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Piperazine-1,4-dium bis(2,4,5-tricarboxybenzoate) dihydrate

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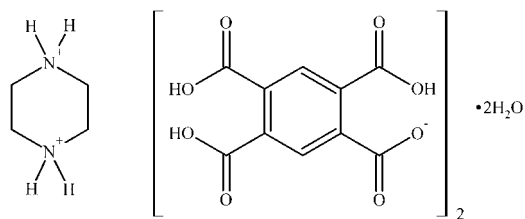
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.030; wR factor = 0.085; data-to-parameter ratio = 10.0.

In the title hydrated salt, $\text{C}_4\text{H}_{12}\text{N}_2^{2+} \cdot 2\text{C}_{10}\text{H}_5\text{O}_8^- \cdot 2\text{H}_2\text{O}$, the piperazinedium cation, lying about an inversion center, adopts a chair conformation. The benzene ring of the anion makes dihedral angles of 25.17 (8°) with the carboxylate group and angles of 8.50 (7°), 20.07 (7°) and 80.86 (8°) with the three carboxylic acid groups. In the crystal, the cations, anions and water molecules are connected by $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds into double layers parallel to (110).

Related literature

For supramolecular architectures involving benzene-1,2,4,5-tetracarboxylic acid and its anions, see: Aghabozorg *et al.* (2006, 2008); Chiwei *et al.* (2005); Pasban *et al.* (2012); Pasdard *et al.* (2010); Smith *et al.* (2008); Smith & Wermuth (2010); Vaidhyanathan *et al.* (2002). For proton-transfer systems, see: Aghabozorg *et al.* (2010). For intermolecular interactions, see: Janiak (2000).



Experimental

Crystal data

 $\text{C}_4\text{H}_{12}\text{N}_2^{2+} \cdot 2\text{C}_{10}\text{H}_5\text{O}_8^- \cdot 2\text{H}_2\text{O}$ $M_r = 630.46$ Triclinic, $P\bar{1}$ $a = 8.2521$ (2) Å $b = 8.4810$ (2) Å $c = 9.6369$ (2) Å $\alpha = 87.117$ (5°) $\beta = 89.527$ (5°) $\gamma = 70.962$ (4°) $V = 636.73$ (3) Å³ $Z = 1$ Mo $K\alpha$ radiation $\mu = 0.14$ mm⁻¹ $T = 293$ K $0.30 \times 0.20 \times 0.20$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2008)

 $T_{\min} = 0.945$, $T_{\max} = 0.985$

11108 measured reflections

2234 independent reflections

2061 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.028$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.030$ $wR(F^2) = 0.085$ $S = 1.06$

2234 reflections

224 parameters

4 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O1}-\text{H1} \cdots \text{O8}$	0.82	1.63	2.4225 (15)	161
$\text{O3}-\text{H3} \cdots \text{O7}^{\text{i}}$	0.82	1.80	2.6100 (13)	167
$\text{O6}-\text{H6} \cdots \text{O2}^{\text{ii}}$	0.82	1.78	2.5884 (13)	170
$\text{N1}-\text{H1A} \cdots \text{O9}^{\text{iii}}$	0.90 (2)	1.83 (2)	2.7283 (17)	176 (2)
$\text{N1}-\text{H1B} \cdots \text{O5}^{\text{iv}}$	0.90 (2)	1.94 (2)	2.7420 (16)	147 (2)
$\text{O9}-\text{H9A} \cdots \text{O8}^{\text{v}}$	0.86 (2)	2.14 (2)	2.9904 (18)	174 (3)
$\text{O9}-\text{H9B} \cdots \text{O7}$	0.82 (2)	2.18 (2)	2.9799 (19)	163 (3)

Symmetry codes: (i) $x, y, z - 1$; (ii) $x + 1, y - 1, z$; (iii) $-x + 1, -y, -z + 1$; (iv) $x, y, z + 1$; (v) $-x, -y + 1, -z + 1$.

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012) and Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: SHELXL97 and DIAMOND (Brandenburg, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YK2086).

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supplementary materials

Acta Cryst. (2013). E69, o574–o575 [doi:10.1107/S160053681300723X]

Piperazine-1,4-dium bis(2,4,5-tricarboxybenzoate) dihydrate

Nagaraju Narayanam, Kranthi Kumar Gangu, Balakrishna Kurra and Saratchandra Babu Mukkamala

Comment

Benzene-1,2,4,5-tetracarboxylic acid (H4btc) with its ability to donate four protons is a versatile ligand in building supramolecular architectures and proton transfer compounds with nitrogen containing organic amines such as piperazine (Aghabozorg *et al.*, 2008; Vaidhyanathan *et al.*, 2002) and diethylenetriamine (Pasban *et al.*, 2012). A number of proton transfer compounds and supramolecular architectures were reported earlier with H4btc and other organic bases such as 1,10-phenanthroline (Chiwei *et al.*, 2005) and propane-1,2-diammonium (Pasdar *et al.*, 2010). These proton transfer compounds have the ability to absorb various metals into their crystal lattices (Aghabozorg *et al.*, 2006) with profound applications in metal separation and storage (Aghabozorg *et al.*, 2010; Smith *et al.*, 2008; Smith & Wermuth, 2010).

We report here one such proton transfer compound, piperazinedium bis(benzene-1,2,4,5-tetracarboxylate) dihydrate. As shown in Fig. 1, the asymmetric unit contain one mono-deprotonated residue of benzene-1,2,4,5-tetracarboxylic acid (H3btc⁻), a half of diprotonated piperazine (pipz) and one water molecule. Layered 2D supramolecular structure of title compound was built by connecting H3btc⁻, pipz and water molecules through hydrogen bonding (Fig.2). Inter- and intramolecular hydrogen bonding was observed between H3btc⁻ molecular species through O–H⁺⋯O hydrogen bonds. Piperazinedium cations and water molecules are involved in building this supramolecular structure through N–H⁺⋯O and O–H⁺⋯O bonds. Besides hydrogen bonding, the weak aromatic π - π stacking interactions between aromatic rings of H3btc⁻ molecules could contribute for further stabilization of this layered supramolecular crystal structure. As shown in Fig. 3, the π - π stacking interactions between H3btc⁻ (intercentroid separation of 3.7954 Å) were found to be in agreement with the reported values (Janiak, 2000). The packing diagram of the title compound is shown in Fig.4.

Experimental

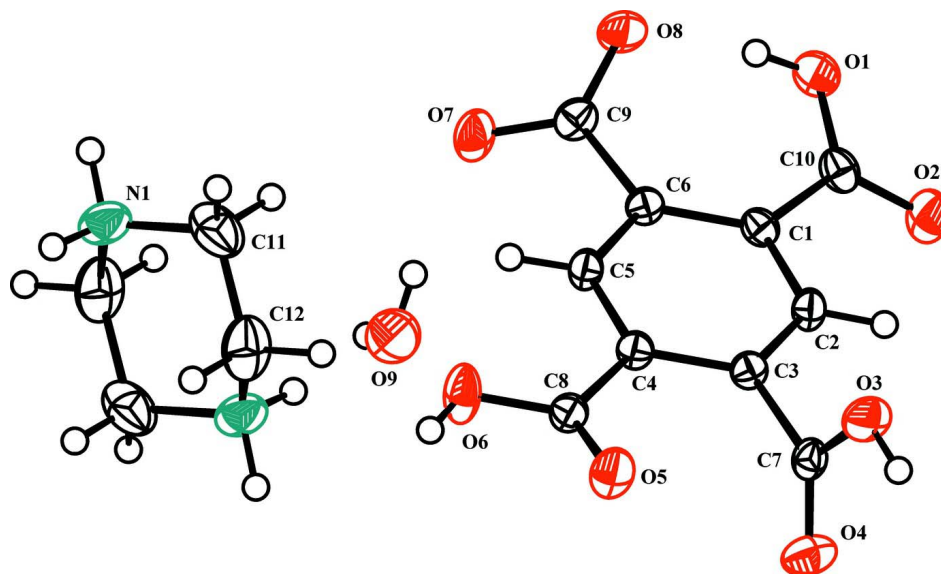
0.2974 g (1.0 mmol) of Zn(NO₃)₂·6H₂O, 0.1270 g (0.5 mmol) of 1,2,4,5-benzenetetracarboxylic acid, 0.2583 g (3 mmol) of piperazine and 0.2 mL (4 mmol) of sulfuric acid were dissolved in 10.0 mL distilled water and heated in a stainless steel Teflon-lined autoclave at 120°C for 24 hours. The mixture was cooled to room temperature at a cooling rate of 6 °/min. Colorless cubic shaped crystals were obtained from the reaction mixture. Yield: 68% (based on H4btc). IR (KBr): 3473.27 (s), 3033.5 (w), 2548 (m), 1707 (s), 1621(s), 1475 (m), 734 (s), 596(s), 471(s).

Refinement

Refinement on F₂ against ALL reflections. The weighted R-factor wR₂ and goodness of fit S were based on F₂ and conventional R-factors R were based on F, with F set to zero for negative F₂. The threshold expression of F₂ > 2sig(F₂) was used only for calculating R factors(gt) etc. and was not relevant to the choice of reflections for refinement. R-factors based on F₂ were statistically about twice as large as those based on F and R- factors based on ALL data will be even larger.

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 2007).

**Figure 1**

The view of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

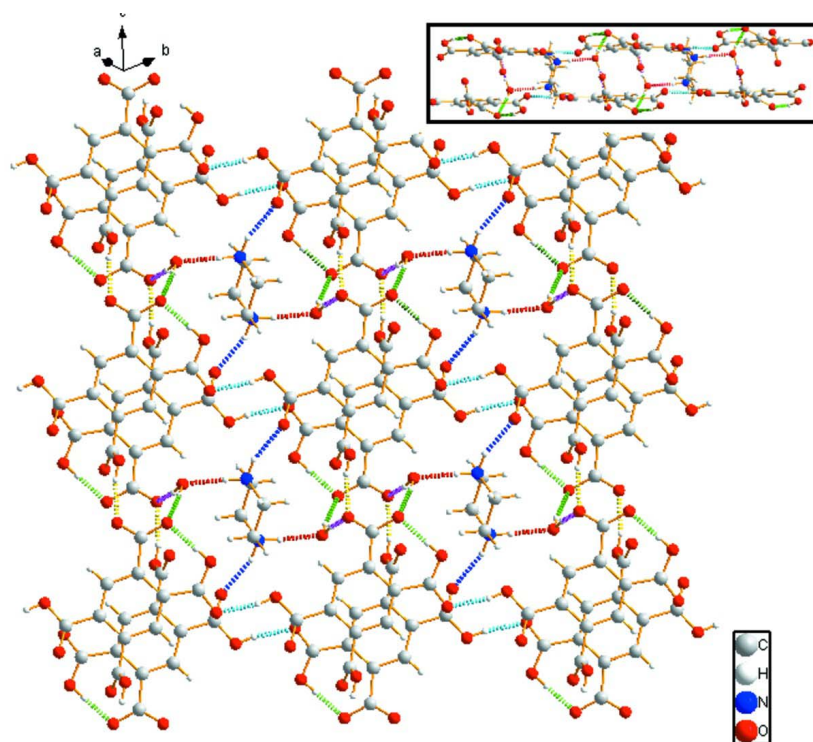


Figure 2

A perspective view of hydrogen bonding interactions represented by dashed lines. Insert: 2D supramolecular representation along *c* axis.

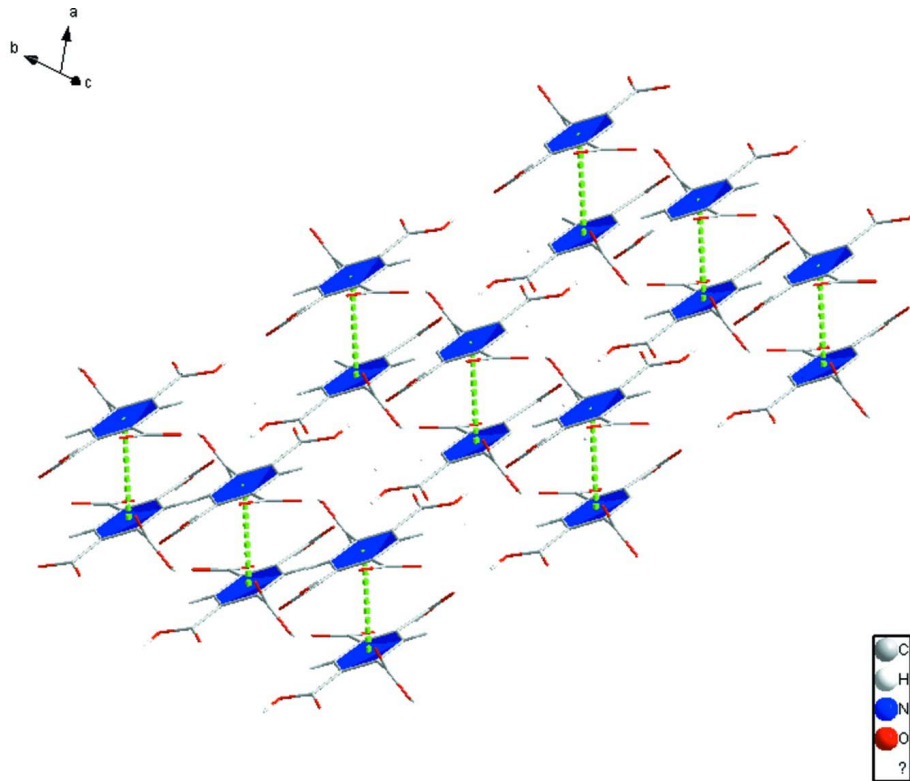
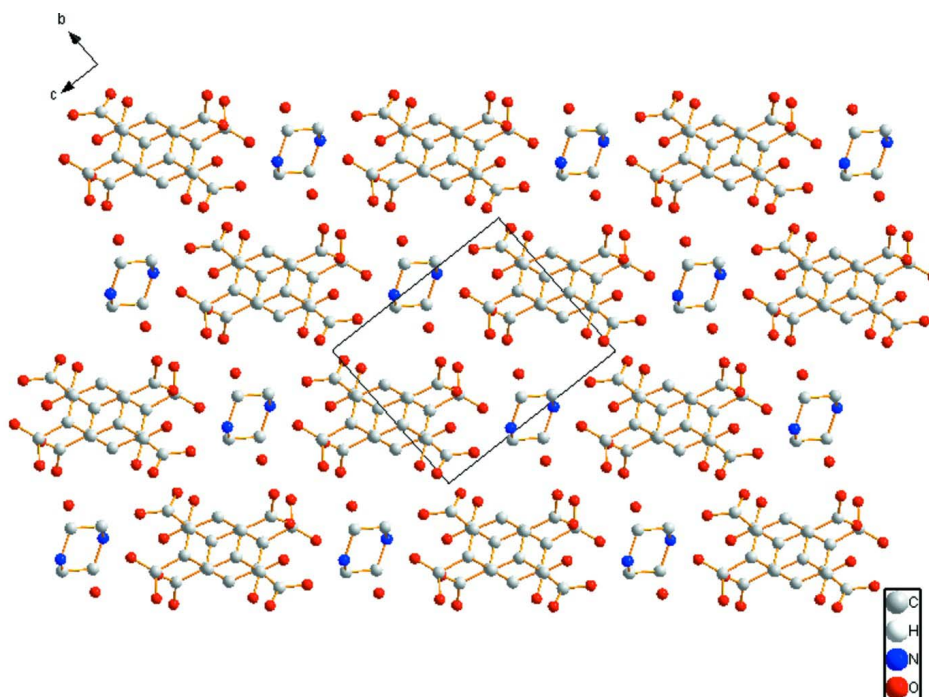


Figure 3

π - π stacking interactions between benzene-1,2,4,5-tetracarboxylates. Cations, water molecules and hydrogen atoms are omitted for clarity.


Figure 4

Crystal packing viewed along a axis. Hydrogen atoms are omitted for clarity.

Piperazine-1,4-dium bis(2,4,5-tricarboxybenzoate) dihydrate

Crystal data

$C_4H_{12}N_2^{2+} \cdot 2C_{10}H_5O_8^{3-} \cdot 2H_2O$

$M_r = 630.46$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 8.2521$ (2) Å

$b = 8.4810$ (2) Å

$c = 9.6369$ (2) Å

$\alpha = 87.117$ (5)°

$\beta = 89.527$ (5)°

$\gamma = 70.962$ (4)°

$V = 636.73$ (3) Å³

$Z = 1$

$F(000) = 328$

$D_x = 1.644$ Mg m⁻³

Melting point: 560 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7490 reflections

$\theta = 2.5$ – 33.0 °

$\mu = 0.14$ mm⁻¹

$T = 293$ K

Block, colourless

$0.30 \times 0.20 \times 0.20$ mm

Data collection

Bruker Kappa APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω and ϕ scan

Absorption correction: multi-scan

(*SADABS*; Bruker, 2008)

$T_{\min} = 0.945$, $T_{\max} = 0.985$

11108 measured reflections

2234 independent reflections

2061 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 25.0$ °, $\theta_{\min} = 2.5$ °

$h = -9 \rightarrow 9$

$k = -9 \rightarrow 10$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.085$

$S = 1.06$

2234 reflections

224 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

$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 0.2345P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kF_c[1 + 0.001xF_c^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.075 (5)

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.06597 (16)	0.70913 (15)	0.05612 (13)	0.0202 (3)
C2	0.08629 (16)	0.66166 (16)	-0.08067 (13)	0.0219 (3)
H2	0.0098	0.7264	-0.1480	0.026*
C3	0.21641 (16)	0.52142 (16)	-0.12023 (13)	0.0196 (3)
C4	0.32899 (16)	0.42177 (15)	-0.01920 (13)	0.0198 (3)
C5	0.30804 (16)	0.46709 (16)	0.11737 (13)	0.0212 (3)
H5	0.3824	0.3995	0.1846	0.025*
C6	0.18074 (16)	0.60918 (16)	0.15847 (13)	0.0200 (3)
C7	0.22375 (16)	0.47981 (16)	-0.27035 (13)	0.0217 (3)
C8	0.47247 (16)	0.27185 (16)	-0.05880 (13)	0.0217 (3)
C9	0.18506 (17)	0.63946 (17)	0.31156 (14)	0.0241 (3)
C10	-0.08355 (17)	0.86608 (16)	0.07812 (14)	0.0244 (3)
C11	0.5751 (2)	0.1289 (2)	0.4758 (2)	0.0448 (4)
H11A	0.6750	0.1621	0.4555	0.054*
H11B	0.4771	0.2293	0.4836	0.054*
C12	0.5451 (2)	0.0307 (2)	0.36090 (17)	0.0436 (4)
N1	0.60207 (17)	0.02802 (17)	0.60844 (14)	0.0371 (3)
O1	-0.08934 (13)	0.95103 (12)	0.18574 (11)	0.0337 (3)
H1	-0.0039	0.9062	0.2342	0.051*
O2	-0.19676 (13)	0.91343 (13)	-0.01098 (11)	0.0368 (3)
O3	0.29608 (14)	0.56784 (13)	-0.34862 (10)	0.0327 (3)
H3	0.2974	0.5411	-0.4293	0.049*
O4	0.16086 (14)	0.38163 (14)	-0.31255 (10)	0.0348 (3)
O5	0.50699 (13)	0.24168 (13)	-0.17891 (10)	0.0337 (3)

O6	0.55721 (13)	0.18159 (13)	0.04674 (10)	0.0373 (3)
H6	0.6341	0.1014	0.0187	0.056*
O7	0.25414 (14)	0.51726 (13)	0.39115 (10)	0.0359 (3)
O8	0.12443 (14)	0.78708 (13)	0.35313 (10)	0.0339 (3)
O9	0.10687 (16)	0.23958 (16)	0.41278 (14)	0.0453 (3)
H12B	0.646 (3)	-0.068 (3)	0.352 (2)	0.051 (5)*
H12A	0.518 (3)	0.099 (3)	0.276 (2)	0.061 (6)*
H1A	0.700 (2)	-0.060 (2)	0.605 (2)	0.046 (5)*
H1B	0.615 (3)	0.092 (3)	0.677 (2)	0.064 (6)*
H9A	0.043 (3)	0.224 (4)	0.479 (2)	0.097 (10)*
H9B	0.135 (4)	0.323 (3)	0.421 (3)	0.106 (11)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0199 (6)	0.0181 (6)	0.0217 (7)	-0.0048 (5)	0.0028 (5)	-0.0015 (5)
C2	0.0211 (6)	0.0216 (6)	0.0193 (6)	-0.0023 (5)	-0.0020 (5)	0.0016 (5)
C3	0.0203 (6)	0.0203 (6)	0.0177 (6)	-0.0061 (5)	0.0017 (5)	-0.0012 (5)
C4	0.0192 (6)	0.0200 (6)	0.0188 (6)	-0.0042 (5)	0.0012 (5)	-0.0016 (5)
C5	0.0216 (6)	0.0216 (6)	0.0172 (6)	-0.0029 (5)	-0.0011 (5)	0.0006 (5)
C6	0.0209 (6)	0.0211 (6)	0.0175 (6)	-0.0062 (5)	0.0021 (5)	-0.0021 (5)
C7	0.0194 (6)	0.0222 (7)	0.0192 (6)	-0.0010 (5)	-0.0010 (5)	-0.0009 (5)
C8	0.0211 (6)	0.0216 (7)	0.0204 (7)	-0.0039 (5)	-0.0003 (5)	-0.0029 (5)
C9	0.0229 (7)	0.0286 (7)	0.0200 (7)	-0.0067 (5)	0.0033 (5)	-0.0047 (5)
C10	0.0233 (7)	0.0205 (7)	0.0262 (7)	-0.0030 (5)	0.0047 (6)	-0.0002 (5)
C11	0.0417 (9)	0.0311 (8)	0.0642 (12)	-0.0163 (7)	-0.0010 (8)	0.0049 (8)
C12	0.0459 (10)	0.0509 (10)	0.0267 (8)	-0.0069 (8)	0.0089 (7)	0.0058 (7)
N1	0.0346 (7)	0.0356 (7)	0.0356 (7)	-0.0016 (6)	-0.0049 (6)	-0.0169 (6)
O1	0.0353 (6)	0.0245 (5)	0.0334 (6)	0.0025 (4)	0.0003 (4)	-0.0098 (4)
O2	0.0298 (6)	0.0326 (6)	0.0350 (6)	0.0084 (4)	-0.0040 (5)	-0.0059 (5)
O3	0.0481 (6)	0.0378 (6)	0.0168 (5)	-0.0202 (5)	0.0040 (4)	-0.0023 (4)
O4	0.0428 (6)	0.0422 (6)	0.0262 (5)	-0.0221 (5)	0.0022 (4)	-0.0100 (4)
O5	0.0354 (6)	0.0339 (6)	0.0210 (5)	0.0042 (4)	0.0023 (4)	-0.0086 (4)
O6	0.0347 (6)	0.0350 (6)	0.0230 (5)	0.0147 (4)	-0.0001 (4)	-0.0012 (4)
O7	0.0478 (7)	0.0342 (6)	0.0168 (5)	-0.0008 (5)	-0.0016 (4)	-0.0016 (4)
O8	0.0418 (6)	0.0303 (6)	0.0258 (5)	-0.0049 (5)	-0.0001 (4)	-0.0127 (4)
O9	0.0401 (7)	0.0411 (7)	0.0463 (8)	-0.0006 (6)	-0.0027 (6)	-0.0112 (6)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.3906 (18)	C10—O2	1.2269 (17)
C1—C6	1.4127 (18)	C10—O1	1.2841 (17)
C1—C10	1.5138 (17)	C11—N1	1.479 (2)
C2—C3	1.3851 (18)	C11—C12	1.487 (3)
C2—H2	0.9300	C11—H11A	0.9700
C3—C4	1.3947 (18)	C11—H11B	0.9700
C3—C7	1.5024 (17)	C12—N1 ⁱ	1.478 (2)
C4—C5	1.3833 (18)	C12—H12B	0.98 (2)
C4—C8	1.4911 (17)	C12—H12A	0.96 (2)
C5—C6	1.3885 (18)	N1—C12 ⁱ	1.478 (2)

C5—H5	0.9300	N1—H1A	0.904 (15)
C6—C9	1.5129 (18)	N1—H1B	0.901 (16)
C7—O4	1.2031 (17)	O1—H1	0.8200
C7—O3	1.3074 (16)	O3—H3	0.8200
C8—O5	1.2096 (16)	O6—H6	0.8200
C8—O6	1.3014 (16)	O9—H9A	0.858 (17)
C9—O7	1.2367 (17)	O9—H9B	0.824 (18)
C9—O8	1.2715 (17)		
C2—C1—C6	118.71 (11)	O8—C9—C6	119.92 (12)
C2—C1—C10	114.30 (11)	O2—C10—O1	120.53 (12)
C6—C1—C10	126.99 (12)	O2—C10—C1	118.69 (12)
C3—C2—C1	122.32 (12)	O1—C10—C1	120.74 (12)
C3—C2—H2	118.8	N1—C11—C12	110.21 (13)
C1—C2—H2	118.8	N1—C11—H11A	109.6
C2—C3—C4	119.08 (12)	C12—C11—H11A	109.6
C2—C3—C7	117.76 (11)	N1—C11—H11B	109.6
C4—C3—C7	123.09 (11)	C12—C11—H11B	109.6
C5—C4—C3	118.88 (11)	H11A—C11—H11B	108.1
C5—C4—C8	120.72 (11)	N1 ⁱ —C12—C11	110.90 (13)
C3—C4—C8	120.38 (11)	N1 ⁱ —C12—H12B	107.1 (11)
C4—C5—C6	122.85 (12)	C11—C12—H12B	109.1 (12)
C4—C5—H5	118.6	N1 ⁱ —C12—H12A	106.8 (12)
C6—C5—H5	118.6	C11—C12—H12A	110.4 (13)
C5—C6—C1	118.16 (11)	H12B—C12—H12A	112.5 (17)
C5—C6—C9	114.29 (11)	C12 ⁱ —N1—C11	110.89 (13)
C1—C6—C9	127.54 (11)	C12 ⁱ —N1—H1A	110.4 (12)
O4—C7—O3	124.71 (12)	C11—N1—H1A	110.1 (12)
O4—C7—C3	122.38 (12)	C12 ⁱ —N1—H1B	109.9 (14)
O3—C7—C3	112.81 (11)	C11—N1—H1B	108.3 (14)
O5—C8—O6	124.18 (12)	H1A—N1—H1B	107.1 (19)
O5—C8—C4	121.94 (12)	C10—O1—H1	109.5
O6—C8—C4	113.86 (11)	C7—O3—H3	109.5
O7—C9—O8	122.61 (12)	C8—O6—H6	109.5
O7—C9—C6	117.42 (11)	H9A—O9—H9B	112 (3)
C6—C1—C2—C3	-0.96 (19)	C4—C3—C7—O4	81.46 (17)
C10—C1—C2—C3	179.89 (12)	C2—C3—C7—O3	80.87 (15)
C1—C2—C3—C4	1.5 (2)	C4—C3—C7—O3	-102.12 (14)
C1—C2—C3—C7	178.63 (12)	C5—C4—C8—O5	-170.29 (13)
C2—C3—C4—C5	-0.59 (19)	C3—C4—C8—O5	7.9 (2)
C7—C3—C4—C5	-177.56 (12)	C5—C4—C8—O6	8.07 (18)
C2—C3—C4—C8	-178.79 (11)	C3—C4—C8—O6	-173.75 (12)
C7—C3—C4—C8	4.23 (19)	C5—C6—C9—O7	-24.00 (18)
C3—C4—C5—C6	-0.9 (2)	C1—C6—C9—O7	157.18 (13)
C8—C4—C5—C6	177.35 (12)	C5—C6—C9—O8	153.59 (13)
C4—C5—C6—C1	1.4 (2)	C1—C6—C9—O8	-25.2 (2)
C4—C5—C6—C9	-177.56 (12)	C2—C1—C10—O2	18.28 (18)
C2—C1—C6—C5	-0.46 (18)	C6—C1—C10—O2	-160.79 (13)

C10—C1—C6—C5	178.57 (12)	C2—C1—C10—O1	-159.58 (12)
C2—C1—C6—C9	178.31 (12)	C6—C1—C10—O1	21.4 (2)
C10—C1—C6—C9	-2.7 (2)	N1—C11—C12—N1 ⁱ	56.82 (19)
C2—C3—C7—O4	-95.55 (16)	C12—C11—N1—C12 ⁱ	-56.81 (19)

Symmetry code: (i) $-x+1, -y, -z+1$.

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O1—H1...O8	0.82	1.63	2.4225 (15)	161
O3—H3...O7 ⁱⁱ	0.82	1.80	2.6100 (13)	167
O6—H6...O2 ⁱⁱⁱ	0.82	1.78	2.5884 (13)	170
N1—H1A...O9 ⁱ	0.90 (2)	1.83 (2)	2.7283 (17)	176 (2)
N1—H1B...O5 ^{iv}	0.90 (2)	1.94 (2)	2.7420 (16)	147 (2)
O9—H9A...O8 ^v	0.86 (2)	2.14 (2)	2.9904 (18)	174 (3)
O9—H9B...O7	0.82 (2)	2.18 (2)	2.9799 (19)	163 (3)

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $x, y, z-1$; (iii) $x+1, y-1, z$; (iv) $x, y, z+1$; (v) $-x, -y+1, -z+1$.