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4-Hydroxy-*N*-methylproline

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.026
wR factor = 0.061
Data-to-parameter ratio = 9.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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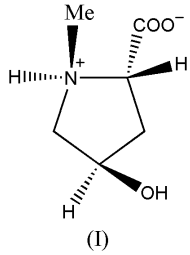
The title compound, $C_6H_{11}NO_3$, was isolated from methanolic extracts of *Lansium domesticum*, a plant with reported antimalarial activity. The structure is a cyclic hydroxy-amino acid with the carboxyl and hydroxyl groups *trans* to one another.

Comment

The leaves, fruit skin, seed and bark of *Lansium domesticum*, a fruit tree found in Malaysia, have been used to treat malaria by traditional healers. The title compound, (I), a cyclic hydroxy-amino acid was isolated from methanol extracts of the fruit skin during studies into the antimalarial activity of the plant (Yapp & Yap, 2002). The free structure of (I) has not been previously described, although the hydrochloride salt of the compound has (Jones *et al.*, 1988). We also report here, for the first time, the ^{1}H and ^{13}C NMR spectra of (I) in solution.



(I)

Compound (I) adopts an envelope conformation typical of five-membered rings, with the carboxyl and hydroxyl groups *trans* to one another (Fig. 1). The structure of the free compound is essentially the same as the hydrochloride salt; no significant differences in bond lengths and angles between the two structures were found. In the free compound, however, two types of hydrogen bonds were found (Fig. 2 and Table 1).

Compound (I) was tested for antiplasmodial activity towards a strain of chloroquine resistant *plasmodium falciparum*, t9, but activity was found only at concentrations greater than 1 mg ml^{-1}.

Experimental

Dehydrated and pulverized fruit skin of *Lansium domesticum* was extracted with methanol following extraction with hexanes and chloroform. The resulting brown solutions were concentrated and stored at 277 K. The crystals which formed were collected and washed with cold ethanol. ^{1}H and ^{13}C NMR spectra were run on a Bruker Avance 360 and Bruker DRX 500, respectively (D_2O). The chemical shifts and multiplicity corresponded well to the solid state structure. ^{13}C data (p.p.m.): 173, C; 70.39, CH; 69.81, CH; 62.90, CH_2;

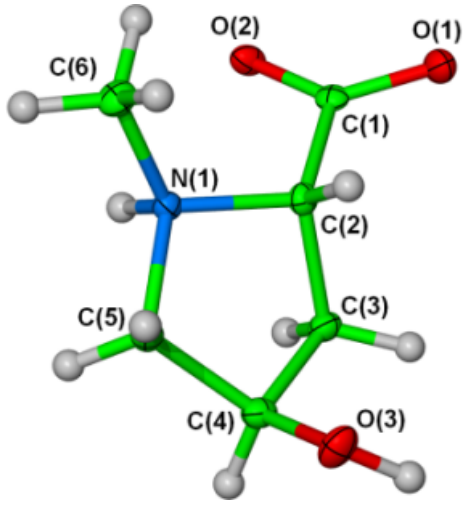


Figure 1
The molecular structure of (I), with ellipsoids at the 50% probability level. **[Please provide missing information]**

43.47, CH_3; 38.58, CH_2 for C1 to C6, respectively. ^{1}H data (p.p.m.): 4.5 (*q*, H); 4.08 (*m*, H); 3.85 (*dd*, H); 3.09 (*dd*, H); 2.94 (*s*, 3H); 2.38 (*q*, H); 2.14 (*m*, H).

Crystal data

$C_6H_{11}NO_3$
M_r = 145.16
Monoclinic, $P2_1$
a = 6.6709 (6) Å
b = 5.7814 (5) Å
c = 8.8881 (6) Å
β = 100.684 (5)°
V = 336.85 (5) Å^3
Z = 2
D_x = 1.431 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1174 reflections
θ = 3.1–27.4°
μ = 0.12 mm^{-1}
T = 120 (2) K
Block, colourless
0.40 × 0.30 × 0.30 mm

Data collection

KappaCCD diffractometer
φ and ω scans
Absorption correction: empirical (Otwinowski & Minor, 1997)
T_{min} = 0.956, T_{max} = 0.966
1732 measured reflections
1174 independent reflections
1112 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 27.4°
$h = -8 \rightarrow 7$
$k = -6 \rightarrow 7$
$l = -8 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.061
S = 1.07
1174 reflections
125 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0251P)^2 + 0.0385P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.086 (12)
Absolute structure: (Flack, 1983), 323 Friedel pairs
Flack parameter = −0.3 (11)

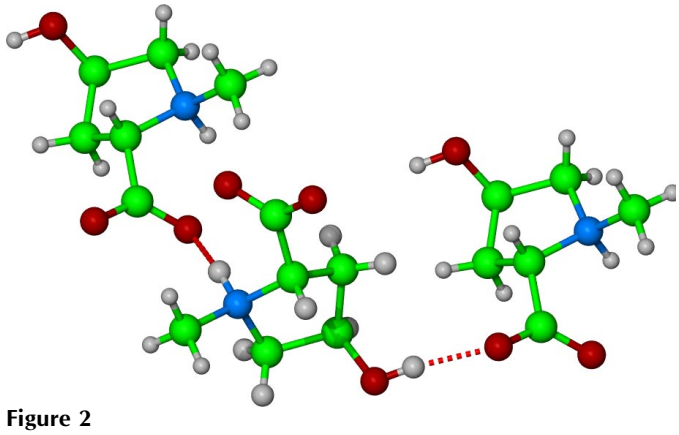


Figure 2
Packing diagram of (I), showing the hydrogen bonding.

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.971 (19)	1.74 (2)	2.6907 (16)	164.7 (18)
O3—H3···O1^{ii}	0.87 (2)	1.84 (2)	2.6985 (13)	173 (2)

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

The methyl H atoms were positioned geometrically, and the other H atoms were refined isotropically. The C—H, N—H and O—H bond lengths are 0.94 (2)–1.01 (2), 0.97 (2) and 0.87 (2) Å, respectively. The model structure is consistent with the known absolute configuration of the molecule although the Flack (1983) test results are meaningless.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *Xseed* (Barbour, 1999).

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