C11	0.8944 (2)	0.9763 (2)	0.29332 (11)	0.0443 (4)
C12	0.9798 (2)	1.0694 (2)	0.26900 (12)	0.0495 (4)
N13	1.0007 (2)	1.09721 (13)	0.19693 (10)	0.0490 (4)
C14	0.9521 (2)	1.0614 (2)	0.06480 (13)	0.0564 (5)
C15	0.8899 (3)	0.9985 (2)	0.00826 (12)	0.0601 (6)
C16	0.8067 (2)	0.8995 (2)	0.02591 (10)	0.0463 (4)
C17	0.7868 (2)	0.86631 (15)	0.10045 (9)	0.0389 (3)
C18	0.8479 (2)	0.93167 (14)	0.16128 (10)	0.0363 (3)
C19	0.9344 (2)	1.02994 (15)	0.14293 (11)	0.0427 (4)
O20	0.7504 (2)	0.84474 (14)	−0.03643 (7)	0.0590 (4)
C21	0.6583 (3)	0.7478 (2)	−0.02329 (12)	0.0647 (6)
O22	0.78629 (13)	0.76352 (11)	0.34025 (7)	0.0469 (3)
C26	0.2602 (2)	0.9395 (2)	0.37587 (11)	0.0467 (4)
O27	0.2900 (2)	1.03828 (15)	0.39006 (10)	0.0808 (5)
O28	0.2032 (2)	0.86660 (15)	0.42692 (9)	0.0685 (4)
C29	0.1717 (3)	0.9094 (3)	0.50414 (13)	0.0688 (7)
C30	0.0219 (3)	0.9510 (3)	0.5082 (2)	0.0787 (8)

Table 2. *Selected geometric parameters* (Å, °)

N1—C2	1.472 (2)	C11—C12	1.398 (3)
N1—C8	1.475 (2)	C12—N13	1.310 (3)
N1—C6	1.476 (2)	N13—C19	1.365 (2)
C2—C3	1.552 (2)	C14—C15	1.351 (3)
C3—C26	1.509 (3)	C14—C19	1.418 (3)
C3—C4	1.531 (2)	C15—C16	1.410 (3)
C4—C5	1.529 (3)	C16—O20	1.360 (2)
C4—C7	1.531 (2)	C16—C17	1.365 (2)
C5—C6	1.535 (3)	C17—C18	1.418 (2)
C7—C8	1.541 (2)	C18—C19	1.422 (2)
C8—C9	1.542 (2)	O20—C21	1.424 (3)
C9—O22	1.421 (2)	C26—O27	1.193 (2)
C9—C10	1.520 (2)	C26—O28	1.331 (2)
C10—C11	1.372 (2)	O28—C29	1.462 (3)
C10—C18	1.429 (2)	C29—C30	1.477 (4)
C2—N1—C8	107.51 (13)	C10—C11—C12	120.5 (2)
C2—N1—C6	108.04 (14)	N13—C12—C11	124.2 (2)
C8—N1—C6	110.42 (13)	C12—N13—C19	117.08 (15)
N1—C2—C3	112.20 (14)	C15—C14—C19	120.9 (2)
C26—C3—C4	113.95 (14)	C14—C15—C16	120.5 (2)
C26—C3—C2	113.22 (15)	O20—C16—C17	125.3 (2)
C4—C3—C2	107.64 (13)	O20—C16—C15	114.2 (2)
C5—C4—C7	108.55 (14)	C17—C16—C15	120.4 (2)
C5—C4—C3	107.0 (2)	C16—C17—C18	120.6 (2)
C7—C4—C3	109.90 (13)	C17—C18—C19	118.6 (2)
C4—C5—C6	108.01 (14)	C17—C18—C10	124.08 (15)
N1—C6—C5	112.28 (14)	C19—C18—C10	117.3 (2)
C4—C7—C8	108.88 (13)	N13—C19—C14	117.7 (2)
N1—C8—C7	110.75 (12)	N13—C19—C18	123.4 (2)
N1—C8—C9	112.28 (12)	C14—C19—C18	118.9 (2)
C7—C8—C9	112.67 (12)	C16—O20—C21	117.72 (14)
O22—C9—C10	110.22 (13)	O27—C26—O28	123.5 (2)
O22—C9—C8	105.58 (12)	O27—C26—C3	124.9 (2)
C10—C9—C8	111.74 (12)	O28—C26—C3	111.6 (2)
C11—C10—C18	117.6 (2)	C26—O28—C29	118.9 (2)
C11—C10—C9	118.75 (15)	O28—C29—C30	110.1 (2)
C18—C10—C9	123.68 (14)		

Data collection: CAD-4 diffractometer software. Cell refinement: CAD-4 diffractometer software. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL*93; *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AB1208). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-(4-Pyridyl)-5,6,7,8-tetrahydro-9*H*-1,2,4-triazolo[4,3-*a*][1,3]diazepine Monohydrate

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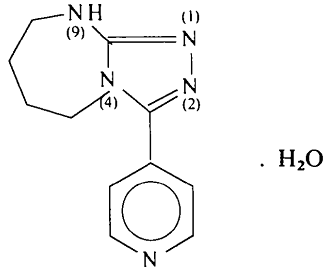
Abstract

The 1,2,4-triazole ring is approximately planar while the saturated 1,3-diazepine ring adopts a twist-chair conformation. The three intermolecular hydrogen bonds form a complicated network consisting of racemic dimers spanned by water molecules.

Comment

Interest in the structure of diazepine rings arises from their presence in many psychoactive benzodiazepines, some of them also having a fused fivemembered hetero-ring, or diazepines having such a ring instead of benzene. Examples of crystal structures of derivatives are 1,2,4-triazolo[4,3-*a*][1,4]-benzodiazepine (Kemmish & Hamor, 1989), 1,2,4-triazolo[5,1-*a*][2,4]benzodiazepine (Wade, Kissick, Vogt & Toeplitz, 1979) and 1,2,4-triazolo[4,3-*b*][1,2]-diazepine (Bromko, Gorazd, Miha, Ljub & Iwan, 1989). Compounds very similar to that of the present study, 6,7,8,9-tetrahydro-7-methyl-3-(4-pyridinyl)-5*H*-1,2,4-triazolo[4,3-*d*]diazepine and its 8-methyl analogue, exhibit depressive and analgesic properties (Guryn, Szadowska & Kiełek, 1988).



(I)

The title compound, (I), was synthesized by Foks & Orlewka (1995) in connection with a program directed towards the search for potential antihypertensive agents. The π electrons in the triazole ring are localized mainly at N(1)=C(10) and N(2)=C(3), as observed from the respective bond lengths (Table 2). The seven-membered ring has a twisted C_7-chair conformation with the best asymmetry parameter $\Delta C_s^7 = 10.1°$ (Duax & Norton, 1975). The twist is evidenced by the torsion angle C(5)—N(4)—C(10)—N(9) of −15.5 (5)° and is in accord with lack of conjugation of the lone pair at N(9) with the triazole π system. This results in a lengthening of the N(9)—C(10) bond by 0.02 Å as compared with the analogous bond lengths of 1.342 (2) and 1.336 (4) Å in the pyrimidine moiety, where conjugation with the lone pair at N(9) was observed (Główka, Foks & Orlewska, 1994).

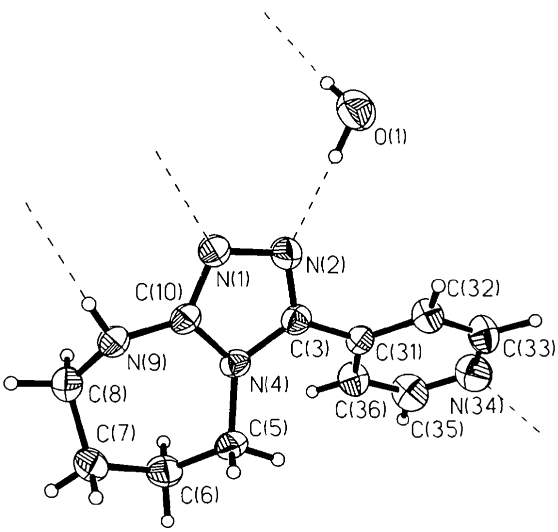


Fig. 1. A view of the molecule with the atomic labelling scheme and hydrogen-bonding directions. Anisotropic displacement parameters are at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

The molecules in the crystal are connected by three hydrogen bonds. Racemic dimers formed by means of N(9)—H···N(1) hydrogen bonds are spanned additionally by water (Table 2). The smallest distance between parallel aromatic systems is 3.71 Å for triazole rings partially overlapping at their N(2) corners.

Experimental

Crystal data

$C_{11}H_{13}N_5.H_2O$
$M_r = 233.3$
Monoclinic
$P2_1/c$
$a = 7.025$ (1) Å
$b = 15.417$ (3) Å
$c = 11.111$ (2) Å
$\beta = 102.38$ (1)°
$V = 1175.4$ (3) Å^3
$Z = 4$
$D_x = 1.318$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10$–18°
$\mu = 0.08$ mm^{-1}
$T = 293$ K
Prism
0.39 × 0.35 × 0.24 mm
White
Crystal source: recrystallized from 2-propanol solution

Data collection

Siemens *P*3 diffractometer
ω–2θ scans
Absorption correction: none
3608 measured reflections
3269 independent reflections
1417 observed reflections [$F > 5\sigma(F_o)$]
$R_{\rm int} = 0.014$

$\theta_{\rm max} = 30°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 21$
$l = -15 \rightarrow 15$
2 standard reflections monitored every 100 reflections
intensity decay: <3%

Refinement

Refinement on *F*
$R = 0.0485$
$wR = 0.0594$
$S = 0.63$
1417 reflections
215 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o) + 0.0038F_o^2]$
$(\Delta/\sigma)_{\rm max} = 0.03$

$\Delta\rho_{\rm max} = 0.27$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}
Extinction correction: Larson (1967)
Extinction coefficient: 0.0038 (7)
Atomic scattering factors from *SHELXTL/PC* (Sheldrick, 1990)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$B_{\rm eq}$
O(1)	0.8406 (4)	0.3884 (2)	0.2130 (3)	4.81 (9)
N(1)	0.7152 (4)	0.4402 (2)	0.5001 (2)	3.09 (6)
N(2)	0.8711 (4)	0.3932 (2)	0.4750 (2)	3.09 (7)

C(3)	0.9782 (4)	0.3678 (2)	0.5801 (2)	2.53 (7)
N(4)	0.9009 (3)	0.3974 (2)	0.6766 (2)	2.29 (5)
C(5)	0.9999 (4)	0.4056 (2)	0.8076 (3)	2.85 (7)
C(6)	0.8762 (5)	0.3774 (3)	0.8963 (3)	3.39 (9)
C(7)	0.7107 (5)	0.4399 (3)	0.9031 (3)	3.57 (9)
C(8)	0.5533 (3)	0.4443 (3)	0.7855 (3)	3.29 (8)
N(9)	0.6178 (4)	0.4854 (2)	0.6821 (2)	2.77 (6)
C(10)	0.7375 (4)	0.4422 (2)	0.6209 (2)	2.41 (6)
C(31)	1.1501 (4)	0.3119 (2)	0.5906 (2)	2.35 (7)
C(32)	1.2773 (5)	0.3228 (2)	0.5122 (3)	2.95 (8)
C(33)	1.4358 (5)	0.2683 (2)	0.5219 (3)	3.67 (10)
N(34)	1.4754 (5)	0.2041 (2)	0.6051 (3)	4.01 (8)
C(35)	1.3489 (6)	0.1931 (2)	0.6773 (3)	3.78 (10)
C(36)	1.1881 (5)	0.2443 (2)	0.6750 (3)	3.10 (8)

Table 2. *Selected geometric parameters* (Å, °)

	This study	Pyrimidine analogues*
N(1)—N(2)	1.390 (4)	1.386 (3)
N(1)—C(10)	1.318 (4)	1.317 (3)
N(2)—C(3)	1.307 (3)	1.306 (4)
C(3)—N(4)	1.378 (4)	1.387 (3)
N(4)—C(10)	1.369 (3)	1.365 (3)
N(9)—C(10)	1.363 (4)	1.339 (3)
N(2)—N(1)—C(10)	106.9 (2)	109.7 (2)
N(1)—N(2)—C(3)	107.7 (2)	108.1 (2)
N(2)—C(3)—N(4)	110.4 (2)	110.0 (2)
C(3)—N(4)—C(10)	104.3 (2)	104.1 (2)
N(1)—C(10)—N(4)	110.7 (3)	111.0 (2)
C(8)—N(9)—C(10)	120.4 (3)	120.3 (2)

D	H	*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(9)	H(9)	N(1^{i})	0.82 (3)	2.16 (3)	2.976 (3)	171 (3)
O(1)	H(1)	N(2)	0.91 (5)	1.97 (5)	2.873 (4)	177 (3)
O(1)	H(2)	N(34^{ii})	0.84 (5)	2.11 (5)	2.956 (4)	176 (4)

Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $-1 + x, \frac{1}{2} - y, -\frac{1}{2} + z$.

* Average values for 1,3-diazepine analogues (Głowka, Foks & Orlewska, 1994). Atoms C(10), N(9) and C(8) correspond to C(9), N(8) and C(7), respectively, in the pyrimidine analogues.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AS1114). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-3-Hydroxy-*N*-methyl-L-proline Hydrochloride

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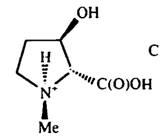
Abstract

The characterization of *trans*-3-hydroxy-*N*-methyl-L-proline, isolated from *Tamarix ramosissima*, as its hydrochloride salt, $C_6H_{12}NO_3^+.Cl^-$, is reported. The carboxylate group occupies a position *trans* to the hydroxyl substituent.

Comment

trans-3-Hydroxy-*N*-methyl-L-proline, (I), has been characterized as part of a study of the role of proline analogues which accumulate in plants as a result of environmental stress (Jones, Naidu, Paleg, Tiekink & Snow, 1987; Solomon, Beer, Waisel, Jones & Paleg, 1994). While the parent amino acid and the *N*,*N*′-dimethylated analogue have been found in plants (Sung & Fowden, 1968; Cornforth & Henry, 1952; Delaveau, Koudogbo & Pousset, 1973), the occurrence of (I) in the *Tamarix* species has not been reported previously.



(I).HCl

The molecular structure of (I) characterized as its HCl salt is shown in Fig. 1. The structure is comprised of protonated cations of (I) and Cl^- anions. In the cation, the disposition of the carboxylate and hydroxyl substituents is *trans* with respect to each other, as is the relationship between the carboxylate group and the *N*-methyl group. In the lattice, there are significant hydrogen-bonding contacts. The separation of 3.082 (5) Å between O(1) and Cl suggests some inter-