Crystal structure of 4-hydroxypyridin-1-ium 3,5-dicarboxybenzoate

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The structure of the title salt, $C_5H_6NO^+ \cdot C_9H_5O_6^-$, (**I**), shows that 4-hydroxypyridine has abstracted an H atom from benzene-1,3,5-tricarboxylic acid, yielding a pyridinium cation and carboxylate anion. The two ions form an extensive three-dimensional hydrogen-bonded network throughout the crystal. The hydrogen bonds that comprise the core of the network are considered strong, with O—H···O and N—H···O donor-to-acceptor distances ranging from 2.533 (2) to 2.700 (2) Å. Packing is further enhanced by π-stacking of the cations and anions with like species [centroid–centroid distance = 3.6206 (13) Å].

1. Chemical Context

As a study in crystal engineering utilizing hydrogen bonding between disparate molecules (Desiraju, 2003), we have been investigating the cocrystallization of various pyridine compounds with benzene carboxylic acids (Staun & Oliver, 2012). From previous work, 4-hydroxypyridine undergoes hydrogen migration from the hydroxy O to the pyridine N atom, yielding 4-pyridone (Tyl *et al.*, 2008). We were surprised to find that in the case of 4-hydroxypyridin-1-ium 3,5-dicarboxybenzoate, (**I**), an H atom is abstracted from one carboxylic acid group, yielding a pyridinium salt. This result allows for the hydroxy O and pyridine N atom to both act as hydrogen-bond donors, rather than the donor/acceptor situation of the 4-pyridone species. These two molecules have been incorporated as linker species in metal–organic frameworks (Guo *et al.*, 2011).
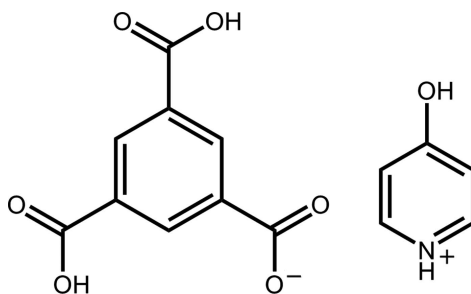
2. Structural Commentary

The structure of (**I**) shows that the 4-hydroxypyridine has abstracted an H atom from the benzenetricarboxylic acid, yielding a pyridinium cation and a carboxylate anion (Fig. 1). Bond distances about the pyridine ring show some localization of the bonds: C1—C2 and C4—C5 are slightly shorter than the ideal aromatic distance [1.367 (3) and 1.369 (3) Å, respec-

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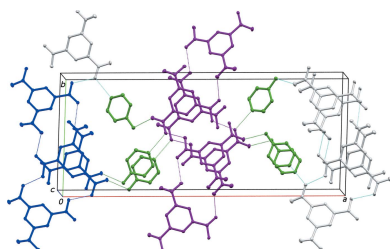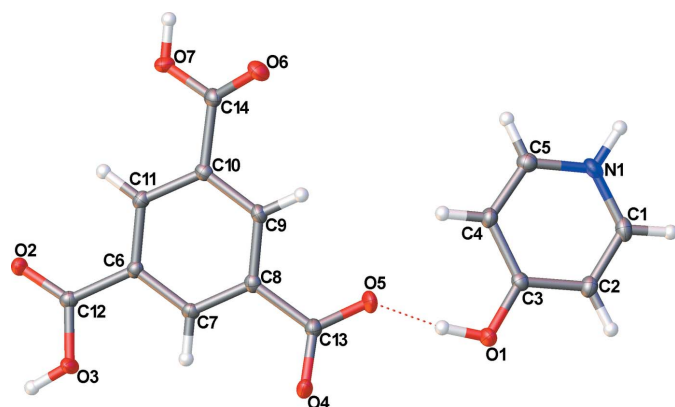
Figure 1
Labeling scheme for (I). Displacement ellipsoids are depicted at the 50% probability level. The inter-ion hydrogen bond is shown as a dashed red line.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1O···O5	0.95 (3)	1.59 (4)	2.533 (2)	171 (3)
N1—H1N···O4^i	0.98 (3)	1.73 (3)	2.700 (2)	173 (4)
O3—H3O···O4^{ii}	0.93 (3)	1.65 (3)	2.574 (2)	172 (3)
O7—H7O···O2^{iii}	0.90 (3)	1.79 (3)	2.678 (2)	166 (3)

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

Table 2
π-stacking interactions within (I).

Interaction	$Cg\cdots Cg$ (Å)	$Cg\cdots$perp (Å)
$Cg1\cdots Cg1^i$	3.6206 (13)	3.4373 (9)
$Cg2\cdots Cg2^i$	3.6206 (13)	3.3627 (9)

$Cg1$ is the centroid of the 3,5-dicarboxybenzoate ring, $Cg2$ is the centroid of the 4-hydroxypyridinium ring [symmetry code: (i) $x, y, 1 + z$], $Cg\cdots Cg$ is the centroid-to-

tively, *cf.* 1.390 Å for an aromatic C—C bond]. The N1—C1 and N1—C5 distances are typical for an aromatic N atom [1.345 (3) and 1.348 (3) Å, respectively]. The remaining bonds within the ring display typical aromatic distances [C2—C3 = 1.405 (3) Å and C3—C4 = 1.402 (3) Å]. The C3—O1 distance of 1.326 (2) Å is typical for a hydroxy O atom bound to an aromatic ring. Bond angles within the pyridine ring are unexceptional.

Two of the three carboxylic acid groups show distinct single- and double-bond character [C12—O3 = 1.305 (3) Å and C14—O7 = 1.332 (3) Å; C12—O2 = 1.224 (2) Å and C14—O6 = 1.204 (3) Å]. The remaining carboxylate group displays C—O bond distances that are similar to each other and indicate delocalization of the C—O bonds [1.268 (3) and 1.249 (2) Å for C13—O4 and C4—O5, respectively], supporting the proposed single negative charge on the benzenetricarboxylic acid molecule. This is further supported by the presence of H atoms, located in a difference Fourier map, on atoms O3 and O7. Bond distances and angles within the benzene ring are as expected.
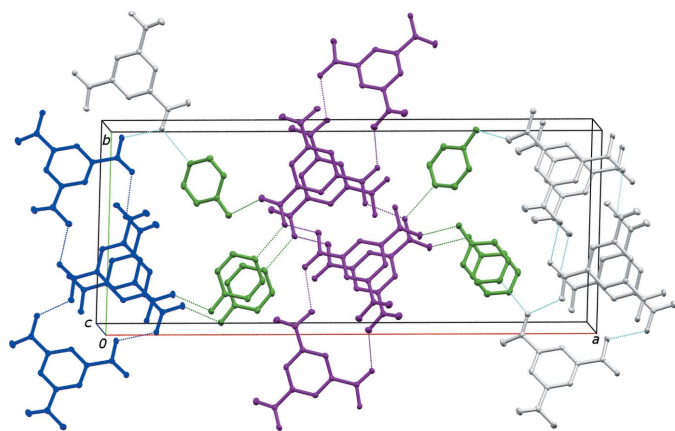
Figure 2
A view of (I) approximately along the crystallographic *c* axis. Color code: blue represents the $R_4^4(28)$ ring, purple the $R_6^6(44)$ ring, and green the bridging 4-hydroxypyridinium cations.

3. Supramolecular Features

The local intermolecular contacts consist of the pyridinium cation forming a hydrogen bond from the hydroxy group to the anionic carboxylate group (O1···O5; see Table 1 for detailed contacts) and from pyridine atom N1 to carboxylate atom O4^i [symmetry code: (i) $-x + \frac{1}{2}, y + \frac{1}{2}, z - \frac{1}{2}$]. Carboxylic acid atoms O3 and O7 are donors for hydrogen bonds to atoms O4^{ii} and O2^{iii}, respectively [symmetry codes: (ii) $-x, -y, z - \frac{1}{2}$; (iii) $-x, -y + 1, z + \frac{1}{2}$]. Since these hydrogen bonds extend over several molecules, an extensive hydrogen-bonded network exists in this structure.

Pertinent features of this extended network are an $R_4^4(28)$ ring comprised of 3,5-dicarboxybenzoate ions (Fig. 2) (Bernstein *et al.*, 1995). The carboxylic acid groups are involved in the hydrogen bonding within this ring. There is also an $R_6^6(44)$ ring of 3,5-dicarboxybenzoate ions, that incorporate a different chain of carboxylic acid groups. These rings are bridged by the 4-hydroxypyridinium cations resulting in the three-dimensional network. The hydrogen bonds within the structure are surprisingly strong, with O—H···O and N—H···O distances ranging from 2.533 (2) to 2.700 (2) Å (Table 1).

The cations and anions form homogeneous π-stacked columns parallel to the *c* axis, that is, 4-pyridinium cations stacking with other cations and 3,5-dicarboxybenzoate anions stacking with other anions. The centroid-to-centroid distances for both the pyridinium and the dicarboxybenzoate inter-actions are 3.6206 (13) Å, *i.e.* the *c*-axis spacing. The centroid-to-perpendicular distances are 3.3629 (9) Å for the cation and 3.4372 (9) Å for the anion. Both measurements are within accepted π–π contact ranges (see Table 2; Spek, 2009).

4. Database Survey

A search of the Cambridge Structural Database (CSD, Version 5.36 plus 3 updates; Groom & Allen, 2014) for 4-hydroxypyridine and benzenetricarboxylic acid gave only five

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_5H_6NO^+ \cdot C_9H_5O_6^-$
M_r	305.24
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	122
a, b, c (Å)	29.3465 (10), 12.2113 (5), 3.6206 (2)
V (Å^3)	1297.47 (10)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.10
Crystal size (mm)	$0.11 \times 0.06 \times 0.06$
Data collection	
Diffractometer	Bruker APEXII
Absorption correction	Numerical (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.694, 0.753
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6000, 2322, 2172
R_{int}	0.018
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.614
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.028, 0.071, 1.05
No. of reflections	2322
No. of parameters	213
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.15, −0.19
Absolute structure	Flack x determined using 786 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.20 (8)

Computer programs: *APEX2* and *SAINT* (Bruker 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009), *Mercury* (Macrae *et al.*, 2008), *POVRay* (Cason, 2003), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

hits. In the compound that is most closely related to the title compound, namely benzene-1,3,5-tricarboxylic acid pyridin-4(1*H*)-one (Campos-Gaxiola *et al.*, 2014), there are three molecules of 4-pyridone present in the asymmetric unit. Benzenetricarboxylic acid and a tetrakis[(pyridin-4-yl-oxy)methyl]methane moiety (incorporating a 4-hydroxy-pyridine functionality) have been utilized in the devlopment of frameworks incorporating copper and cadmium (Guo *et al.*, 2011).

5. Synthesis and Crystallization

To a solution of benzene-1,3,5-tricarboxylic acid (0.035 g, 1.24 mmol) in MeOH (3 ml) in a 20 ml vial was added a solution of 4-hydroxypyridine (0.0218 g, 1.77 mmol) in MeOH (3 ml). The mixture was shaken vigorously, covered with perforated Parafilm and allowed to evaporate slowly over a period of 5 d, yielding colorless rod-like crystals.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Carboxylic, hydroxy, and pyridinium H atoms were initally located in a difference Fourier map. H atoms on the 4-hydroxypyridinium cation were refined freely. H atoms on the carboxylic acid groups were included with refined coordinates and atomic displacement parameters tied to that of the O atom to which they are bonded. C—H hydrogens were included in idealized positions riding on the C atom to which they are bonded, with C—H distances constrained to 0.95 Å and $U_{iso}(H) = 1.2\ U_{eq}(C)$.

The compound is achiral, but crystallizes with a noncentrosymmetric, polar space group. The Flack x parameter refined to 0.20 (8), which suggests the possibility of a small amount of inversion twinnning (Parsons *et al.*, 2013), but the strength of the anomalous signal is very weak. We compared both a model twinned by inversion and the untwinned model, and there was no significant difference. We therefore elected to model the structure without inclusion of a twin component.

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supporting information

Crystal structure of 4-hydroxypyridin-1-ium 3,5-dicarboxybenzoate

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Computing details

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker 2012); data reduction: *SAINT* (Bruker 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009), *Mercury* (Macrae *et al.*, 2008) and POVRay (Cason, 2003); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

4-Hydroxypyridin-1-ium 3,5dicarboxybenzoate

Crystal data

$C_5H_6NO^+ \cdot C_9H_5O_6^-$	$D_x = 1.563$ Mg m^{-3}
$M_r = 305.24$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Orthorhombic, $Pna2_1$	Cell parameters from 2373 reflections
$a = 29.3465$ (10) Å	$\theta = 3.4$–71.2°
$b = 12.2113$ (5) Å	$\mu = 1.10$ mm^{-1}
$c = 3.6206$ (2) Å	$T = 122$ K
$V = 1297.47$ (10) Å^3	Rod, colorless
$Z = 4$	$0.11 \times 0.06 \times 0.06$ mm
$F(000) = 632$	

Data collection

Bruker APEXII diffractometer	6000 measured reflections
Radiation source: Incoatec micro-focus	2322 independent reflections
Detector resolution: 8.33 pixels mm^{-1}	2172 reflections with $I > 2\sigma(I)$
combination of ω and φ–scans	$R_{int} = 0.018$
Absorption correction: numerical *SADABS* (Krause *et al.*, 2015)	$\theta_{max} = 71.2°$, $\theta_{min} = 3.0°$
	$h = -27 \rightarrow 35$
	$k = -13 \rightarrow 14$
$T_{min} = 0.694$, $T_{max} = 0.753$	$l = -4 \rightarrow 4$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.028$	
$wR(F^2) = 0.071$	$w = 1/[\sigma^2(F_o^2) + (0.0441P)^2 + 0.1815P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	
2322 reflections	$(\Delta/\sigma)_{max} = 0.001$
213 parameters	$\Delta\rho_{max} = 0.15$ e Å^{-3}
1 restraint	$\Delta\rho_{min} = -0.19$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack x determined using 786 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Secondary atom site location: difference Fourier map	Absolute structure parameter: 0.20 (8)

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.

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

	x	y	z	$U_{iso}*/U_{eq}$
O1	0.23997 (5)	0.04165 (12)	0.3072 (5)	0.0195 (4)
H1O	0.2112 (11)	0.062 (3)	0.405 (11)	0.057 (11)*
N1	0.32385 (6)	0.30371 (16)	0.1767 (5)	0.0181 (4)
H1N	0.3450 (9)	0.365 (3)	0.166 (11)	0.048 (10)*
C1	0.33796 (7)	0.2029 (2)	0.0796 (6)	0.0186 (5)
H1A	0.3679	0.1933	−0.0157	0.022*
C2	0.30997 (6)	0.11410 (19)	0.1159 (7)	0.0169 (4)
H2A	0.3201	0.0433	0.0444	0.020*
C3	0.26602 (6)	0.12932 (17)	0.2608 (6)	0.0145 (4)
C4	0.25177 (7)	0.23551 (18)	0.3521 (6)	0.0160 (4)
H4A	0.2218	0.2481	0.4424	0.019*
C5	0.28155 (7)	0.32085 (18)	0.3095 (6)	0.0174 (4)
H5A	0.2723	0.3929	0.3741	0.021*
O2	−0.06609 (4)	0.25817 (12)	0.5787 (5)	0.0182 (3)
O3	−0.04180 (5)	0.08670 (13)	0.5070 (5)	0.0215 (4)
H3O	−0.0703 (9)	0.067 (2)	0.417 (9)	0.032*
O4	0.11707 (4)	−0.02696 (12)	0.7027 (5)	0.0196 (4)
O5	0.16718 (4)	0.10740 (13)	0.6111 (6)	0.0252 (4)
O6	0.11934 (5)	0.47723 (13)	1.1519 (5)	0.0220 (4)
O7	0.05337 (5)	0.53129 (13)	0.8992 (5)	0.0204 (4)
H7O	0.0611 (8)	0.598 (3)	0.987 (9)	0.031*
C6	0.01288 (6)	0.21569 (17)	0.6846 (6)	0.0127 (4)
C7	0.04618 (7)	0.13490 (17)	0.6462 (6)	0.0128 (4)
H7A	0.0380	0.0638	0.5628	0.015*
C8	0.09153 (6)	0.15874 (17)	0.7304 (6)	0.0133 (4)
C9	0.10330 (6)	0.26321 (18)	0.8480 (6)	0.0142 (4)
H9A	0.1341	0.2795	0.9056	0.017*
C10	0.07019 (6)	0.34410 (18)	0.8820 (6)	0.0131 (4)
C11	0.02463 (6)	0.32021 (17)	0.8012 (6)	0.0134 (4)
H11A	0.0019	0.3751	0.8261	0.016*
C12	−0.03557 (6)	0.18988 (17)	0.5860 (6)	0.0138 (4)
C13	0.12794 (6)	0.07323 (18)	0.6782 (6)	0.0150 (4)
C14	0.08419 (7)	0.45642 (18)	0.9953 (6)	0.0156 (5)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0143 (7)	0.0151 (8)	0.0291 (9)	−0.0021 (6)	0.0044 (7)	−0.0012 (7)
N1	0.0152 (8)	0.0205 (10)	0.0185 (9)	−0.0067 (8)	−0.0004 (7)	0.0002 (8)

C1	0.0122 (9)	0.0270 (12)	0.0166 (11)	−0.0006 (9)	0.0006 (8)	0.0004 (10)
C2	0.0142 (9)	0.0208 (11)	0.0157 (10)	0.0018 (8)	0.0008 (8)	−0.0017 (9)
C3	0.0133 (9)	0.0166 (11)	0.0135 (10)	−0.0014 (8)	−0.0015 (8)	−0.0005 (9)
C4	0.0132 (9)	0.0180 (11)	0.0170 (10)	0.0010 (8)	0.0016 (8)	−0.0006 (10)
C5	0.0190 (10)	0.0171 (11)	0.0161 (11)	−0.0012 (8)	0.0000 (8)	−0.0007 (9)
O2	0.0116 (6)	0.0130 (7)	0.0299 (9)	0.0009 (5)	−0.0012 (6)	0.0000 (7)
O3	0.0124 (7)	0.0138 (8)	0.0382 (11)	−0.0007 (6)	−0.0073 (7)	−0.0059 (7)
O4	0.0122 (6)	0.0134 (7)	0.0331 (9)	0.0017 (6)	0.0034 (6)	0.0022 (7)
O5	0.0120 (7)	0.0220 (9)	0.0417 (11)	−0.0023 (6)	0.0085 (7)	−0.0032 (8)
O6	0.0179 (7)	0.0204 (8)	0.0277 (9)	−0.0050 (6)	−0.0047 (7)	−0.0032 (7)
O7	0.0179 (8)	0.0126 (8)	0.0308 (10)	0.0003 (6)	−0.0019 (7)	−0.0057 (7)
C6	0.0125 (8)	0.0126 (10)	0.0130 (9)	0.0008 (8)	0.0013 (7)	0.0010 (8)
C7	0.0142 (8)	0.0112 (10)	0.0129 (10)	−0.0014 (7)	0.0007 (7)	0.0018 (8)
C8	0.0128 (9)	0.0143 (10)	0.0129 (10)	−0.0005 (8)	0.0009 (8)	0.0022 (9)
C9	0.0111 (9)	0.0166 (11)	0.0150 (10)	−0.0017 (8)	−0.0008 (8)	0.0002 (9)
C10	0.0139 (9)	0.0136 (10)	0.0117 (10)	−0.0017 (8)	0.0003 (8)	0.0002 (8)
C11	0.0139 (9)	0.0138 (10)	0.0127 (10)	0.0024 (8)	0.0004 (8)	0.0006 (8)
C12	0.0152 (9)	0.0125 (10)	0.0137 (10)	0.0000 (8)	−0.0002 (8)	0.0009 (9)
C13	0.0129 (9)	0.0159 (11)	0.0164 (10)	0.0001 (8)	0.0000 (8)	−0.0007 (9)
C14	0.0139 (9)	0.0168 (11)	0.0162 (11)	−0.0012 (8)	0.0026 (9)	−0.0015 (9)

Geometric parameters (Å, º)

O1—C3	1.326 (2)	O5—C13	1.249 (2)
O1—H1O	0.95 (3)	O6—C14	1.204 (3)
N1—C1	1.345 (3)	O7—C14	1.332 (3)
N1—C5	1.348 (3)	O7—H7O	0.90 (3)
N1—H1N	0.98 (3)	C6—C11	1.388 (3)
C1—C2	1.367 (3)	C6—C7	1.396 (3)
C1—H1A	0.9500	C6—C12	1.499 (3)
C2—C3	1.405 (3)	C7—C8	1.396 (3)
C2—H2A	0.9500	C7—H7A	0.9500
C3—C4	1.402 (3)	C8—C9	1.389 (3)
C4—C5	1.369 (3)	C8—C13	1.506 (3)
C4—H4A	0.9500	C9—C10	1.391 (3)
C5—H5A	0.9500	C9—H9A	0.9500
O2—C12	1.224 (2)	C10—C11	1.399 (3)
O3—C12	1.305 (3)	C10—C14	1.489 (3)
O3—H3O	0.93 (3)	C11—H11A	0.9500
O4—C13	1.268 (3)		
C3—O1—H1O	111 (2)	C6—C7—C8	119.90 (19)
C1—N1—C5	121.26 (19)	C6—C7—H7A	120.1
C1—N1—H1N	119.9 (18)	C8—C7—H7A	120.1
C5—N1—H1N	118.7 (18)	C9—C8—C7	119.70 (18)
N1—C1—C2	121.04 (19)	C9—C8—C13	119.95 (17)
N1—C1—H1A	119.5	C7—C8—C13	120.29 (19)
C2—C1—H1A	119.5	C8—C9—C10	120.39 (18)

C1—C2—C3	118.9 (2)	C8—C9—H9A	119.8
C1—C2—H2A	120.6	C10—C9—H9A	119.8
C3—C2—H2A	120.6	C9—C10—C11	120.1 (2)
O1—C3—C4	123.01 (18)	C9—C10—C14	119.06 (17)
O1—C3—C2	118.02 (19)	C11—C10—C14	120.86 (18)
C4—C3—C2	118.97 (19)	C6—C11—C10	119.51 (18)
C5—C4—C3	119.15 (19)	C6—C11—H11A	120.2
C5—C4—H4A	120.4	C10—C11—H11A	120.2
C3—C4—H4A	120.4	O2—C12—O3	123.40 (18)
N1—C5—C4	120.7 (2)	O2—C12—C6	123.77 (19)
N1—C5—H5A	119.7	O3—C12—C6	112.83 (17)
C4—C5—H5A	119.7	O5—C13—O4	124.62 (19)
C12—O3—H3O	117.1 (18)	O5—C13—C8	116.56 (19)
C14—O7—H7O	110.8 (17)	O4—C13—C8	118.82 (17)
C11—C6—C7	120.42 (17)	O6—C14—O7	124.0 (2)
C11—C6—C12	120.09 (17)	O6—C14—C10	124.07 (19)
C7—C6—C12	119.45 (18)	O7—C14—C10	111.89 (17)
C5—N1—C1—C2	−0.7 (3)	C7—C6—C11—C10	−0.3 (3)
N1—C1—C2—C3	−0.7 (3)	C12—C6—C11—C10	−178.11 (19)
C1—C2—C3—O1	−177.5 (2)	C9—C10—C11—C6	−0.5 (3)
C1—C2—C3—C4	2.1 (3)	C14—C10—C11—C6	177.9 (2)
O1—C3—C4—C5	177.3 (2)	C11—C6—C12—O2	4.8 (4)
C2—C3—C4—C5	−2.2 (3)	C7—C6—C12—O2	−173.0 (2)
C1—N1—C5—C4	0.5 (3)	C11—C6—C12—O3	−175.4 (2)
C3—C4—C5—N1	0.9 (3)	C7—C6—C12—O3	6.7 (3)
C11—C6—C7—C8	0.9 (3)	C9—C8—C13—O5	−25.5 (3)
C12—C6—C7—C8	178.7 (2)	C7—C8—C13—O5	151.8 (2)
C6—C7—C8—C9	−0.7 (3)	C9—C8—C13—O4	154.9 (2)
C6—C7—C8—C13	−178.04 (19)	C7—C8—C13—O4	−27.8 (3)
C7—C8—C9—C10	−0.1 (3)	C9—C10—C14—O6	−20.3 (3)
C13—C8—C9—C10	177.3 (2)	C11—C10—C14—O6	161.3 (2)
C8—C9—C10—C11	0.7 (3)	C9—C10—C14—O7	159.0 (2)
C8—C9—C10—C14	−177.74 (19)	C11—C10—C14—O7	−19.4 (3)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O5	0.95 (3)	1.59 (4)	2.533 (2)	171 (3)
N1—H1N···O4[i]	0.98 (3)	1.73 (3)	2.700 (2)	173 (4)
O3—H3O···O4[ii]	0.93 (3)	1.65 (3)	2.574 (2)	172 (3)
O7—H7O···O2[iii]	0.90 (3)	1.79 (3)	2.678 (2)	166 (3)

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