Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Amino-5-methylpyridinium 3-(4-hydroxy-3-methoxyphenyl)-2-propenoate monohydrate

Richeng Xuan,[a]* Yuehua Wan,[b] Weixiao Hu,[a] Zhongyu Yang,[a] Dongping Cheng[a] and Rirong Xuan[c]

[a]College of Pharmaceutical Sciences, Zhejiang University of Technology, Hangzhou 310023, People's Republic of China, [b]Library, Zhejiang University of Technology, Hangzhou 310023, People's Republic of China, and [c]Zhejiang Research Institute of Metallurgy, Hangzhou 310023, People's Republic of China

Correspondence e-mail: xuanrch@mail.hz.zj.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.049
wR factor = 0.137
Data-to-parameter ratio = 12.7

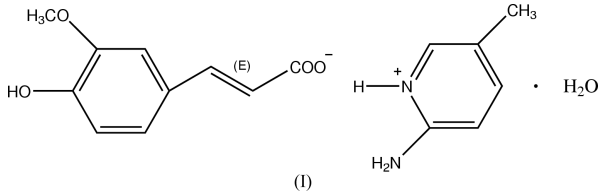
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 2 October 2003
Accepted 6 October 2003
Online 15 October 2003

In the title compound, $C_6H_9N_2^+\cdot C_{10}H_9O_4^-\cdot H_2O$, the N atom in the pyridine ring is protonated in preference to the amino N atom. O—H···O, N—H···O and C—H···O interactions generate a three-dimensional molecular network.

Comment

3-(4-Hydroxy-3-methoxyphenyl)-2-propenoic acid is an active component in Chinese medicinal plants, and it can be used to prevent liver toxicity, to inhibit inflammation and as an anti-allergic agent. In fact, it also exists in many other plants. In order to isolate it from plant components, we attempted to find a molecular recognition agent. 2-Amino-5-methylpyridine can form good crystals with 3-(4-hydroxyl-3-methoxyphenyl)-2-propenoic acid in an aqueous solution, giving the title compound, (I). In order to understand how the compounds crystallize and what the structural features are, the crystal structure analysis of (I) has been performed and the results are presented here.



(I)

In the asymmetric unit of (I), there is one 2-amino-5-methyl pyridinium cation, one 3-(4-hydroxyl-3-methoxyphenyl)-2-propenoate anion and one water molecule (Fig. 1, Table 1). The amino atom N2 has predominantly sp^2 character, as is shown by the C11—N2 bond length of 1.333 (3) Å, which is shorter than the mean value (1.360 Å) reported for planar amino groups bonded to aromatic systems (Allen *et al.*, 1987). Atom N1 in the pyridine ring is protonated, and delocalization involving the amino group is indicated by very similar bond

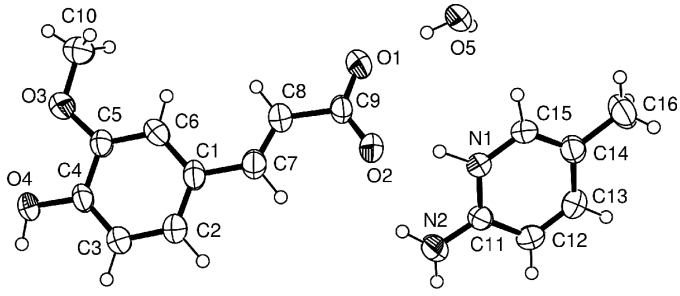


Figure 1
The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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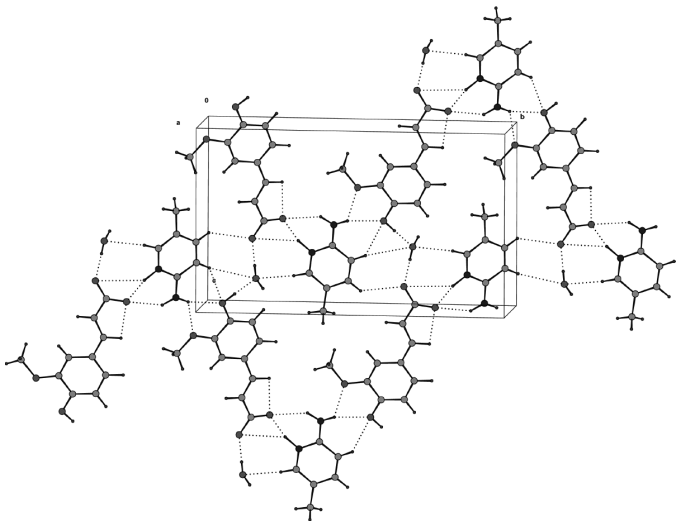

Figure 2
A packing diagram for (I), showing the hydrogen-bonding interactions (dotted lines) in a two-dimensional molecular network.

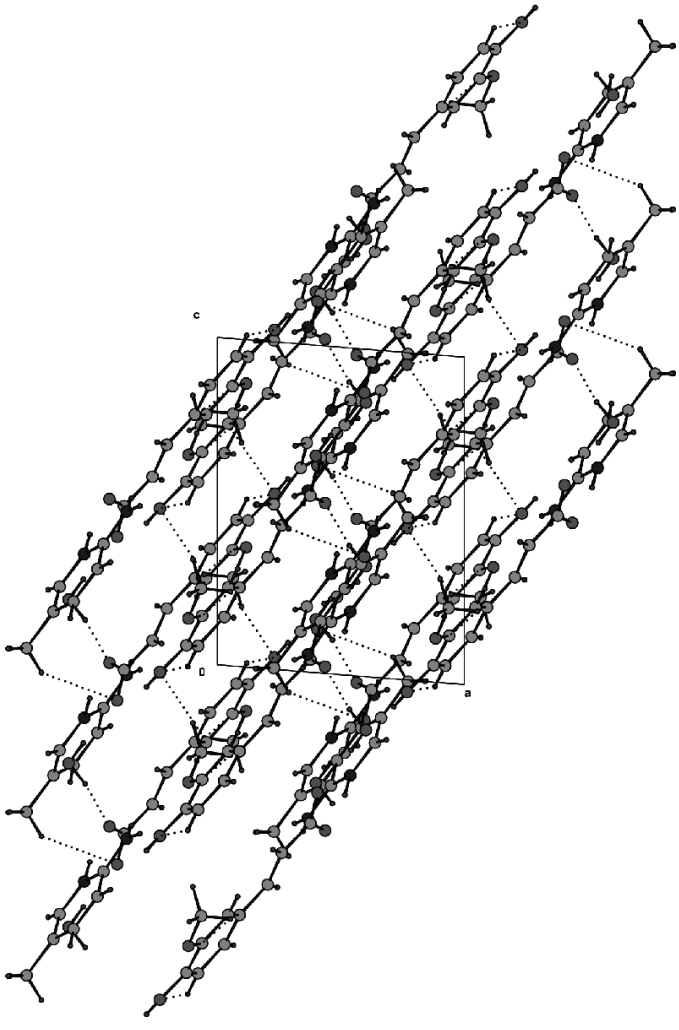

Figure 3
A packing diagram for (I), viewed along the *b* axis, showing the hydrogen-bonding interactions (dotted lines) between adjacent two-dimensional molecular networks.

lengths for N1—C11 and N2—C11. All non-H atoms in the anion are almost coplanar, as seen from the torsion angles (Table 1).

In the crystal structure of (I), O—H···O, N—H···O and C—H···O interactions (Table 2) generate two-dimensional molecular networks (Fig. 2). The O5—H5*B*···O2^{i}, C16—H16*B*···O1^{i} and C10—H10*C*···O4^{v} interactions connect adjacent molecular networks (Fig. 3) [symmetry codes: (i) x, $\frac{3}{2}-y$, $z-\frac{1}{2}$; (v) $-x$, $1-y$, $1-z$].

Experimental

3-(4-Hydroxy-3-methoxyphenyl)-2-propenoic acid (0.20 g, 1.03 mmol) and 2-amino-5-methyl pyridine (0.11 g, 1.02 mmol) were added to and dissolved in water (10 ml), and heated to boiling. The solution was filtered while it was still warm. The filtrate was then cooled to room temperature and allowed to evaporate slowly. Yellow crystals of (I) suitable for X-ray diffraction analysis were obtained in 3 d.

Crystal data

$C_6H_9N_2^+\cdot C_{10}H_9O_4^-\cdot H_2O$
M_r = 320.34
Monoclinic, $P2_1/c$
a = 8.2127 (4) Å
b = 17.9985 (10) Å
c = 10.8564 (5) Å
β = 94.603 (3)°
V = 1599.58 (14) Å^3
Z = 4
D_x = 1.330 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8466 reflections
θ = 2.2–27.4°
μ = 0.1 mm^{-1}
T = 293 (2) K
Prism, yellow
0.30 × 0.15 × 0.14 mm

Data collection

Rigaku RAXIS-RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.908, T_{max} = 0.986
7122 measured reflections
3666 independent reflections
2100 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -23 \rightarrow 23$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.137
S = 0.93
3666 reflections
289 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0768P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0077 (16)

Table 1
Selected geometric parameters (Å, °).

O4—C4	1.360 (2)	N1—C11	1.342 (2)
O3—C5	1.366 (2)	N1—C15	1.358 (3)
O3—C10	1.416 (3)	C9—C8	1.494 (3)
O2—C9	1.259 (3)	C7—C8	1.296 (3)
O1—C9	1.230 (3)	C1—C7	1.477 (3)
N2—C11	1.333 (3)		
O2—C9—C8—C7	−6.6 (3)	C2—C1—C7—C8	173.0 (2)
O1—C9—C8—C7	172.9 (2)	C1—C7—C8—C9	179.9 (2)
C10—O3—C5—C6	2.3 (3)		

Table 2
Hydrogen-bonding and short-contact geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O1	0.84 (3)	1.82 (3)	2.655 (2)	178 (3)
O5—H5B···O2^{i}	0.94 (4)	1.79 (4)	2.729 (3)	171 (3)
O4—H4···O5^{ii}	0.94 (3)	1.67 (3)	2.604 (2)	174 (3)
N2—H2A···O2	0.91 (3)	2.27 (3)	3.013 (3)	139 (2)
N2—H2B···O4^{iii}	0.89 (3)	2.17 (3)	3.023 (3)	162 (2)
N2—H2B···O3^{iii}	0.89 (3)	2.39 (3)	3.021 (2)	129 (2)
N1—H1···O2	1.06 (3)	1.66 (3)	2.687 (2)	162 (2)
N1—H1···O1	1.06 (3)	2.91 (3)	3.792 (2)	140.4 (18)
C16—H16B···O1^{i}	0.95 (4)	2.89 (4)	3.654 (4)	138 (3)
C15—H15···O5	0.95 (2)	2.40 (2)	3.180 (3)	139.7 (16)
C13—H13···O1^{iv}	0.93 (2)	2.55 (2)	3.278 (3)	136.0 (18)
C12—H12···O5^{iv}	0.93 (2)	2.80 (2)	3.675 (3)	157.4 (19)
C10—H10C···O4^{v}	0.98 (3)	2.64 (3)	3.348 (4)	130 (2)
C12—H12···O4^{iii}	0.93 (2)	2.59 (2)	3.349 (3)	139.6 (19)
C7—H7···O2	1.05 (2)	2.47 (2)	2.868 (2)	101.6 (15)

Symmetry codes: (i) $x, \frac{3}{2}-y, z-\frac{1}{2}$; (ii) $1+x, y, 1+z$; (iii) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iv) $-1-x, \frac{1}{2}+y, -\frac{1}{2}-z$; (v) $-x, 1-y, 1-z$.

All H atoms were located in difference Fourier maps and refined isotropically, with refined distances of C—H = 0.90 (3)–1.05 (3) Å, N—H = 0.89 (3)–1.06 (3) Å and O—H = 0.84 (3)–0.94 (3) Å.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Molecular Structure Corporation & Rigaku, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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