0·1135 (2)	0·7694 (5)	0·3732 (2)	0·051 (1)
C(7)	0·1370 (3)	0·5956 (5)	0·3704 (2)	0·051 (1)
C(8)	0·3171 (3)	0·6441 (5)	0·3647 (3)	0·060 (1)
C(9)	0·2028 (3)	0·4614 (5)	0·2651 (2)	0·047 (1)
C(10)	0·2893 (2)	0·4180 (5)	0·2279 (2)	0·048 (1)
C(11)	0·3448 (3)	0·2830 (5)	0·2563 (3)	0·060 (1)
C(12)	0·3331 (4)	0·1948 (6)	0·3330 (3)	0·080 (2)
C(13)	0·4115 (3)	0·2327 (6)	0·2108 (3)	0·074 (2)
C(14)	0·4216 (3)	0·3113 (7)	0·1410 (3)	0·076 (2)
C(15)	0·3019 (3)	0·4984 (5)	0·1576 (2)	0·056 (1)
C(16)	0·2464 (4)	0·6450 (6)	0·1231 (3)	0·073 (2)
C(17)	0·1646 (4)	0·5123 (6)	0·4571 (3)	0·070 (2)
Disordered water molecules [s.o.f. = 0·52 (1)]				
O(10)	0·5603 (7)	0·827 (1)	0·1291 (7)	0·132 (4)
O(11)	0·4497 (9)	0·809 (2)	0·2470 (8)	0·155 (4)
Disordered water molecules [s.o.f. = 0·48 (1)]				
O(12)	0·4967 (6)	0·719 (1)	0·2488 (5)	0·083 (3)
O(13)	0·4462 (9)	0·881 (1)	0·0913 (8)	0·142 (5)

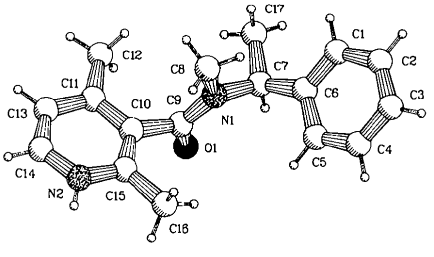

Fig. 1. *PLUTO* (*EUCLID* version; Spek, 1982) drawing of the title compound with the adopted atom numbering. Only one of the two independent (nearly identical) molecules is shown.

Scattering factors were taken from Cromer & Mann (1968), anomalous-dispersion corrections from Cromer & Liberman (1970). All calculations were carried out on a CDC Cyber-855 with the programs mentioned above and with the *EUCLID* package (Spek, 1982; molecular geometry and illustrations). A view of the molecule with the adopted atom numbering is shown in Fig. 1. Final atomic parameters are given in Table 1,* and geometrical data are listed in Table 2.

Related literature. Relevant chemical details involving the synthesis and CD spectra of the title compound were published previously (Bastiaansen, Vermeulen,

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* Lists of structure factors, anisotropic thermal parameters and complete geometrical data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51260 (47 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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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Acta Cryst. (1988). C**44**, 2206–2208

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}, $\lambda(\text{Mo } K\alpha_1) = 0{\cdot}70927$ 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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* This work was performed under the auspices of the US Department of Energy.

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