are all at least 45° less than sp^3 and 60° less than sp^2 hybrid C—N bonds. The C(6)—C(3) distance of 1·495 Å is typical for a C—C bond shortened by two adjacent partial double bonds with the two C(6)—C(3)—N angles of 117·5 and 118·3° being considerably larger than those found at normal sp^3 carbon. This again reflects the bonding distortion at C(3) created by its being a member of a diaziridine ring. The enlargement of the exocyclic diaziridine ring angle is also shown in compounds (3) and (4).

There is a short distance of 3·17 Å between N(1)(x,y,z) and N(3)′(x, y − 1, z) which may be attributed to intermolecular hydrogen bonding involving H(13). Strong evidence for this is given by the angles C(2)—N(1)···N(13)′ = 111·1°, C(1)—N(1)···N(3)′ = 112·7°, and N(1)—H(13)···N(3)′ = 172·5°. The hydrogen-bonded strings of molecules stacked along the b axis are shown in Fig. 2.

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2-[1-(4-Quinazolinyl)hydrazino]ethanol Hydrochloride

By A. Forbes Cameron and Andrew A. Freer

*Department of Chemistry, University of Glasgow, Glasgow G*12 8*QQ, Scotland*

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Abstract. $C_{10}H_{13}N_4O^+.Cl^-$, M_r = 240·7, monoclinic, a = 14·083 (2), b = 5·019 (1), c = 16·182 (2) Å, β = 103·8 (2)°, U = 1111·00 Å^3, D_m = 1·46, Z = 4, D_c = 1·44 Mg m^{-3}, $F(000)$ = 504, space group $P2_1/c$. Mo $K\alpha$ radiation, λ = 0·7107 Å, μ(Mo $K\alpha$) = 0·087 mm^{-1}. The reaction of hydroxyethylhydrazine with 4-chloroquinazoline in isopropanol yielded a product for which several plausible structures could be postulated, but for which no one structure could be chosen unambiguously. An X-ray analysis has confirmed that the product is the title compound, N-protonation taking place at N(1) of the quinazoline ring.

Introduction. Exposure of a small crystal to Zr-filtered Mo $K\alpha$ radiation on a Hilger & Watts Y290 diffractometer yielded 1696 independent reflexions [$I \geq 3\sigma_I$, $\sigma_I = (I + B_1 + B_2)^{1/2}$], measured by the θ,ω-scan technique in the 2θ range 0–54°. The intensities were corrected for Lorentz and polarization effects, but not for absorption. The structure was solved by the heavy-atom technique with XRAY (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Refinement of positional and thermal parameters by full-matrix least-squares calculations converged when R was 0·050. Difference syntheses, evaluated after the preliminary cycles of refinement, revealed all the H atoms, and positional parameters were refined for these atoms in later cycles. Throughout the refinement it was not found necessary to apply weights other than unity.

Final fractional coordinates are presented in Table 1.* Table 2 contains details of the molecular geometry and interionic associations. The e.s.d.'s are derived from the least-squares calculations and should be regarded as minimum values. A view of one molecule, defining the atomic numbering, is shown in Fig. 1.

Discussion. The organic moiety of the title compound (I) may be regarded as a protonated and amine-substituted pyrimidine system which is fused *via* C(9) and C(10) to a benzene ring. Comparisons may thus be made with pyrimidine itself (Wheatley, 1960), and with other pyrimidine-containing systems, including bis(2-pyrimidyl) disulphide (Furberg & Solbakk, 1973) and numerous purine and adenine derivatives and analogues (*e.g.* Stewart & Jensen, 1964; Watson, Sweet & Marsh, 1965; McMullan & Sundaralingam,

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34271 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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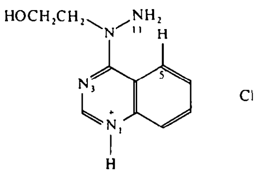
1971; Shefter, Evans & Taylor, 1971; Voet & Rich, 1972; Hecht & Sundaralingam, 1972; Sundaralingam & Abola, 1972; Shikata, Ueki & Mitsui, 1973; Stroud, 1973). We thus note that in amine-substituted examples the exocyclic C–N bond is consistently short [1·331 (5) Å in (I)], which not only suggests a high degree of double-bond character for this bond in all examples, but in the case of (I) itself, when considered in conjunction with the other dimensions of the pyrimidine ring, a slight tendency towards *p*-quinonoid character is indicated for the latter ring of (I). This observation is consistent with the protonation at N(1) in the present example.



(I)

An apparently unique feature of (I) is that the pyrimidine ring is markedly non-planar, possessing a shallow, but pronounced, boat conformation in which N(1) and C(4) deviate from the plane of the other four atoms. This conformation may well result from steric constraints within the present molecule, which in other examples are either absent or are less severe. Thus the C(4) substituent in (I) is a *β*-hydroxyethylhydrazine group rather than a simple amine. Moreover, N(12) is planar, and the plane defined by N(11), N(12) and C(13) does not deviate greatly (5·4°) from the plane of N(3), C(4) and C(10). This latter feature is a probable consequence of the high degree of double-bond character in the C(4)–N(12) bond. The shallow boat conformation of the pyrimidine ring, in conjunction with expansion of the C(4)C(10)C(5) [128·0 (3)°] and C(10)C(4)N(12) [125·4 (3)°] angles, thus contributes to the relief of the severe interactions which would otherwise occur between N(11) and C(5)–H(5). However, N(11) remains in close proximity to C(5) and H(5) [N(11)···C(5) 2·881, N(11)···H(5) 2·33 Å] and is aligned (all H atoms were located from difference

Table 1. *Fractional atomic coordinates* (×10^5 *for* Cl, ×10^4 *for* O, N, C *and* ×10^3 *for* H)

	x	*y*	*z*
Cl(1)	25975 (8)	45155 (21)	70882 (7)
O(15)	4476 (2)	2832 (7)	6523 (2)
N(1)	2754 (3)	6091 (6)	3496 (2)
N(3)	3603 (3)	2727 (7)	4375 (2)
N(11)	2188 (3)	−463 (8)	5666 (2)
N(12)	2941 (2)	252 (6)	5260 (2)
C(2)	3556 (3)	4680 (9)	3821 (3)
C(4)	2788 (3)	2082 (8)	4647 (2)
C(5)	925 (3)	2704 (9)	4406 (3)
C(6)	98 (3)	4044 (9)	3994 (3)
C(7)	159 (4)	6135 (9)	3437 (3)
C(8)	1040 (3)	6821 (9)	3283 (3)
C(9)	1879 (3)	5428 (8)	3688 (2)
C(10)	1852 (3)	3344 (8)	4273 (2)
C(13)	3909 (3)	−949 (8)	5609 (3)
C(14)	4356 (3)	−2 (9)	6505 (3)
H(1)	276 (4)	733 (12)	306 (3)
H(2)	420 (4)	502 (12)	359 (3)
H(5)	85 (4)	190 (12)	488 (3)
H(6)	−53 (4)	390 (12)	417 (3)
H(7)	−44 (4)	712 (12)	316 (3)
H(8)	110 (4)	814 (12)	282 (3)
H(111)	222 (4)	28 (13)	609 (3)
H(112)	242 (4)	−195 (13)	605 (3)
H(131)	379 (4)	−290 (13)	561 (3)
H(132)	442 (4)	−49 (12)	518 (3)
H(141)	390 (4)	−55 (12)	697 (3)
H(142)	512 (4)	−88 (12)	671 (3)
H(15)	403 (4)	327 (13)	669 (4)

Table 2. *Bonded distances* (Å), *interbond angles* (°), *torsion angles* (°), *pertinent intramolecular non-bonded distances* (Å) *and hydrogen bonding*

(*a*) Bonded distances

N(1)–C(2)	1·331 (5)	C(4)–C(10)	1·459 (5)
N(1)–C(9)	1·381 (5)	C(5)–C(6)	1·372 (7)
N(3)–C(2)	1·319 (6)	C(5)–C(10)	1·409 (6)
N(3)–C(4)	1·363 (5)	C(6)–C(7)	1·399 (7)
N(11)–N(12)	1·420 (5)	C(7)–C(8)	1·367 (7)
N(12)–C(4)	1·331 (5)	C(8)–C(9)	1·395 (6)
N(12)–C(13)	1·475 (6)	C(9)–C(10)	1·418 (5)
O(15)–C(14)	1·432 (5)	C(13)–C(14)	1·514 (6)

Average C–H distance 1·04 (6) Å

(*b*) Interbond angles

C(2)–N(1)–C(9)	120·1 (3)	C(6)–C(7)–C(8)	120·3 (4)
C(2)–N(3)–C(4)	119·1 (3)	C(7)–C(8)–C(9)	119·5 (3)
N(11)–N(12)–C(4)	120·9 (2)	N(1)–C(9)–C(8)	118·4 (3)
N(11)–N(12)–C(13)	116·4 (3)	N(1)–C(9)–C(10)	119·7 (2)
C(4)–N(12)–C(13)	122·5 (3)	C(8)–C(9)–C(10)	121·9 (3)
N(1)–C(2)–N(3)	124·4 (2)	C(4)–C(10)–C(5)	128·0 (3)
N(3)–C(4)–N(12)	113·9 (2)	C(4)–C(10)–C(9)	115·6 (2)
N(3)–C(4)–C(10)	120·7 (2)	C(5)–C(10)–C(9)	116·4 (3)
N(12)–C(4)–C(10)	125·4 (3)	N(12)–C(13)–C(14)	111·9 (3)
C(6)–C(5)–C(10)	121·6 (3)	O(15)–C(14)–C(13)	110·6 (3)
C(5)–C(6)–C(7)	120·4 (3)		

(*c*) Selected torsion angles (average e.s.d. for torsion angles is 0·6°)

C(9)–N(1)–C(2)–N(3)	−4·6
N(1)–C(2)–N(3)–C(4)	−0·4
C(2)–N(3)–C(4)–C(10)	5·9
N(3)–C(4)–C(10)–C(9)	−6·2
C(4)–C(10)–C(9)–N(1)	1·4
C(10)–C(9)–N(1)–C(2)	3·8
N(3)–C(4)–N(12)–C(13)	0·0
C(10)–C(4)–N(12)–N(11)	−6·0
C(10)–C(5)–C(6)–C(7)	−1·2
C(5)–C(6)–C(7)–C(8)	1·6
C(6)–C(7)–C(8)–C(9)	−0·3
C(7)–C(8)–C(9)–C(10)	−1·4
C(8)–C(9)–C(10)–C(5)	1·7
C(9)–C(10)–C(5)–C(6)	−0·4

(*d*) Intramolecular non-bonded distances

N(11)···C(5)	2·881 (6)	N(3)···C(13)	2·677 (6)
N(11)···H(5)	2·33 (6)	N(3)···H(132)	2·21 (6)

(*e*) Hydrogen bonding

X–H···Cl$^-$	*X*–H	*X*···Cl$^-$	H···Cl$^-$	∠*X*–H···Cl$^-$
N(1)–H(1)···Cl$^-$	0·94 (8) Å	3·138 (4) Å	2·20 (6) Å	172°
N(11)–H(111)···Cl$^-$	0·82 (7)	3·353 (4)	2·61 (7)	150
N(11)–H(112)···Cl$^-$	0·98 (8)	3·369 (4)	2·42 (8)	165
O(15)–H(15)···Cl$^-$	0·79 (8)	3·114 (4)	2·33 (8)	176

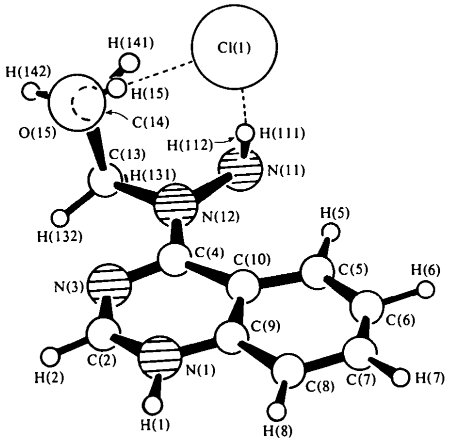


Fig. 1. A view of one molecule showing the atomic numbering. H atoms are numbered according to the atoms to which they are bonded.

syntheses) such that the lone pair of electrons on N(11) is directed towards the C(5)—H(5) bond. In this context we note that the NMR spectrum of (I) [hydrochloride in Me_2SO-d_6] contains an exceptionally low-field 5-proton signal at δ 9·52 (Barker & Taylor, 1976).

Consideration of the structural formula of (I) suggests three sites at which protonation could plausibly have occurred, namely N(1), N(3) and N(11). However, we have already noted the short separation between N(11) and the C(5)—H(5) bond, while N(3) is also in close proximity to the C(13)—H(132) bond. On steric grounds, therefore, N(3) and N(11) would appear to be less favourable sites for protonation than N(1), where, in addition, the interactions between H(1) and H(8) are partly relieved by the boat conformation of the pyrimidine ring.

The organic cations are linked to the Cl^- ions by a complex network of N—H···Cl^- and O—H···Cl^- hydrogen bonds. Other intermolecular distances correspond to, or are greater than, van der Waals contact distances.

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1,4,5,6-Tetrahydro-1,5,*N*-trimethyl-4,6-dioxo-1,3,5-triazine-2-carboxamide

By R. Curtis Haltiwanger, K. J. Kolonko, R. H. Shapiro and R. E. Sievers

Department of Chemistry, University of Colorado, Boulder, Colorado 80309, *USA*

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Abstract. $C_7H_{10}N_4O_3$, M_r = 198·18, monoclinic, $P2_1/c$, a = 9·394 (2), b = 12·461 (4), c = 7·739 (2) Å, β = 93·45 (2)°, U = 904·2 (4) Å^3, Z = 4, d_m = 1·45 (flotation), d_c = 1·46 Mg m^{-3}, F(000) = 416. The structure was solved by direct methods. Full-matrix least-squares refinement converged to R = 0·053 for 1297 observed reflections. The molecules dimerize, presumably as a result of a dipole–dipole interaction of the type found in nucleic acids. This interaction appears to be of sufficient strength to distort the aromaticity of

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