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3-Hydroxypyridinium 2,5-dihydroxybenzoate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.179
Data-to-parameter ratio = 15.6

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

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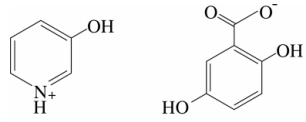
3–Hydroxypyridine forms the title salt, $C_5H_6NO^+\cdot C_7H_5O_4^-$, with 2,5-dihydroxybenzoic acid. Hydrogen bonds in the crystal link the cation and the anion to form a hydrogen-bonded ribbon.

Comment

The title compound, (I), was investigated as part of a study on *D*—H···A hydrogen bonding (*D* = N, O or C; *A* = N or O) in carboxylic acid and pyridine systems (Kashino *et al.*, 2001; Ishida *et al.*, 2001, 2002). 2,5–Dihydroxybenzoic acid is of potential interest in crystal engineering for the formation of specific two- or three-dimensional aggregates with organic bases, because the carboxyl and two hydroxyl groups of the acid may act as hydrogen-bond donors, as well as acceptors. The crystal structure of 2,5-dihydroxybenzoic acid itself was determined by Haisa *et al.* (1982), who found dimorphs of the compound grown from two different solvents. We have found only one example (Aakeröy *et al.*, 1995) of a structure containing the 2,5-dihydroxybenzoic acid moiety in the Cambridge Structural Database (Version 5.24 of November 2002; Allen, 2002), but no crystal data for compounds composed of 2,5-dihydroxybenzoic acid and pyridine derivatives are recorded.



(I)

In the title crystal structure, the 3-hydroxypyridinium cation and the 2,5-dihydroxybenzoate anion are held together by an N—H···O hydrogen bond (Table 2), forming the $C_5H_6NO^+\cdot C_7H_5O_4^-$ unit (Fig. 1). An intramolecular hydrogen bond, O4—H7···O3, is observed in the unit. The units are linked by O—H···O hydrogen bonds, *viz*. O1—H3···O2^{i} between the cation and anion, and O5—H10···O4^{ii} between the anions, to form a hydrogen-bonded ribbon running along the *b* axis [Fig. 2; symmetry codes: (i) $x, 1+y, z$; (ii) $x, y-1, z$]. The dihedral angle between the planes of the benzene and pyridine rings is 3.96 (10)°. Furthermore, C—H···O hydrogen bonds (C2—H2···O2^{i} and C6—H6···O1^{ii}) are formed in the ribbon. These hydrogen bonds play an important role, resulting in the formation of a planar molecular ribbon. A C—H···O hydrogen bond, C5—H5···O5^{iii} [symmetry code: (iii) $1+x, \frac{1}{2}-y, \frac{1}{2}+z$], connects the ribbons to form a two-dimensional hydrogen-bonded network. These planes are stacked along the [102] direction by a π–π-stacking interaction between the pyridine and benzene rings. The interplanar separation and the centroid offset are 3.370 (19) and 2.11 (2) Å, respectively.

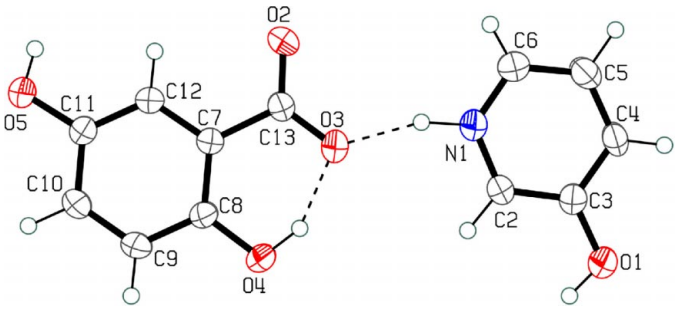


Figure 1
ORTEP-3 (Farrugia, 1997) drawing, showing the atomic numbering of the asymmetric unit of (I). Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. N—H···O and O—H···O hydrogen bonds are indicated by dashed lines.

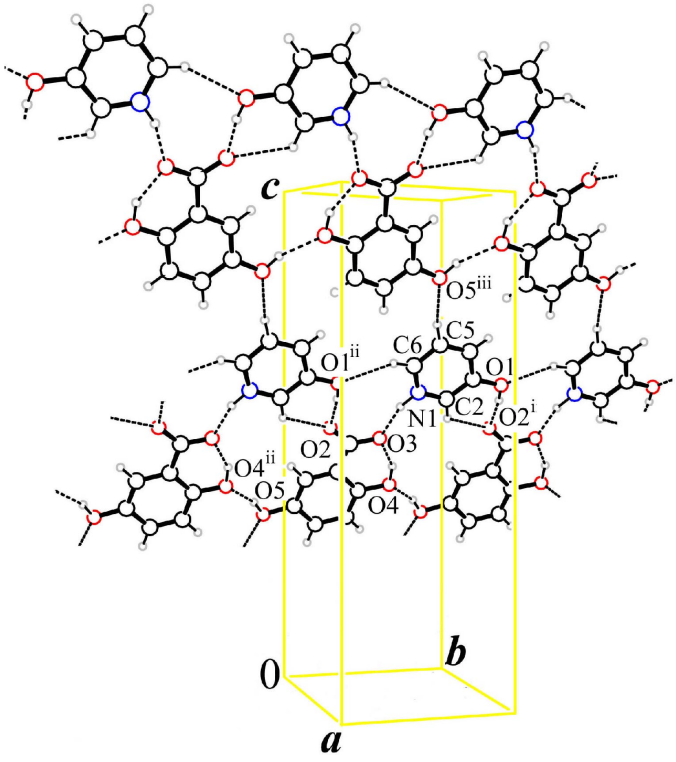


Figure 2
Packing diagram of the molecular ribbons formed *via* N—H···O, O—H···O and C—H···O hydrogen bonds. The symmetry codes are as in Table 2.

Experimental

The crystals were grown by slow evaporation from an ethanol solution of 3-hydroxypyridine and 2,5-dihydroxybenzoic acid, with a molar ratio of 1:1.

Crystal data

$C_5H_6NO^+\cdot C_7H_5O_4^-$
M_r = 249.22
Monoclinic, $P2_1/c$
a = 7.659 (2) Å
b = 7.1731 (17) Å
c = 20.468 (4) Å
β = 95.454 (18)°
V = 1119.4 (5) Å^3
Z = 4
D_x = 1.479 Mg m^{-3}
Mo Kα radiation
Cell parameters from 25 reflections
θ = 11.3–11.5°
μ = 0.12 mm^{-1}
T = 298 K
Plate, colorless
0.50 × 0.27 × 0.21 mm

Data collection

Rigaku AFC-5R diffractometer
ω–2θ scans
Absorption correction: none
4496 measured reflections
3252 independent reflections
1878 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 30.0°
$h = -1 \rightarrow 10$
$k = -1 \rightarrow 10$
$l = -28 \rightarrow 28$
3 standard reflections
every 97 reflections
intensity decay: 2.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.179
S = 1.10
3252 reflections
208 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o) + 0.00203|F_o|^2]$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Extinction correction: Zachariasen (1967)
Extinction coefficient: 1.7 (3) × 10^{-6}

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.334 (3)	C4—C5	1.375 (3)
O2—C13	1.239 (3)	C5—C6	1.369 (3)
O3—C13	1.267 (3)	C7—C8	1.402 (3)
O4—C8	1.363 (3)	C7—C12	1.386 (3)
O5—C11	1.364 (3)	C7—C13	1.500 (3)
N1—C2	1.332 (3)	C8—C9	1.393 (3)
N1—C6	1.345 (3)	C9—C10	1.376 (3)
C2—C3	1.387 (3)	C10—C11	1.394 (3)
C3—C4	1.391 (3)	C11—C12	1.383 (3)
O1—C3—C2	122.5 (2)	O5—C11—C10	117.43 (19)
O1—C3—C4	119.8 (2)	O5—C11—C12	123.28 (19)
C8—C7—C13	120.82 (18)	O2—C13—O3	124.78 (19)
C12—C7—C13	119.88 (18)	O2—C13—C7	117.88 (18)
O4—C8—C7	122.07 (18)	O3—C13—C7	117.33 (18)
O4—C8—C9	118.37 (18)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H3···O2^{i}	0.90 (3)	1.63 (4)	2.530 (2)	171 (3)
O4—H7···O3	0.92 (3)	1.70 (3)	2.533 (2)	150 (3)
O5—H10···O4^{ii}	0.87 (4)	1.93 (4)	2.773 (3)	163 (3)
N1—H1···O3	0.99 (3)	1.70 (3)	2.652 (2)	161 (2)
C2—H2···O2^{i}	0.99 (2)	2.39 (2)	3.048 (3)	123.3 (16)
C5—H5···O5^{iii}	0.99 (3)	2.43 (3)	3.414 (3)	169.2 (19)
C6—H6···O1^{ii}	0.91 (3)	2.42 (3)	3.257 (3)	152 (2)

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

All H atoms were located from a difference Fourier map and refined isotropically, with C—H = 0.91 (3)–1.00 (2) Å, N—H = 0.99 (3) Å and O—H = 0.87 (4)–0.92 (3) Å.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1990); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997–1999); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *TEXSAN for Windows*; molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: *TEXSAN for Windows*.

X-ray measurements were made at the X-ray Laboratory of Okayama University.

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