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

GRAHAM P. JONES AND LESLIE G. PALEG

Department of Horticulture, Viticulture and Oenology, Waite Agricultural Research Institute, The University of Adelaide, Glen Osmond, SA 5064, Australia

YOAV WAISEL, AARON SOLOMON AND SVEN BEER

Department of Botany, Tel Aviv University, Ramat Aviv 69978, Israel

EDWARD R. T. TIEKINK

Department of Chemistry, The University of Adelaide, Adelaide, SA 5005, Australia

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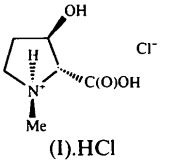
Abstract

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-

action; however, the hydroxyl H atom was not located in the X-ray study. Other interactions include O(3)—H(O3)···Cl [H(O3)···Cl 2.04 (9), O(3)···Cl 3.059 (5) Å and O(3)—H(O3)···Cl 136 (6)°] and N(1)—H(1)···Cl [H(1)···Cl 2.16 (6), N(1)···Cl 3.110 (5) Å and N(1)—H(1)···Cl 147 (5)°].

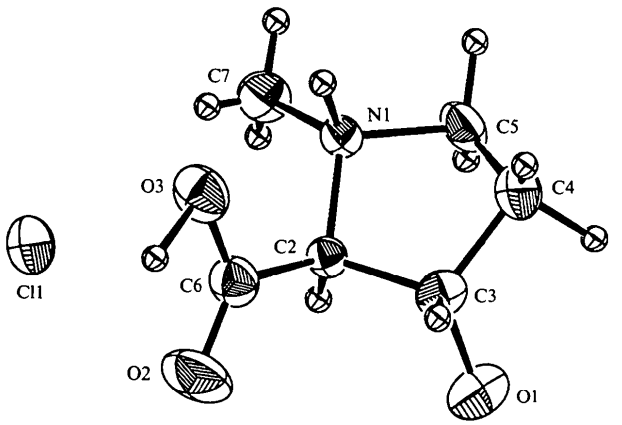


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the molecular structure of protonated (I) showing 40% probability ellipsoids.

Experimental

The compound was isolated, along with other *N*-methylated proline derivatives, from *Tamarix ramosissima* by ion-exchange chromatography; crystals suitable for X-ray analysis were grown by diffusion of diethyl ether into a methanolic solution of the compound held at room temperature.

Crystal data

$C_6H_{12}NO_3^+.Cl^-$
M_r = 181.6
Orthorhombic
$P2_12_12_1$
a = 10.3118 (7) Å
b = 11.5912 (6) Å
c = 7.1818 (6) Å
V = 858.41 (8) Å^3
Z = 4
D_x = 1.405 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 77.8–79.6°
μ = 3.671 mm^{-1}
T = 293 K
Block
0.24 × 0.10 × 0.10 mm
Colourless

Data collection

AFC-6*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
881 measured reflections
881 independent reflections
802 observed reflections [$I > 3.0\sigma(I)$]
θ_{max} = 65.0°
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 8$
3 standard reflections monitored every 400 reflections
intensity decay: 0.87%

Refinement

Refinement on F
R = 0.050
wR = 0.059
S = 4.25
802 reflections
145 parameters
All H-atom parameters refined; O(1) H atom not located
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{max}$ = 1.432 (for H atom)
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: Zachariasen (1967) type 2, Gaussian isotropic
Extinction coefficient: 34.9
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Cl(1)	0.5662 (1)	0.5725 (1)	0.2833 (2)	0.0482 (4)
O(1)	−0.0438 (5)	0.3447 (4)	0.3767 (7)	0.062 (2)
O(2)	0.2135 (5)	0.5255 (3)	0.217 (1)	0.084 (2)
O(3)	0.3623 (4)	0.3877 (4)	0.2008 (8)	0.058 (1)
N(1)	0.1939 (5)	0.2206 (4)	0.1086 (6)	0.033 (1)
C(2)	0.1452 (6)	0.3329 (4)	0.1840 (8)	0.032 (1)
C(3)	0.0864 (6)	0.3039 (5)	0.3769 (9)	0.042 (2)
C(4)	0.0925 (9)	0.1708 (6)	0.387 (1)	0.060 (2)
C(5)	0.1032 (7)	0.1323 (5)	0.190 (1)	0.050 (2)
C(6)	0.2436 (6)	0.4265 (5)	0.2015 (9)	0.048 (2)
C(7)	0.2026 (9)	0.2165 (7)	−0.0999 (10)	0.051 (2)

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

O(1)—C(3)	1.423 (8)	N(1)—C(7)	1.501 (8)
O(2)—C(6)	1.194 (7)	C(2)—C(3)	1.550 (8)
O(3)—C(6)	1.305 (7)	C(2)—C(6)	1.492 (8)
N(1)—C(2)	1.496 (6)	C(3)—C(4)	1.546 (9)
N(1)—C(5)	1.505 (8)	C(4)—C(5)	1.49 (1)
C(2)—N(1)—C(5)	104.0 (4)	O(1)—C(3)—C(4)	111.7 (6)
C(2)—N(1)—C(7)	114.1 (5)	C(2)—C(3)—C(4)	104.1 (5)
C(5)—N(1)—C(7)	113.9 (6)	C(3)—C(4)—C(5)	104.8 (6)
N(1)—C(2)—C(3)	105.4 (4)	N(1)—C(5)—C(4)	102.3 (6)
N(1)—C(2)—C(6)	115.8 (5)	O(2)—C(6)—O(3)	125.2 (6)
C(3)—C(2)—C(6)	110.4 (5)	O(2)—C(6)—C(2)	122.0 (6)
O(1)—C(3)—C(2)	107.2 (5)	O(3)—C(6)—C(2)	112.8 (5)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including bond distances and angles involving H atoms, have been deposited with the IUCr (Reference: AS1139). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-γ-L-Glutamyl-β-cyano-L-alanine, an Antinutritional Factor *ex Vicia sativa L.*, as its Ammonium Salt

IAN M. DELAERE AND MAX E. TATE

Department of Plant Science, Waite Agricultural Research Institute, The University of Adelaide, Glen Osmond, SA 5064, Australia

GRAHAM P. JONES

Department of Horticulture, Viticulture and Oenology, Waite Agricultural Research Institute, The University of Adelaide, Glen Osmond, SA 5064, Australia

EDWARD R. T. TIEKINK

Department of Chemistry, The University of Adelaide, Adelaide, SA 5005, Australia

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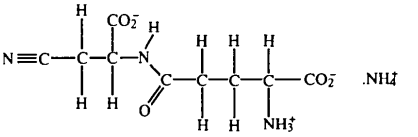
Abstract

The characterization of *N*-γ-L-glutamyl-β-cyano-L-alanine, an antinutritional factor present in *Vicia sativa L.*, as its ammonium salt, $NH_4^+.C_9H_{12}N_3O_5^-$, is reported.

Comment

Seeds of the *Vicia* species are known to contain a wide variety of toxic components, some of which have been implicated as causal agents in human and animal poisoning (Tate & Enneking, 1992). *N*-γ-L-Glutamyl-β-cyano-L-alanine was first isolated as the major biologically active component from *V. sativa L.* by Ressler, Nigam & Giza (1969). It was shown quantitatively to account for the lethality of diets containing 50% *V. sativa* given to week-old chickens, and higher doses showed growth retardent effects in rats. Tate & Enneking (1992) pointed out the unsuitability of the *V. sativa* cultivar Blanche Fleur (which contains 0.5–0.8% *N*-γ-L-glutamyl-β-cyano-L-alanine) for human consumption, which at the time was being exported from Australia as a cheap substitute for red lentils (*Lens culinaris*). Subsequently, the importation of dehulled split Blanche Fleur cotyledons was banned by both India and Egypt. The present report details the X-ray structure determination of *N*-γ-L-glutamyl-β-cyano-L-alanine, characterized as its ammonium salt (I).



(I)

The molecular structure of the anion in (I) is shown in Fig. 1. The end of the molecule possessing the C(1) carboxylate group is zwitterionic with the N(1) atom protonated. The charge balance for the C(7) carboxylate group is provided by the ammonium cation.

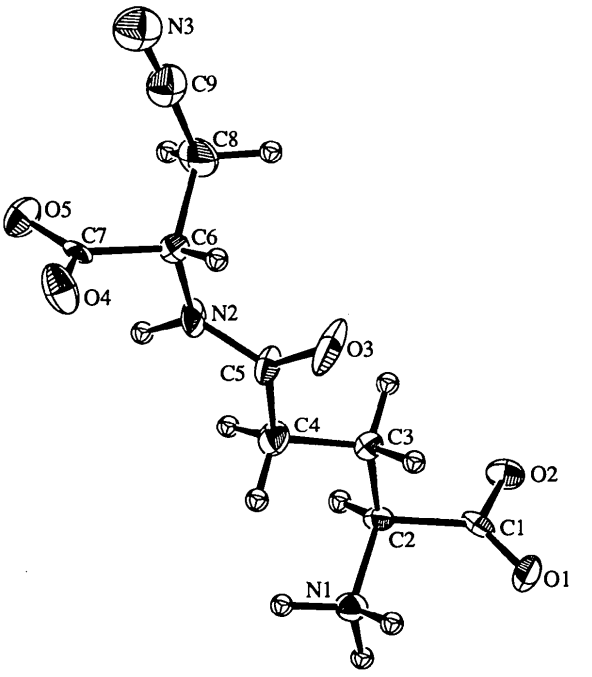


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the molecular structure of the anion in (I) showing 40% probability ellipsoids.