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Structure of 11β-[4-(Dimethylamino)phenyl]-17β-hydroxy-17α-(2-propenyl)-estra-4,9-dien-3-one

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Abstract. $C_{29}H_{37}NO_2$, $M_r = 431{\cdot}62$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}3527$ (9), $b = 12{\cdot}035$ (1), $c = 17{\cdot}594$ (1) Å, $V = 2403{\cdot}8$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}192$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54184$ Å, $\mu(\mathrm{Cu}\,K\alpha) = 5{\cdot}4$ cm^{-1}, $F(000) = 936$, room temperature, $R = 0{\cdot}060$ for 1817 unique reflexions with $I \geq 2{\cdot}5\sigma(I)$. The overall conformation of the title compound is slightly more bent than that of the 17α-(1-propynyl) parent compound but compared with other $\Delta^{4,9}$ steroids the molecular skeleton is rather flat. The *A* and *B* rings are statistically disordered (1:2), which is indicative of the flexibility of the steroid skeleton. The molecules are hydrogen-bonded head-to-tail.

Introduction. The title compound is the 17α-(2-propenyl) analogue of RU 38 486 {11β-[4-(dimethylamino)phenyl]-17β-hydroxy-17α-(1-propynyl)estra-4,9-dien-3-one; Sakiz, Euvrard & Baulieu (1984)}, which is the lead compound in studies aimed at finding new drugs with antiprogestational and antiglucocorticoid activities (*e.g.* Neef, Beier, Elger, Henderson & Wiechert, 1984) and whose structure was reported recently (van Geerestein, Kanters, van der Sluis & Kroon, 1986). In contrast with RU 38 486, which suffered from serious crystallization problems, the title compound could be readily crystallized and here we report its X-ray analysis.

Experimental. Crystals (ORG 30761) obtained through the Scientific Development Group of Organon, Oss, The Netherlands, were prepared by slow evaporation from acetone at room temperature. Data measured on a crystal of approximate dimensions $0{\cdot}15 \times 0{\cdot}45 \times 0{\cdot}28$ mm on an Enraf–Nonius CAD-4 diffractometer with Ni-filtered Cu $K\alpha$ radiation; lattice parameters refined by least-squares fitting of four alternative settings [*SET*4; de Boer & Duisenberg (1984)] of eight symmetry-related reflexions (668, $\bar{6}68$ *etc.*) with $\theta = \pm 41{\cdot}3°$; ω–2θ scan mode, $\Delta\omega = (0{\cdot}50 + 0{\cdot}15\tan\theta)°$, 2528 independent reflexions measured up to $\theta = 70°$, h,k,l (max. range 13, 14, 21), 1817 of these considered observed $[I \geq 2{\cdot}5\sigma(I)]$ and used for structure refinement. Three periodically measured standard reflexions ($\bar{2}11$, 111, 201) showed intensity variations less than 0·5%; Lp corrections, no correction for absorption or secondary extinction.

Structure solved by direct methods using a preliminary version of *SHELXS*84 (Sheldrick, 1984). Default run gave no solution, possibly due to the fact that only 4941 unique triples were found using 265 largest E values, sorted according to $|E|$. The ratio of phase relations to unknown phases was increased by sorting 300 largest E values according to the estimated α, which resulted in 9544 triplets. Best E map of this run gave all 32 non-H atoms as the 32 highest peaks.

Anisotropic refinement, using *SHELX*76 (Sheldrick, 1976), resulted in very large values for the $U(1,1)$ component of the temperature tensor for atoms C(2), C(6) and C(7), indicating an unrealistic mean-square amplitude of vibration of about 1 Å, and short bond distances involving these atoms [*e.g.* C(6)—C(7) = 1·33 Å]. During subsequent refinement the bond lengths involving these atoms, which were assigned fixed isotropic thermal parameters, were constrained to realistic values. The difference map revealed one satellite peak for C(2), and for C(6) and C(7) as well, indicating a statistical disorder over two positions, here labelled *A* and *B*. The coordinates of

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these three peaks were included in further refinement. The site occupation factor (s.o.f.) for C(2) and the obligatorily coupled s.o.f. for C(6) and C(7) refined to values equal within 1σ. So it was decided to describe the observed disorder with only one s.o.f., which physically means that the C(6)—C(7) flip-flop in ring *B* is considered to be coupled with the disorder of C(2) in ring *A*.

H atoms were included at calculated positions riding on their bonded atoms, except the hydroxyl-group H atom and two H atoms bonded to C(22) (see Fig. 1) of the propenyl group, which were located on a difference map. Bond lengths involving the disordered C atoms were restrained to values found in other $\Delta^{4,9}$ steroids (Griffin, Duax & Weeks, 1984). In final cycles of two-block full-matrix refinement, 62 and 233 parameters varied respectively, including overall scale factor, s.o.f., positional parameters for C, O and N atoms, individual isotropic parameters for the three disordered C atoms, individual anisotropic parameters for O, N and remaining C atoms, positional parameters for H[O(17)] and for H atoms bonded to C(22), and overall thermal parameter for H atoms. The refinement on F converged at $R = 0{\cdot}060$ and $wR = 0{\cdot}045$, where $w = 1/\sigma^2(F)$. Overall thermal parameter for H atoms refined to 0·092 (4) Å^2 and s.o.f. for conformation *A* to 0·68 (1). $\Delta/\sigma = 0{\cdot}05$ (4) (av.) and 0·26 (max.) for non-H-atom parameters. $\Delta/\sigma = 0{\cdot}10$ (5) (av.) and 0·17 (max.) for H-atom parameters; final residual electron density $-0{\cdot}34 < \Delta\rho < 0{\cdot}36$ e Å^{-3}. Scattering factors were taken from Cromer & Mann (1968) for C, O and N atoms and from Stewart, Davidson & Simpson (1965) for H atoms.*

Table 1. *Positional and equivalent isotropic thermal parameters* (Å^2) *for non-*H *atoms with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
O(3)	0·9645 (3)	0·4340 (3)	0·0262 (2)	0·074 (2)
O(17)	1·6145 (3)	0·3484 (3)	0·4724 (2)	0·075 (2)
N(1)	0·9358 (4)	0·6163 (4)	0·5561 (3)	0·069 (2)
C(1)	1·1289 (4)	0·5052 (4)	0·1958 (3)	0·087 (2)
C(2*A*)	1·0194 (5)	0·5067 (5)	0·1468 (3)	0·057 (2)*
C(2*B*)	1·083 (1)	0·5204 (7)	0·1164 (5)	0·062 (5)*
C(3)	1·0194 (4)	0·4200 (4)	0·0865 (2)	0·061 (2)
C(4)	1·0610 (4)	0·3141 (4)	0·1122 (3)	0·067 (2)
C(5)	1·1274 (4)	0·2991 (3)	0·1742 (2)	0·062 (2)
C(6*A*)	1·1387 (6)	0·1831 (4)	0·2078 (4)	0·059 (2)*
C(6*B*)	1·206 (1)	0·1974 (9)	0·1836 (6)	0·055 (5)*
C(7*A*)	1·2629 (5)	0·1721 (6)	0·2383 (4)	0·056 (2)*
C(7*B*)	1·202 (1)	0·172 (1)	0·2679 (6)	0·047 (4)*
C(8)	1·2736 (4)	0·2595 (3)	0·3072 (2)	0·060 (2)
C(9)	1·2349 (4)	0·3758 (4)	0·2825 (3)	0·048 (2)
C(10)	1·1686 (4)	0·3923 (4)	0·2208 (3)	0·048 (2)
C(11)	1·2750 (4)	0·4736 (4)	0·3315 (3)	0·048 (2)
C(12)	1·4050 (4)	0·4609 (4)	0·3582 (3)	0·050 (2)
C(13)	1·4355 (4)	0·3441 (4)	0·3875 (3)	0·051 (2)
C(14)	1·4048 (4)	0·2616 (4)	0·3251 (3)	0·056 (2)
C(15)	1·4628 (5)	0·1520 (4)	0·3510 (3)	0·073 (2)
C(16)	1·5791 (5)	0·1914 (4)	0·3908 (3)	0·070 (2)
C(17)	1·5712 (5)	0·3194 (4)	0·3986 (3)	0·058 (2)
C(18)	1·3720 (3)	0·3196 (3)	0·4638 (2)	0·060 (2)
C(20)	1·6486 (4)	0·3804 (4)	0·3383 (3)	0·070 (2)
C(21)	1·7700 (5)	0·3373 (5)	0·3374 (4)	0·082 (3)
C(22)	1·8284 (6)	0·3126 (6)	0·2743 (4)	0·094 (3)
C(30)	1·1871 (5)	0·5023 (4)	0·3958 (3)	0·051 (2)
C(31)	1·2164 (5)	0·5799 (4)	0·4516 (3)	0·060 (2)
C(32)	1·1361 (3)	0·6161 (3)	0·5047 (2)	0·062 (2)
C(33)	1·0187 (5)	0·5754 (4)	0·5045 (3)	0·055 (2)
C(34)	0·9913 (5)	0·4965 (4)	0·4504 (3)	0·055 (2)
C(35)	1·0730 (5)	0·4609 (4)	0·3977 (3)	0·055 (2)
C(36)	0·8115 (5)	0·5940 (5)	0·5439 (3)	0·073 (2)
C(37)	0·9709 (5)	0·6958 (5)	0·6128 (3)	0·080 (3)

* Refined isotropically.

Discussion. The atomic parameters are given in Table 1. The two conformations of the steroid molecule and atom numbering are shown in Fig. 1. The intramolecular dimensions involving non-hydrogen atoms are given in Table 2. A scan through the Cambridge Crystallographic Data base on $\Delta^{4,9}$ steroids resulted in data for 19 molecules (van Geerestein *et al.*, 1986) comprising a wide variety of conformations, varying from flat to strongly bent conformations, indicating a strong flexibility for this class of unsaturated steroids (Delettré, Mornon, Lepicard, Ojasoo & Raynaud, 1980). As the bond lengths and angles of ORG 30761 involving the disordered atoms, and also those involving the atoms bonded to these atoms, do not allow a comparison with these data, we will concentrate on discussion of the conformation of the rings and overall conformation of the steroid backbone. The Δ^4 *A* and the Δ^9 *B* rings have 2β-sofa {ΔC_s[C(2)] = 1·5 (5)°} and 6β,7α-half-chair {ΔC_2[C(6)—C(7)] = 4·6 (6)°} conformations respectively for conformation *A*, and 2α-sofa {ΔC_s[C(2)] = 1·0 (6)°} and 6α,7β-half-chair {ΔC_2[C(6)—C(7)] = 7·3 (8)°} conformations respectively for conformation *B*. However, these asymmetry parameters (Duax & Norton, 1975) and as a consequence the descriptions of the conformations are to be handled with some care, because of the difficulty of properly refining the positions of the disordered atoms.

An identical flip-flop disorder of ring *B* was observed for one of the three independent molecules of form II of 17β-hydroxy-11β-methoxy-18-methyl-19-norpregna-4,9-dien-20-yn-3-one (Delettré, Lepicard, Surcouf & Mornon, 1981) and disorder in ring *A*, involving both C(1) and C(2), in the crystal structures of the Δ^4 steroid nortestosterone (Precigoux, Busetta, Courseille & Hospital, 1975) and of 17β-hydroxy-18-methyl-19-norpregna-4,9,11-trien-20-yn-3-one (Delettré, Mornon & Lepicard, 1975). The *C* ring has the usual chair conformation {ΔC_s[C(8)] = 15·9 (4), ΔC_s[C(9)] = 10·1 (4), ΔC_s[C(11)] = 5·9 (4), ΔC_2[C(8)—C(9)] = 18·4 (5), ΔC_2[C(9)—C(11)] = 3·0 (5), ΔC_2[C(11)—C(12)] = 15·4 (5)°} and the *D* ring has an intermediate

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* Lists of structure factors, torsion angles, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43383 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

13β-envelope/13β,14α-half-chair conformation as illustrated by ΔC_s[C(13)] = 13·4 (5) and ΔC_2[C(13)–C(14)] = 16·1 (5)°, which is slightly different from that found for RU 38 486, namely an ideal 13β-envelope, and this causes the intramolecular contact distance C(30)···C(18) = 3·267 (6) Å to be longer than the short distance of 3·108 (4) Å for RU 38 486. The 11β-[4-(dimethylamino)phenyl] group is perpendicular to the steroid skeleton and is coplanar with C(9)—C(11) as indicated by torsion angle C(9)—C(11)—C(30)—C(31) = 171·9 (5)°, which is close to the value of 175·0 (3)° found for RU 38 486. The amino group is significantly distorted from planarity and coplanarity with the phenyl ring as is illustrated by the torsion angles C(36)—N(1)—C(33)—C(34) = −13·1 (8) and C(37)—N(1)—C(33)—C(32) = −3·5 (7)° and atoms N(1), C(36) and C(37) being 0·07 (4), 0·41 (4) and 0·05 (4) Å out of the phenyl plane respectively. The orientation of the propynyl group is given by torsion angle C(15)—C(16)—C(17)—C(20) = −103·3 (5)° [−93·8 (4)° for RU 38 486].

As in RU 38 486 the overall conformation of ORG 30761 is rather flat and this is unusual for $\Delta^{4,9}$ steroids. A least-squares fit of the butadiene system [atoms C(4), C(5), C(9) and C(10)] of ORG 30761 and RU 38 486 resulted in a mean deviation of 0·030 (6) Å for the fitted atoms. A stereoview of this fit is shown in Fig. 2 and illustrates that ORG 30761 is slightly more bent than RU 38 486.

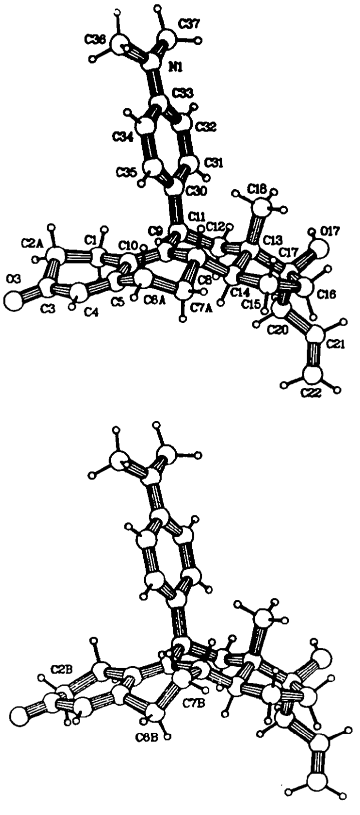

Fig. 1. Molecular structure with atom numbering of conformations *A* (top) [s.o.f.=0·68 (1)] and *B* (bottom) [s.o.f.=0·32 (1)] of the title compound.

Table 2. *Bond distances* (Å) *and bond angles* (°) *for non*-H *atoms with e.s.d.'s in parentheses*

O(3)–C(3)	1·242 (5)	C(9)–C(10)	1·336 (7)
O(17)–C(17)	1·432 (6)	C(9)–C(11)	1·528 (7)
N(1)–C(33)	1·397 (7)	C(11)–C(12)	1·556 (7)
N(1)–C(36)	1·452 (7)	C(11)–C(30)	1·548 (7)
N(1)–C(37)	1·439 (8)	C(12)–C(13)	1·537 (7)
C(1)–C(2*A*)	1·513 (7)	C(13)–C(14)	1·521 (7)
C(1)–C(2*B*)	1·50 (1)	C(13)–C(17)	1·581 (7)
C(1)–C(10)	1·498 (7)	C(13)–C(18)	1·552 (6)
C(2*A*)–C(3)	1·488 (7)	C(14)–C(15)	1·543 (7)
C(2*B*)–C(3)	1·50 (1)	C(15)–C(16)	1·568 (8)
C(3)–C(4)	1·432 (7)	C(16)–C(17)	1·549 (7)
C(4)–C(5)	1·338 (6)	C(17)–C(20)	1·561 (7)
C(5)–C(6*A*)	1·522 (6)	C(20)–C(21)	1·473 (7)
C(5)–C(6*B*)	1·52 (1)	C(21)–C(22)	1·33 (1)
C(5)–C(10)	1·466 (6)	C(30)–C(31)	1·395 (7)
C(6*A*)–C(7*A*)	1·514 (9)	C(30)–C(35)	1·388 (8)
C(6*B*)–C(7*B*)	1·52 (1)	C(31)–C(32)	1·376 (6)
C(7*A*)–C(8)	1·610 (8)	C(32)–C(33)	1·420 (7)
C(7*B*)–C(8)	1·50 (1)	C(33)–C(34)	1·380 (7)
C(8)–C(9)	1·530 (6)	C(34)–C(35)	1·380 (8)
C(8)–C(14)	1·523 (6)		

C(33)–N(1)–C(36)	119·6 (5)	C(9)–C(11)–C(12)	112·2 (4)
C(33)–N(1)–C(37)	119·9 (4)	C(9)–C(11)–C(30)	113·1 (4)
C(36)–N(1)–C(37)	119·6 (5)	C(12)–C(11)–C(30)	114·4 (4)
C(2*A*)–C(1)–C(10)	115·2 (4)	C(11)–C(12)–C(13)	113·9 (4)
C(2*B*)–C(1)–C(10)	119·2 (5)	C(12)–C(13)–C(14)	107·7 (4)
C(1)–C(2*A*)–C(3)	113·4 (4)	C(12)–C(13)–C(17)	115·7 (4)
C(1)–C(2*B*)–C(3)	113·2 (6)	C(12)–C(13)–C(18)	111·1 (4)
O(3)–C(3)–C(2*A*)	120·9 (4)	C(14)–C(13)–C(17)	100·9 (4)
O(3)–C(3)–C(2*B*)	115·5 (5)	C(14)–C(13)–C(18)	113·2 (4)
O(3)–C(3)–C(4)	123·8 (4)	C(17)–C(13)–C(18)	108·1 (4)
C(2*A*)–C(3)–C(4)	113·5 (4)	C(8)–C(14)–C(13)	112·6 (4)
C(2*B*)–C(3)–C(4)	116·5 (5)	C(8)–C(14)–C(15)	117·7 (4)
C(3)–C(4)–C(5)	124·3 (4)	C(13)–C(14)–C(15)	104·3 (4)
C(4)–C(5)–C(6*A*)	119·2 (4)	C(14)–C(15)–C(16)	103·5 (4)
C(4)–C(5)–C(6*B*)	121·8 (5)	C(15)–C(16)–C(17)	107·0 (4)
C(4)–C(5)–C(10)	122·2 (4)	C(13)–C(17)–C(16)	103·4 (4)
C(6*A*)–C(5)–C(10)	117·3 (4)	O(17)–C(17)–C(13)	113·6 (4)
C(6*B*)–C(5)–C(10)	111·5 (5)	O(17)–C(17)–C(16)	107·6 (4)
C(5)–C(6*A*)–C(7*A*)	107·2 (5)	O(17)–C(17)–C(20)	108·0 (4)
C(5)–C(6*B*)–C(7*B*)	104·5 (8)	C(13)–C(17)–C(20)	112·1 (4)
C(6*A*)–C(7*A*)–C(8)	106·3 (5)	C(16)–C(17)–C(20)	112·0 (4)
C(6*B*)–C(7*B*)–C(8)	107·1 (8)	C(17)–C(20)–C(21)	111·6 (4)
C(7*A*)–C(8)–C(9)	111·2 (4)	C(20)–C(21)–C(22)	123·7 (6)
C(7*B*)–C(8)–C(9)	110·8 (5)	C(11)–C(30)–C(31)	120·7 (5)
C(7*A*)–C(8)–C(14)	103·9 (4)	C(11)–C(30)–C(35)	122·6 (4)
C(7*B*)–C(8)–C(14)	129·6 (5)	C(31)–C(30)–C(35)	116·5 (5)
C(9)–C(8)–C(14)	108·9 (3)	C(30)–C(31)–C(32)	122·1 (5)
C(8)–C(9)–C(10)	121·9 (4)	C(31)–C(32)–C(33)	120·7 (4)
C(8)–C(9)–C(11)	117·3 (4)	N(1)–C(33)–C(32)	120·6 (4)
C(10)–C(9)–C(11)	120·8 (4)	N(1)–C(33)–C(34)	122·6 (5)
C(1)–C(10)–C(5)	115·7 (4)	C(32)–C(33)–C(34)	116·8 (5)
C(1)–C(10)–C(9)	122·9 (5)	C(33)–C(34)–C(35)	121·7 (5)
C(5)–C(10)–C(9)	121·4 (4)	C(30)–C(35)–C(34)	122·1 (5)

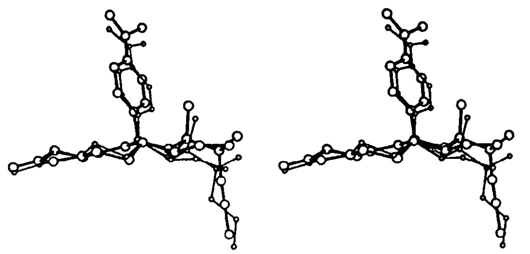

Fig. 2. Stereoview of least-squares fit of the butadiene system of the title compound (thin lines) and RU 38 486 (thick lines).

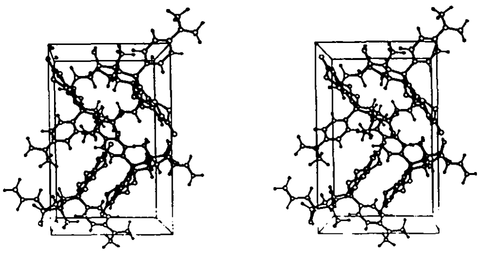

Fig. 3. Stereo packing diagram viewed down **b**.

The above observations lead to the conclusion that, like other $\Delta^{4,9}$ steroids, RU 38 486 and ORG 30761 are flexible molecules and that the angular dimethylaminophenyl group has a fixed position when the steroid molecule is in a flat conformation. It would surely be very interesting to study the flexibility, different conformations and their energy differences of these novel 11β-[4-(dimethylamino)phenyl] $\Delta^{4,9}$ steroids by NMR in solution and by computational techniques like molecular mechanics and molecular dynamics.

The molecular packing is illustrated in Fig. 3, which shows a stereoview down **b**. The steroid molecules are hydrogen-bonded from head to tail, O(17)→O[3, ($\frac{5}{2}-x$, $1-y$, $\frac{1}{2}+z$)] with O···O = 2·926 (5) Å and O—H···O = 171 (5)°. The packing scheme is similar to that of RU 38 486, the difference being the inclusion of the *n*-butyl acetate solvent molecule in the latter crystal structure. There is an unrealistic short intermolecular H···H contact of 1·70 (1) Å between H atoms at C(2*B*) and C(18), indicating that the calculated coordinates of these H atoms are not accurate owing to the only approximate coordinates of C(2*B*); other H···H contact distances are greater than 2·18 Å.

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Hexamethylenetetramine Monohydrochloride*

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Abstract. $C_6H_{13}N_4^+.Cl^-$, $M_r = 176{\cdot}65$, trigonal, $R3m$, $a = 8{\cdot}915$ (2), $c = 8{\cdot}935$ (2) Å (hexagonal axes), $V = 615{\cdot}0$ (3) Å^3, $Z = 3$, $D_m = 1{\cdot}41$ (2), $D_x = 1{\cdot}43$ g cm^{-3}, $F(000) = 282$, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 36{\cdot}44$ cm^{-1}, $T = 297$ K, $R = 0{\cdot}041$ for all 126 independent reflections. The polar adduct possesses full $3m$ symmetry in the crystal. The N^+—H···Cl^- hydrogen bond is also the only close ionic interaction within the crystal.

Introduction. The title compound was prepared during an investigation of hydrated amine salts. Under the crystallization conditions used, however, no water molecules were incorporated into the closely packed

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* 3,5,7-Triaza-1-azoniatricyclo[3.3.1.1^{3,7}]decane chloride.

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