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2-Amino-6-methylpyridinium 3-(4-hydroxy-3-methoxyphenyl)-prop-2-enoate monohydrate

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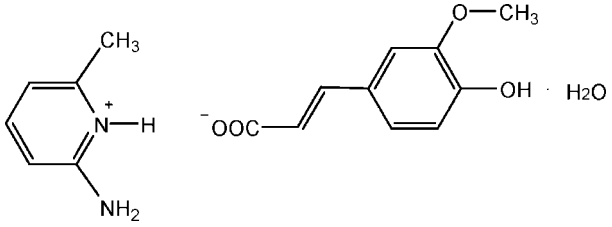
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.033; wR factor = 0.075; data-to-parameter ratio = 8.2.

In the title salt, $C_6H_9N_2^+\cdot C_{10}H_9O_4^-\cdot H_2O$, the cation, anion and solvent molecule are held together by N—H···O hydrogen bonds. A one-dimensional chiral chain along the c axis is formed by O—H···O hydrogen bonds. The chirality of the supramolecular chain-like arrangement is extended into layers parallel to the (101) plane by additional O—H···O hydrogen bonds. The structure is further stabilized by π–π interactions between benzene and pyridine rings [centroid-to-centroid distances = 3.813 (4) and 3.658 (3) Å].

Related literature

For related literature, see: Etter (1990); Inuzuka & Fujimoto (1986, 1990); Ishikawa *et al.* (2002); Jin *et al.* (2000, 2002); Kaminskii *et al.* (2006, 2007); Kiebacha *et al.* (2006); Sarma *et al.* (1997); Thalladi *et al.* (1999); Xuan *et al.* (2003).



Experimental

Crystal data

$C_6H_9N_2^+\cdot C_{10}H_9O_4^-\cdot H_2O$
M_r = 320.34
Monoclinic, Cc
a = 11.266 (2) Å
b = 9.711 (1) Å
c = 15.024 (2) Å
β = 104.547 (8)°
V = 1591.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 (2) K
0.40 × 0.40 × 0.36 mm

Data collection

Siemens $P4$ diffractometer
Absorption correction: multi-scan (*SHELXTL*; Bruker, 1998)
T_{min} = 0.955, T_{max} = 0.960
2235 measured reflections
1920 independent reflections
1571 reflections with $I > 2\sigma(I)$
R_{int} = 0.012
3 standard reflections
every 97 reflections
intensity decay: 1.4%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.075
S = 0.98
1920 reflections
234 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O4	0.86 (2)	1.89 (3)	2.746 (2)	175
N2—H2A···O5	0.85 (2)	1.99 (2)	2.815 (3)	166
N2—H2B···O3	0.86 (2)	2.08 (3)	2.934 (4)	173
O1—H1O···O3^{i}	0.87 (3)	1.73 (3)	2.596 (3)	172
O5—H5A···O1^{ii}	0.82 (1)	2.13 (1)	2.938 (5)	171
O5—H5B···O4^{iii}	0.82 (1)	1.97 (1)	2.785 (2)	171
C6—H6A···O5^{iv}	0.96	2.54	3.302 (3)	136
C13—H13···O1^{v}	0.93	2.52	3.332 (2)	147

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

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2026).

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supplementary materials

Acta Cryst. (2007). E**63**, o3866 [**doi:10.1107/S160053680704038X**]

2-Amino-6-methylpyridinium 3-(4-hydroxy-3-methoxyphenyl)prop-2-enoate monohydrate

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Comment

As reported, the synthesis and characterization of noncentrosymmetric supramolecular systems have attracted a great of interest in th past few years (Sarma *et al.*, 1997; Thalladi *et al.*, 1999; Kaminskii *et al.*, 2006; Kiebacha *et al.*, 2006; Kaminskii *et al.*, 2007). Herein, we report a novel noncentrosymmetric crystal of the title complex (Scheme 1), (I), and discuss the structure (Table 1).

In the structure, 2-amino-6-methylpyridinium (AMP) and 3-(4-hydroxy-3-methoxyphenyl)-2-propenoate (ferulate) are linked through hydrogen bonding of N1—H1N···O4 and N2—H2B···O3 to produce an eight-membered hydrogen bonded ring system of $R_2^2(8)$ arrangement (Etter, 1990). This component shows an interaction with the water molecule by a N2—H2A···O5 hydrogen bond (Fig. 1 & Table 2). This is different from the componentially similar complex 2-amino-5-methylpyridinium ferulate monohydrate (Xuan *et al.*, 2003), where the water molecule is incorporated with AMP by an O—H···O hydrogen bond.

Protonation of 2-aminopyridine always induces aminium-iminium tautomerism (Scheme 2) (Inuzuka & Fujimoto, 1986 and 1990). Obvious features of the iminium tautomer in (I) are revealed by the following bond length comparison. First, the N2—C1 [1.330 (3) Å] bond is slightly but significantly shorter than the N1—C1 [1.349 (3) Å] and N1—C5 [1.361 (3) Å] bonds (Table 1). Second, the C2—C3 [1.352 (4) Å] and C4—C5 [1.353 (4) Å] bonds are shorter than the C1—C2 [1.410 (3) Å] and C3—C4 [1.386 (4) Å] bonds. That means the imimium tautomer makes a great contribution to the structure. This situation is similiar to those observed in 2-amino-5-methylpyridinium ferulate (Xuan *et al.*, 2003), 2-aminopyridinium acetate (Ishikawa *et al.*, 2002), 2-amino-3-methylpyridinium maleate (Jin *et al.*, 2002) and AMP neoabietate (Jin *et al.*, 2000).

As shown in Fig. 2, one-dimensional C_2 chains along the (101) direction are formed by O1—H1O···O3^{i} hydrogen bonds. The presence of water molecules in the structure causes the following hydrogen bonds: N2—H2A···O5, O5—H5A···O1^{ii}, O5—H5B···O4^{iii} and C6—H6A···O5^{iv} (Table 2). These chains link into chiral layers parallel to the (101) plane *via* the O5—H5B···O4^{iii} hydrogen bonds. Neighbouring enantiomeric layers, which are mutual images on the plane of (101) are associated *via* the O5—H5A···O1^{ii} hydrogen bonds. The whole noncentrosymmetric structure is stablized by π–π interactions between neighboring layers, with the relevant centroid···centroid separations between the pyridine and the benzene rings at (x, $1-y$, $z+1/2$), 3.813 (4) Å and ($x-1/2$, $-y+1/2$, $z-1/2$), 3.658 (3) Å.

Experimental

The title compound was synthesized from a mixture of 2-amino-6-methylpyridine (1 mmol, 0.11 g) and ferulic acid (1 mmol, 0.19 g). The mixture was dissolved in 10 ml water, then heated to 373 K and stirred for half an hour. After the reaction system was cooled to room temperature after five days the colorless crystals were collected.

Refinement

H atoms attaching to N and O atoms were deduced from difference Fourier maps and incorporated in the refinement freely. Others were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.93 Å for alkene and aromatic group and 0.96 Å for methyl, with isotropic displacement parameters 1.2 times U_{eq} of the parent atoms.

Figures

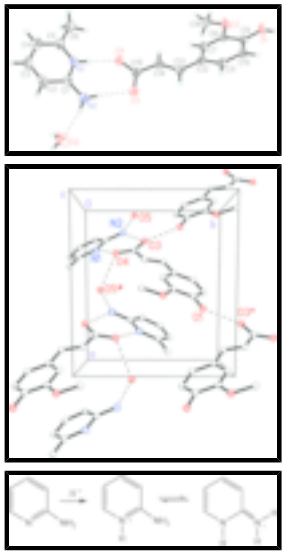

Fig. 1. The cell unit of (I) with atom labels, showing 40% probability displacement ellipsoids. The thin lines denote the hydrogen bonds.
Fig. 2. The chiral hydrogen bond chain of (I) along the [101] direction. Hydrogen bonds are shown by thin lines, All H atoms are omitted for clarity. The superscripts, * and #, indicate the symmetry positions of (x + 1/2, − y + 3/2, z + 1/2) and (x − 1/2, − y + 1/2, z − 1/2), respectively

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Crystal data

$C_6H_9N_2^+·C_{10}H_9O_4^-·H_2O$	F_{000} = 680
M_r = 320.34	D_x = 1.337 Mg m^{-3}
Monoclinic, *Cc*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: C -2yc	Cell parameters from 30 reflections
a = 11.266 (2) Å	θ = 3.7–12.0º
b = 9.711 (1) Å	μ = 0.10 mm^{-1}
c = 15.024 (2) Å	T = 296 (2) K
β = 104.547 (8)º	Block, colourless
V = 1591.0 (4) Å^3	0.40 × 0.40 × 0.36 mm
Z = 4	

Data collection

Siemens P4 diffractometer	R_{int} = 0.012
Radiation source: fine-focus sealed tube	θ_{max} = 28.0º

Monochromator: graphite
T = 296(2) K
ω scans
Absorption correction: empirical (using intensity measurements)
(SHELXTL; Bruker, 1998)
T_{min} = 0.955, T_{max} = 0.960
2235 measured reflections
1920 independent reflections
1571 reflections with $I > 2\sigma(I)$

θ_{min} = 2.8º
h = 0→14
k = 0→12
l = −19→19

3 standard reflections
every 97 reflections
intensity decay: 1.4%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.075
S = 0.98
1920 reflections
234 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0225 (15)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.63139 (17)	0.81309 (17)	0.79651 (11)	0.0505 (5)	
O2	0.55462 (15)	0.56111 (16)	0.80161 (10)	0.0480 (5)	
O3	0.21872 (16)	0.43919 (16)	0.29836 (10)	0.0488 (4)	

O4	0.29544 (17)	0.24240 (15)	0.36111 (11)	0.0446 (4)	
O5	0.0234 (2)	0.3630 (3)	−0.04758 (14)	0.0691 (6)	
N1	0.26566 (16)	0.09244 (19)	0.20202 (13)	0.0365 (4)	
N2	0.1546 (2)	0.2764 (2)	0.12847 (16)	0.0472 (5)	
C1	0.2028 (2)	0.1526 (2)	0.12329 (15)	0.0377 (5)	
C2	0.1912 (2)	0.0791 (3)	0.04057 (16)	0.0472 (6)	
H2	0.1486	0.1168	−0.0152	0.057*	
C3	0.2429 (3)	−0.0470 (3)	0.04365 (19)	0.0556 (7)	
H3	0.2364	−0.0955	−0.0107	0.067*	
C4	0.3054 (3)	−0.1052 (3)	0.1264 (2)	0.0579 (7)	
H4	0.3401	−0.1922	0.1272	0.069*	
C5	0.3159 (2)	−0.0355 (2)	0.20598 (17)	0.0450 (6)	
C6	0.3764 (3)	−0.0885 (3)	0.2995 (2)	0.0630 (8)	
H6A	0.3721	−0.0198	0.3446	0.076*	0.50
H6B	0.4607	−0.1093	0.3029	0.076*	0.50
H6C	0.3352	−0.1704	0.3114	0.076*	0.50
H6D	0.4065	−0.1799	0.2947	0.076*	0.50
H6E	0.3180	−0.0904	0.3364	0.076*	0.50
H6F	0.4434	−0.0292	0.3279	0.076*	0.50
C7	0.4530 (2)	0.5648 (2)	0.63826 (15)	0.0355 (5)	
H7	0.4247	0.4752	0.6404	0.043*	
C8	0.5218 (2)	0.6252 (2)	0.71738 (14)	0.0356 (5)	
C9	0.56482 (19)	0.7597 (2)	0.71563 (14)	0.0366 (5)	
C10	0.5380 (2)	0.8308 (2)	0.63346 (15)	0.0409 (5)	
H10	0.5667	0.9202	0.6313	0.049*	
C11	0.4682 (2)	0.7693 (2)	0.55374 (15)	0.0386 (5)	
H11	0.4502	0.8186	0.4989	0.046*	
C12	0.42502 (19)	0.6361 (2)	0.55453 (14)	0.0334 (5)	
C13	0.3556 (2)	0.5708 (2)	0.46945 (13)	0.0348 (5)	
H13	0.3198	0.6293	0.4211	0.042*	
C14	0.3384 (2)	0.4371 (2)	0.45420 (14)	0.0416 (5)	
H14	0.3665	0.3790	0.5043	0.050*	
C15	0.2784 (2)	0.3701 (2)	0.36480 (14)	0.0357 (5)	
C16	0.5134 (2)	0.4233 (2)	0.80484 (16)	0.0434 (5)	
H16A	0.4254	0.4214	0.7876	0.052*	
H16B	0.5427	0.3880	0.8661	0.052*	
H16C	0.5444	0.3676	0.7629	0.052*	
H2A	0.121 (2)	0.316 (2)	0.0782 (17)	0.038 (7)*	
H1N	0.274 (2)	0.135 (2)	0.2531 (17)	0.036 (6)*	
H2B	0.173 (2)	0.318 (3)	0.181 (2)	0.055 (8)*	
H1O	0.659 (3)	0.896 (3)	0.7919 (19)	0.058 (8)*	
H5A	0.061 (4)	0.348 (5)	−0.087 (2)	0.13 (2)*	
H5B	−0.0448 (18)	0.329 (4)	−0.069 (2)	0.091 (13)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0690 (12)	0.0358 (9)	0.0363 (8)	−0.0158 (9)	−0.0058 (8)	−0.0049 (7)

O2	0.0680 (12)	0.0397 (9)	0.0286 (7)	−0.0130 (8)	−0.0022 (7)	0.0024 (7)
O3	0.0656 (11)	0.0393 (9)	0.0310 (8)	0.0093 (9)	−0.0077 (7)	−0.0041 (7)
O4	0.0623 (10)	0.0338 (8)	0.0308 (8)	0.0009 (8)	−0.0011 (7)	−0.0031 (7)
O5	0.0660 (14)	0.0864 (16)	0.0450 (11)	−0.0116 (13)	−0.0045 (11)	0.0133 (10)
N1	0.0411 (10)	0.0338 (10)	0.0331 (10)	−0.0021 (8)	0.0064 (8)	−0.0079 (8)
N2	0.0603 (14)	0.0447 (11)	0.0302 (10)	0.0074 (10)	−0.0007 (10)	−0.0028 (10)
C1	0.0381 (12)	0.0400 (12)	0.0331 (11)	−0.0055 (10)	0.0054 (9)	−0.0052 (10)
C2	0.0497 (14)	0.0563 (15)	0.0344 (12)	−0.0096 (13)	0.0082 (10)	−0.0123 (11)
C3	0.0592 (16)	0.0570 (17)	0.0514 (15)	−0.0077 (13)	0.0152 (13)	−0.0268 (13)
C4	0.0633 (18)	0.0416 (13)	0.0672 (18)	0.0029 (12)	0.0137 (14)	−0.0182 (13)
C5	0.0450 (15)	0.0340 (11)	0.0523 (14)	−0.0024 (10)	0.0054 (12)	−0.0078 (10)
C6	0.0729 (19)	0.0411 (14)	0.0640 (17)	0.0115 (14)	−0.0035 (14)	0.0022 (13)
C7	0.0441 (12)	0.0287 (10)	0.0307 (9)	−0.0034 (9)	0.0038 (9)	−0.0033 (9)
C8	0.0432 (12)	0.0327 (11)	0.0283 (10)	0.0004 (9)	0.0041 (9)	0.0005 (8)
C9	0.0411 (13)	0.0308 (11)	0.0338 (10)	−0.0014 (10)	0.0016 (9)	−0.0053 (9)
C10	0.0530 (14)	0.0256 (11)	0.0394 (12)	−0.0011 (10)	0.0031 (10)	−0.0001 (9)
C11	0.0505 (13)	0.0321 (11)	0.0291 (10)	0.0057 (10)	0.0025 (9)	0.0035 (8)
C12	0.0379 (12)	0.0305 (10)	0.0297 (10)	0.0046 (9)	0.0047 (9)	−0.0031 (8)
C13	0.0419 (12)	0.0344 (11)	0.0238 (9)	0.0045 (9)	0.0004 (8)	0.0012 (8)
C14	0.0559 (14)	0.0391 (13)	0.0248 (10)	−0.0011 (11)	0.0008 (10)	−0.0005 (9)
C15	0.0441 (12)	0.0346 (11)	0.0253 (10)	−0.0012 (10)	0.0028 (9)	−0.0020 (9)
C16	0.0550 (14)	0.0403 (12)	0.0338 (11)	−0.0027 (11)	0.0091 (10)	0.0045 (10)

Geometric parameters (Å, °)

O1—C9	1.360 (2)	C6—H6B	0.9600
O1—H1O	0.87 (3)	C6—H6C	0.9600
O2—C8	1.375 (3)	C6—H6D	0.9600
O2—C16	1.421 (2)	C6—H6E	0.9600
O3—C15	1.250 (3)	C6—H6F	0.9600
O4—C15	1.258 (3)	C7—C8	1.376 (3)
O5—H5A	0.819 (10)	C7—C12	1.401 (3)
O5—H5B	0.824 (10)	C7—H7	0.9300
N1—C1	1.349 (3)	C8—C9	1.396 (3)
N1—C5	1.361 (3)	C9—C10	1.380 (3)
N1—H1N	0.86 (2)	C10—C11	1.392 (3)
N2—C1	1.330 (3)	C10—H10	0.9300
N2—H2A	0.85 (2)	C11—C12	1.383 (3)
N2—H2B	0.86 (3)	C11—H11	0.9300
C1—C2	1.410 (3)	C12—C13	1.465 (3)
C2—C3	1.352 (4)	C13—C14	1.324 (3)
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.386 (4)	C14—C15	1.493 (3)
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.353 (4)	C16—H16A	0.9600
C4—H4	0.9300	C16—H16B	0.9600
C5—C6	1.491 (4)	C16—H16C	0.9600
C6—H6A	0.9600		
C9—O1—H1O	114.1 (19)	C5—C6—H6F	109.5

C8—O2—C16	116.29 (16)	H6A—C6—H6F	56.3
H5A—O5—H5B	105 (4)	H6B—C6—H6F	56.3
C1—N1—C5	123.7 (2)	H6C—C6—H6F	141.1
C1—N1—H1N	119.1 (16)	H6D—C6—H6F	109.5
C5—N1—H1N	117.2 (16)	H6E—C6—H6F	109.5
C1—N2—H2A	117.2 (16)	C8—C7—C12	121.08 (19)
C1—N2—H2B	118.0 (18)	C8—C7—H7	119.5
H2A—N2—H2B	123 (2)	C12—C7—H7	119.5
N2—C1—N1	118.2 (2)	O2—C8—C7	124.40 (19)
N2—C1—C2	124.1 (2)	O2—C8—C9	115.24 (18)
N1—C1—C2	117.8 (2)	C7—C8—C9	120.35 (19)
C3—C2—C1	119.0 (2)	O1—C9—C10	123.83 (19)
C3—C2—H2	120.5	O1—C9—C8	117.13 (19)
C1—C2—H2	120.5	C10—C9—C8	119.04 (19)
C2—C3—C4	121.2 (2)	C9—C10—C11	120.3 (2)
C2—C3—H3	119.4	C9—C10—H10	119.8
C4—C3—H3	119.4	C11—C10—H10	119.8
C5—C4—C3	120.1 (2)	C12—C11—C10	121.2 (2)
C5—C4—H4	120.0	C12—C11—H11	119.4
C3—C4—H4	120.0	C10—C11—H11	119.4
C4—C5—N1	118.3 (2)	C11—C12—C7	118.00 (19)
C4—C5—C6	125.4 (2)	C11—C12—C13	120.61 (19)
N1—C5—C6	116.3 (2)	C7—C12—C13	121.36 (18)
C5—C6—H6A	109.5	C14—C13—C12	126.75 (18)
C5—C6—H6B	109.5	C14—C13—H13	116.6
H6A—C6—H6B	109.5	C12—C13—H13	116.6
C5—C6—H6C	109.5	C13—C14—C15	126.62 (19)
H6A—C6—H6C	109.5	C13—C14—H14	116.7
H6B—C6—H6C	109.5	C15—C14—H14	116.7
C5—C6—H6D	109.5	O3—C15—O4	123.49 (19)
H6A—C6—H6D	141.1	O3—C15—C14	121.05 (19)
H6B—C6—H6D	56.3	O4—C15—C14	115.43 (19)
H6C—C6—H6D	56.3	O2—C16—H16A	109.5
C5—C6—H6E	109.5	O2—C16—H16B	109.5
H6A—C6—H6E	56.3	H16A—C16—H16B	109.5
H6B—C6—H6E	141.1	O2—C16—H16C	109.5
H6C—C6—H6E	56.3	H16A—C16—H16C	109.5
H6D—C6—H6E	109.5	H16B—C16—H16C	109.5
C5—N1—C1—N2	177.9 (2)	C7—C8—C9—O1	179.5 (2)
C5—N1—C1—C2	−1.3 (3)	O2—C8—C9—C10	178.8 (2)
N2—C1—C2—C3	−179.2 (2)	C7—C8—C9—C10	−0.3 (3)
N1—C1—C2—C3	0.0 (3)	O1—C9—C10—C11	−179.4 (2)
C1—C2—C3—C4	0.8 (4)	C8—C9—C10—C11	0.5 (3)
C2—C3—C4—C5	−0.3 (4)	C9—C10—C11—C12	−0.5 (3)
C3—C4—C5—N1	−1.0 (4)	C10—C11—C12—C7	0.4 (3)
C3—C4—C5—C6	177.5 (3)	C10—C11—C12—C13	−177.7 (2)
C1—N1—C5—C4	1.8 (4)	C8—C7—C12—C11	−0.2 (3)
C1—N1—C5—C6	−176.8 (2)	C8—C7—C12—C13	177.8 (2)
C16—O2—C8—C7	0.1 (3)	C11—C12—C13—C14	159.9 (2)

C16—O2—C8—C9	−179.1 (2)	C7—C12—C13—C14	−18.2 (3)
C12—C7—C8—O2	−178.9 (2)	C12—C13—C14—C15	−173.4 (2)
C12—C7—C8—C9	0.2 (3)	C13—C14—C15—O3	−12.9 (4)
O2—C8—C9—O1	−1.3 (3)	C13—C14—C15—O4	165.1 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O4	0.86 (2)	1.89 (3)	2.746 (2)	175
N2—H2A···O5	0.85 (2)	1.99 (2)	2.815 (3)	166
N2—H2B···O3	0.86 (2)	2.08 (3)	2.934 (4)	173
O1—H1O···O3^{i}	0.87 (3)	1.73 (3)	2.596 (3)	172
O5—H5A···O1^{ii}	0.82 (1)	2.13 (1)	2.938 (5)	171
O5—H5B···O4^{iii}	0.82 (1)	1.97 (1)	2.785 (2)	171
C6—H6A···O5^{iv}	0.96	2.54	3.302 (3)	136
C13—H13···O1^{v}	0.93	2.52	3.332 (2)	147

Symmetry codes: (i) *x*+1/2, −*y*+3/2, *z*+1/2; (ii) *x*−1/2, *y*−1/2, *z*−1; (iii) *x*−1/2, −*y*+1/2, *z*−1/2; (iv) *x*+1/2, −*y*+1/2, *z*+1/2; (v) *x*−1/2, −*y*+3/2, *z*−1/2.

Fig. 1

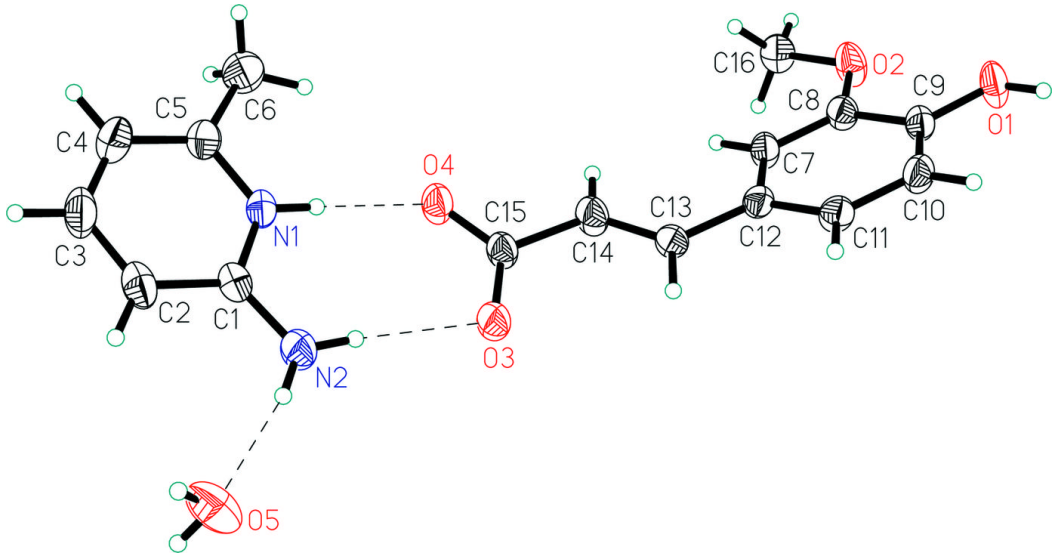
C16
O2
C9
C8
O1
C5
C6
C4
C7
O4
C15
C13
C10
N1
C12
C11
C3
C14
C2
C1
O3
N2
O5

Fig. 2

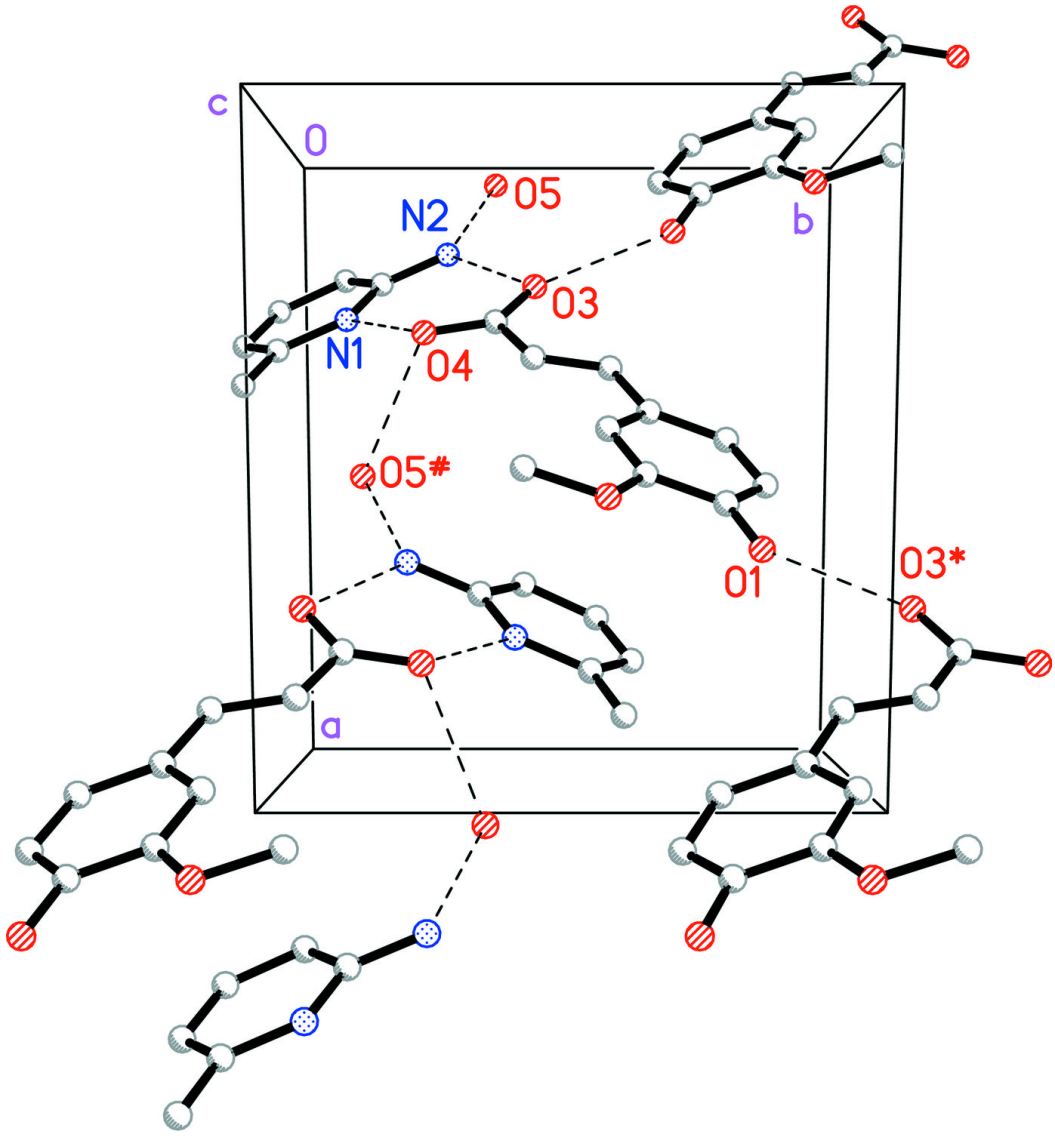

Fig. 3