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3-Aminopyridinium 2-hydroxy-2,2-diphenylacetate monohydrate

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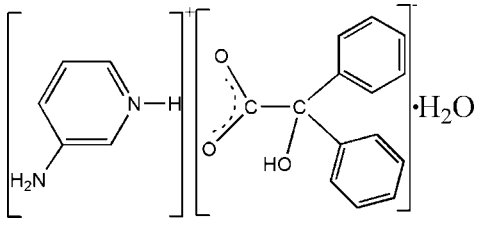
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.086; data-to-parameter ratio = 7.3.

In the title compound, $C_5H_7N_2^+\cdot C_{14}H_{11}O_3^-\cdot H_2O$, the component species are connected by N—H···O and O—H···O hydrogen bonds. An $R_1^2(5)$ ring occurs.

Related literature

For background, see: Zeng *et al.* (2005).



Experimental

Crystal data

$C_5H_7N_2^+\cdot C_{14}H_{11}O_3^-\cdot H_2O$
M_r = 340.37
Orthorhombic, $Pca2_1$
a = 28.903 (6) Å
b = 8.6828 (18) Å
c = 6.7900 (14) Å
V = 1704.0 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.48 × 0.32 × 0.03 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.956, T_{max} = 0.997
6660 measured reflections
1821 independent reflections
1607 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.086
S = 1.08
1821 reflections
250 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O4^{i}	0.826 (10)	1.891 (12)	2.714 (3)	173 (3)
O4—H4A···O2	0.816 (10)	1.918 (12)	2.726 (2)	171 (3)
O4—H4B···O1^{i}	0.820 (10)	1.989 (13)	2.795 (3)	168 (3)
N1—H1A···O2	0.855 (10)	1.827 (13)	2.658 (3)	163 (3)
N1—H1A···O3	0.855 (10)	2.52 (2)	3.070 (3)	123 (2)
N2—H2A···O1^{ii}	0.848 (10)	2.172 (16)	2.975 (3)	158 (3)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2562).

References

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Zeng, Q. D., Wu, D. X., Wang, C., Ma, H. W., Lu, J., Liu, C. M., Xu, S. D., Li, Y. & Bai, C. L. (2005). *Cryst. Growth Des.* **5**, 1889–1896.

  doi:10.1107/S160053680704785X

supplementary materials

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3-Aminopyridinium 2-hydroxy-2,2-diphenylacetate monohydrate

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Comment

Supramolecular hydrogen bonded networks are an important area of current research (Zeng *et al.*, 2005). Herein we report the supramolecular structure of the title compound, (I).

The asymmetric unit of (I) consists of one 2-aminopyridinium cation, one benzylate anion and one crystallization water (Fig. 1), in which 3-aminopyridinium cation interacts with benzylate anion through the doubly intermolecular $N_{protonized}$—*H*···$O_{hydroxyl}$ (N1—H1A···O3) and $N_{protonized}$—*H*···$O_{carboxylate}$ (N1—H1A···O2) hydrogen bonds, and builds a $R_1^2(5)$ ring (Table 1). The crystallization water, in which one of H atoms (H4A) acts as hydrogen bond donor is hydrogen bonded to carboxylate oxygen (O2) of benzylate anion. The other H atom (H4B) of water and hydroxyl H atom (H3A) of benzylate anion linked the adjacent asymmetric units by O_{water}—*H*···$O_{carboxylate}$ and $O_{hydroxyl}$H···O_{water} hydrogen bonds, respectively, into an infinite one-dimensional chain along the direction [001] (Fig. 2). Finally, infinite one-dimensional chains are further extended into a two-dimensional network running parallel to the plane (100) by N_{amino}—*H*···$O_{carboxylate}$ hydrogen bonds (Fig.3).

Experimental

A 5 ml e thanol solution of 3-aminopyridine (1.0 mmol, 0.094 g) was added to 20 ml hot aqueous solution of benzylic acid (1.0 mmol, 0.23 g) and the mixture was stirred for 15 minutes at 373 K. Then the solution was filtered, and the filtrate was kept at the room temperature. After a week, colourless plates of (I) were obtained.

Refinement

Friedel-pair reflections were merged, since anormalous scattering effects were negligible. H atoms bonded to nitrogen atoms, hydroxyl group and water oxygen were located in a difference synthesis and refined isotropically with N—H = 0.85 (1) Å, O—H = 0.82Å and H···H = 1.34 (1) Å, respectively. All the remaining H atoms were placed in calculated positions with C—H = 0.93Å and were refined as riding with $U_{iso} = 1.2U_{eq}$(C).

Figures

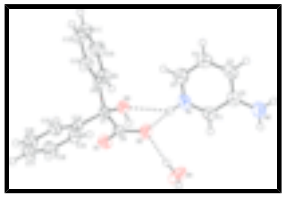

Fig. 1. The molecular structure of (I). Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

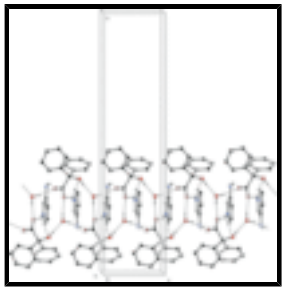

Fig. 2. An infinite one dimensional chain in (I) along the direction [001]. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity.

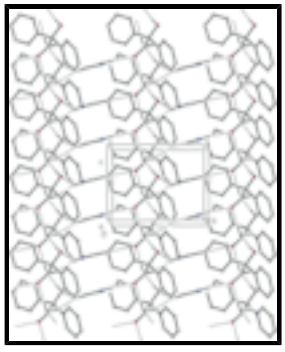

Fig. 3. Two dimensional network running parallel to the plane (100) in (I). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity.

3-Aminopyridinium 2-hydroxy-2,2-diphenylacetate monohydrate

Crystal data

$C_5H_7N_2^+ \cdot C_{14}H_{11}O_3^- \cdot H_2O$	F_{000} = 720
M_r = 340.37	D_x = 1.327 Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2c -2ac	Cell parameters from 2804 reflections
a = 28.903 (6) Å	θ = 2.6–22.3º
b = 8.6828 (18) Å	μ = 0.09 mm^{-1}
c = 6.7900 (14) Å	T = 298 (2) K
V = 1704.0 (6) Å^3	Plate, colourless
Z = 4	0.48 × 0.32 × 0.03 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1821 independent reflections
Radiation source: fine-focus sealed tube	1607 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.028
T = 298(2) K	θ_{max} = 26.0º
ω' scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	h = −35→16
T_{min} = 0.956, T_{max} = 0.997	k = −7→10
6660 measured reflections	l = −8→8

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.086$	$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2 + 0.0365P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} < 0.001$
1821 reflections	$\Delta\rho_{max} = 0.13$ e Å^{-3}
250 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
7 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2$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}
O1	0.17063 (6)	0.2495 (2)	0.7187 (3)	0.0457 (5)
O2	0.20716 (5)	0.4364 (2)	0.8757 (3)	0.0475 (5)
O3	0.13841 (5)	0.48577 (18)	1.1257 (3)	0.0378 (4)
H3A	0.1587 (7)	0.438 (3)	1.186 (4)	0.056 (9)*
O4	0.29689 (6)	0.3420 (3)	0.8495 (3)	0.0616 (6)
H4A	0.2694 (4)	0.363 (3)	0.848 (5)	0.059 (9)*
H4B	0.3028 (11)	0.307 (4)	0.959 (3)	0.062 (10)*
C6	0.17223 (7)	0.3540 (3)	0.8424 (4)	0.0334 (5)
C7	0.12734 (7)	0.3950 (2)	0.9587 (4)	0.0310 (5)
C14	0.10135 (7)	0.2479 (3)	1.0166 (4)	0.0344 (6)
C19	0.09745 (8)	0.2027 (3)	1.2115 (4)	0.0432 (6)
H19	0.1100	0.2637	1.3106	0.052*
C18	0.07485 (9)	0.0665 (3)	1.2595 (5)	0.0551 (8)
H18	0.0726	0.0369	1.3908	0.066*
C17	0.05596 (10)	−0.0240 (3)	1.1177 (6)	0.0618 (9)
H17	0.0413	−0.1157	1.1511	0.074*

C16	0.05879 (10)	0.0216 (3)	0.9236 (6)	0.0588 (8)
H16	0.0457	−0.0392	0.8256	0.071*
C15	0.08091 (8)	0.1569 (3)	0.8737 (5)	0.0452 (6)
H15	0.0821	0.1873	0.7425	0.054*
C8	0.09618 (7)	0.4964 (3)	0.8301 (4)	0.0325 (5)
C13	0.05536 (8)	0.5509 (3)	0.9120 (5)	0.0445 (7)
H13	0.0482	0.5276	1.0422	0.053*
C12	0.02522 (9)	0.6397 (3)	0.8016 (6)	0.0551 (8)
H12	−0.0022	0.6743	0.8576	0.066*
C11	0.03548 (10)	0.6771 (3)	0.6100 (6)	0.0594 (9)
H11	0.0151	0.7370	0.5365	0.071*
C10	0.07602 (10)	0.6254 (3)	0.5276 (5)	0.0542 (8)
H10	0.0833	0.6511	0.3983	0.065*
C9	0.10598 (9)	0.5353 (3)	0.6367 (4)	0.0440 (6)
H9	0.1332	0.5002	0.5792	0.053*
N1	0.22005 (8)	0.7051 (3)	1.0551 (3)	0.0437 (5)
H1A	0.2120 (9)	0.6156 (18)	1.015 (4)	0.051 (8)*
N2	0.32649 (8)	0.9158 (3)	1.1201 (4)	0.0565 (6)
H2A	0.3349 (11)	1.005 (2)	1.157 (6)	0.072 (10)*
H2B	0.3463 (8)	0.843 (2)	1.104 (5)	0.058 (10)*
C1	0.26516 (8)	0.7347 (3)	1.0651 (4)	0.0409 (6)
H1	0.2864	0.6564	1.0415	0.049*
C2	0.28080 (8)	0.8822 (3)	1.1107 (4)	0.0398 (6)
C3	0.24702 (10)	0.9941 (3)	1.1417 (5)	0.0456 (6)
H3	0.2559	1.0947	1.1696	0.055*
C4	0.20121 (9)	0.9583 (3)	1.1315 (5)	0.0497 (7)
H4	0.1791	1.0339	1.1547	0.060*
C5	0.18779 (10)	0.8111 (3)	1.0872 (4)	0.0490 (7)
H5	0.1566	0.7857	1.0797	0.059*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0465 (10)	0.0357 (9)	0.0550 (11)	0.0014 (8)	0.0115 (9)	−0.0106 (9)
O2	0.0306 (8)	0.0482 (11)	0.0635 (13)	−0.0051 (7)	0.0052 (8)	−0.0112 (10)
O3	0.0363 (9)	0.0364 (9)	0.0406 (10)	−0.0005 (7)	−0.0030 (8)	−0.0090 (8)
O4	0.0340 (10)	0.0979 (18)	0.0530 (15)	0.0072 (10)	0.0054 (10)	0.0192 (13)
C6	0.0330 (11)	0.0254 (11)	0.0418 (15)	0.0032 (10)	0.0010 (11)	0.0034 (11)
C7	0.0303 (11)	0.0272 (12)	0.0354 (13)	−0.0011 (9)	0.0003 (10)	−0.0039 (10)
C14	0.0257 (10)	0.0318 (12)	0.0458 (15)	0.0023 (9)	0.0045 (10)	−0.0006 (12)
C19	0.0386 (13)	0.0416 (14)	0.0493 (16)	−0.0028 (11)	0.0037 (12)	0.0042 (14)
C18	0.0568 (16)	0.0475 (17)	0.061 (2)	−0.0075 (14)	0.0100 (15)	0.0128 (15)
C17	0.0579 (17)	0.0372 (15)	0.090 (3)	−0.0154 (13)	0.0123 (19)	0.0087 (17)
C16	0.0597 (18)	0.0397 (15)	0.077 (2)	−0.0144 (14)	0.0030 (16)	−0.0113 (16)
C15	0.0469 (13)	0.0384 (14)	0.0502 (17)	−0.0052 (11)	0.0011 (13)	−0.0033 (13)
C8	0.0296 (11)	0.0231 (11)	0.0448 (14)	−0.0029 (9)	−0.0041 (11)	−0.0049 (11)
C13	0.0356 (13)	0.0389 (13)	0.0590 (18)	0.0016 (11)	0.0024 (12)	−0.0019 (14)
C12	0.0342 (13)	0.0440 (16)	0.087 (3)	0.0088 (12)	−0.0051 (14)	−0.0078 (17)

C11	0.0566 (17)	0.0378 (15)	0.084 (3)	0.0088 (13)	−0.0290 (18)	−0.0016 (17)
C10	0.0696 (19)	0.0459 (16)	0.0470 (18)	0.0066 (15)	−0.0138 (15)	0.0057 (14)
C9	0.0464 (14)	0.0409 (15)	0.0448 (16)	0.0057 (12)	−0.0001 (13)	−0.0017 (13)
N1	0.0516 (13)	0.0386 (13)	0.0409 (13)	−0.0107 (11)	−0.0026 (10)	−0.0010 (11)
N2	0.0500 (14)	0.0500 (16)	0.0696 (17)	−0.0082 (13)	0.0021 (13)	−0.0076 (15)
C1	0.0495 (15)	0.0355 (13)	0.0378 (14)	0.0014 (11)	0.0012 (11)	0.0014 (12)
C2	0.0461 (13)	0.0421 (14)	0.0314 (13)	−0.0053 (12)	0.0012 (12)	−0.0002 (12)
C3	0.0622 (15)	0.0324 (12)	0.0422 (15)	−0.0013 (13)	0.0058 (14)	−0.0028 (13)
C4	0.0539 (15)	0.0450 (16)	0.0502 (16)	0.0059 (13)	0.0034 (14)	0.0017 (14)
C5	0.0473 (14)	0.0541 (16)	0.0456 (17)	−0.0038 (14)	0.0023 (13)	0.0029 (14)

Geometric parameters (Å, °)

O1—C6	1.237 (3)	C13—H13	0.9300
O2—C6	1.258 (3)	C12—C11	1.374 (5)
O3—C7	1.417 (3)	C12—H12	0.9300
O3—H3A	0.826 (10)	C11—C10	1.374 (4)
O4—H4A	0.816 (10)	C11—H11	0.9300
O4—H4B	0.820 (10)	C10—C9	1.382 (4)
C6—C7	1.560 (3)	C10—H10	0.9300
C7—C8	1.533 (3)	C9—H9	0.9300
C7—C14	1.533 (3)	N1—C5	1.328 (4)
C14—C15	1.384 (4)	N1—C1	1.331 (3)
C14—C19	1.384 (4)	N1—H1A	0.855 (10)
C19—C18	1.390 (4)	N2—C2	1.354 (3)
C19—H19	0.9300	N2—H2A	0.848 (10)
C18—C17	1.357 (5)	N2—H2B	0.858 (10)
C18—H18	0.9300	C1—C2	1.393 (4)
C17—C16	1.379 (5)	C1—H1	0.9300
C17—H17	0.9300	C2—C3	1.393 (4)
C16—C15	1.379 (4)	C3—C4	1.361 (4)
C16—H16	0.9300	C3—H3	0.9300
C15—H15	0.9300	C4—C5	1.370 (4)
C8—C9	1.385 (4)	C4—H4	0.9300
C8—C13	1.388 (3)	C5—H5	0.9300
C13—C12	1.384 (4)		
C7—O3—H3A	106 (2)	C8—C13—H13	119.7
H4A—O4—H4B	107 (3)	C11—C12—C13	120.6 (3)
O1—C6—O2	124.7 (2)	C11—C12—H12	119.7
O1—C6—C7	118.69 (19)	C13—C12—H12	119.7
O2—C6—C7	116.5 (2)	C12—C11—C10	119.5 (3)
O3—C7—C8	105.64 (17)	C12—C11—H11	120.3
O3—C7—C14	111.62 (19)	C10—C11—H11	120.3
C8—C7—C14	109.68 (18)	C11—C10—C9	120.1 (3)
O3—C7—C6	110.14 (17)	C11—C10—H10	120.0
C8—C7—C6	109.4 (2)	C9—C10—H10	120.0
C14—C7—C6	110.29 (17)	C10—C9—C8	121.2 (3)
C15—C14—C19	118.2 (2)	C10—C9—H9	119.4
C15—C14—C7	120.3 (2)	C8—C9—H9	119.4

C19—C14—C7	121.4 (2)	C5—N1—C1	123.1 (2)
C14—C19—C18	120.2 (3)	C5—N1—H1A	119.4 (19)
C14—C19—H19	119.9	C1—N1—H1A	117.3 (19)
C18—C19—H19	119.9	C2—N2—H2A	119 (2)
C17—C18—C19	121.0 (3)	C2—N2—H2B	119 (2)
C17—C18—H18	119.5	H2A—N2—H2B	121 (3)
C19—C18—H18	119.5	N1—C1—C2	120.5 (2)
C18—C17—C16	119.2 (3)	N1—C1—H1	119.8
C18—C17—H17	120.4	C2—C1—H1	119.8
C16—C17—H17	120.4	N2—C2—C3	121.8 (2)
C17—C16—C15	120.5 (3)	N2—C2—C1	121.7 (2)
C17—C16—H16	119.8	C3—C2—C1	116.5 (2)
C15—C16—H16	119.8	C4—C3—C2	121.0 (2)
C16—C15—C14	120.8 (3)	C4—C3—H3	119.5
C16—C15—H15	119.6	C2—C3—H3	119.5
C14—C15—H15	119.6	C3—C4—C5	119.9 (3)
C9—C8—C13	118.1 (2)	C3—C4—H4	120.0
C9—C8—C7	124.1 (2)	C5—C4—H4	120.0
C13—C8—C7	117.8 (2)	N1—C5—C4	119.0 (3)
C12—C13—C8	120.5 (3)	N1—C5—H5	120.5
C12—C13—H13	119.7	C4—C5—H5	120.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···O4^{i}	0.826 (10)	1.891 (12)	2.714 (3)	173 (3)
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O4—H4B···O1^{i}	0.820 (10)	1.989 (13)	2.795 (3)	168 (3)
N1—H1A···O2	0.855 (10)	1.827 (13)	2.658 (3)	163 (3)
N1—H1A···O3	0.855 (10)	2.52 (2)	3.070 (3)	123 (2)
N2—H2A···O1^{ii}	0.848 (10)	2.172 (16)	2.975 (3)	158 (3)

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

Fig. 1

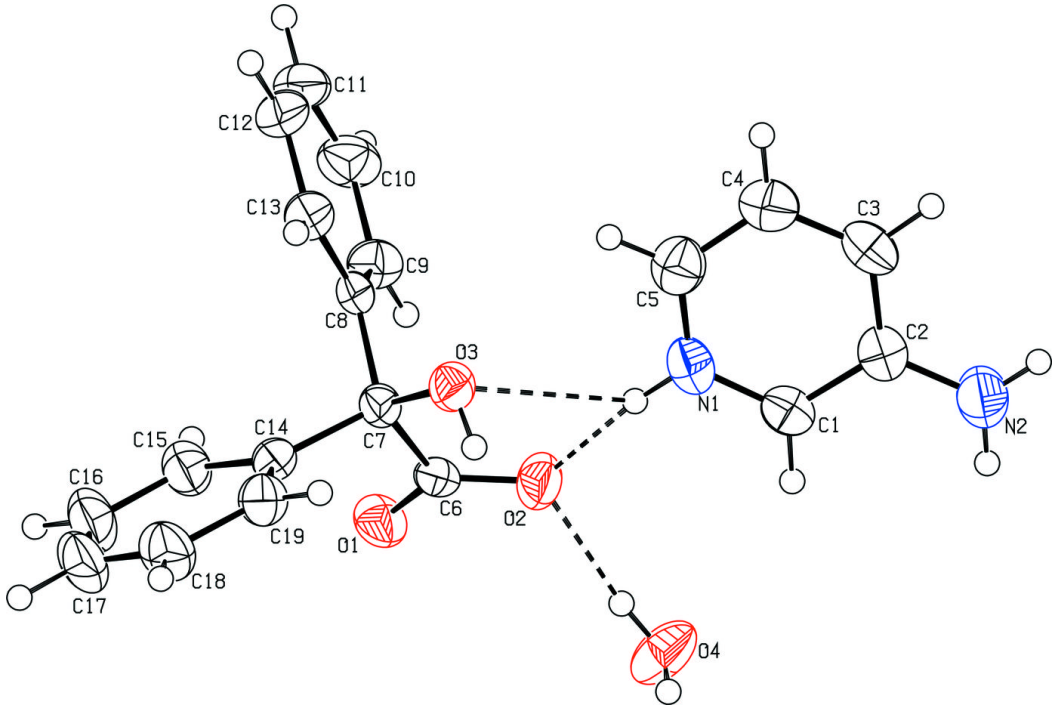
C11
C12
C10
C13
C9
C8
C4
C3
C5
C2
O3
N1
C1
N2
C15
C14
C7
C16
C6
O2
O1
C19
C18
C17
O4

Fig. 2

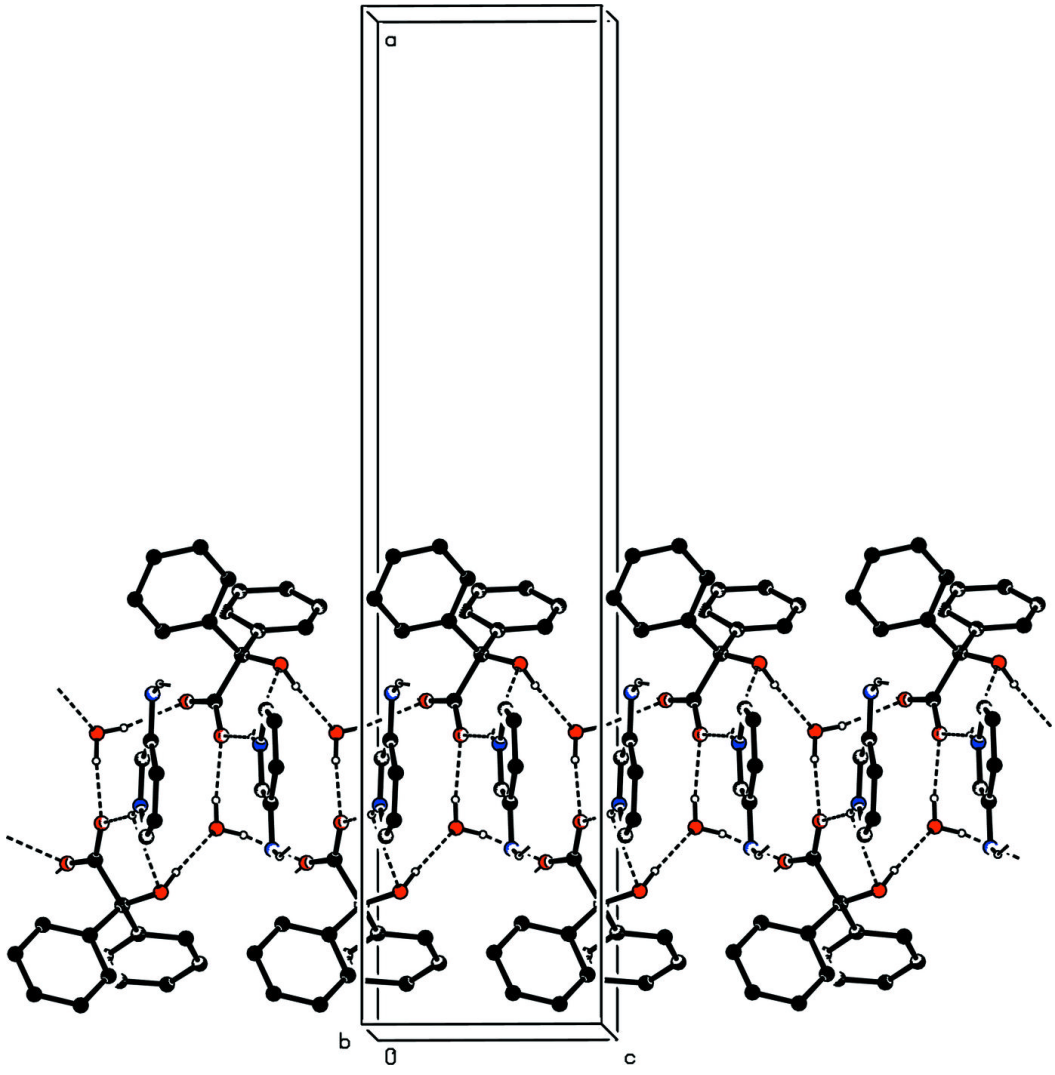

Fig. 3