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Benzene-1,2,4,5-tetracarboxylic acid bis(1,3,7-trimethyl-2,3,6,7-tetrahydro-1*H*-purine-2,6-dione)

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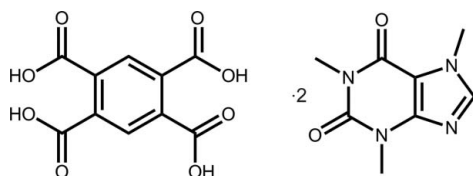
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 Key indicators: single-crystal X-ray study; $T = 98$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.044; wR factor = 0.120; data-to-parameter ratio = 14.7.

The asymmetric unit of the title co-crystal, $\text{C}_{10}\text{H}_6\text{O}_8 \cdot 2\text{C}_8\text{H}_{10}\text{N}_4\text{O}_2$, comprises a centrosymmetric benzene-1,2,4,5-tetracarboxylic acid (LH_4) molecule and a molecule of caffeine in a general position. LH_4 is nonplanar, with the dihedral angles between the ring and pendent carboxylic acid groups being 44.22 (7) and 49.74 (7)°. By contrast, the caffeine molecule is planar (r.m.s. deviation = 0.040 Å). Supramolecular layers parallel to $(\bar{1}\bar{1}0)$ are sustained by carboxylic acid $\text{O}-\text{H} \cdots \text{O}(\text{carbonyl})$ and $\text{O}-\text{H} \cdots \text{N}(\text{imidazole})$ hydrogen bonds, as well as by methyl-carbonyl $\text{C}-\text{H} \cdots \text{O}$ interactions. These stack *via* $\pi-\pi$ interactions between the benzene and imidazole rings [inter-centroid distance = 3.4503 (10) Å].

Related literature

For cocrystallization studies with benzene-1,2,4,5-tetracarboxylic acid, see: Arman & Tiekink (2013).



Experimental

Crystal data

 $\text{C}_{10}\text{H}_6\text{O}_8 \cdot 2\text{C}_8\text{H}_{10}\text{N}_4\text{O}_2$
 $M_r = 642.55$

 Triclinic, $P\bar{1}$
 $a = 7.4570$ (15) Å

 $b = 9.0490$ (15) Å
 $c = 11.782$ (2) Å
 $\alpha = 68.800$ (11)°
 $\beta = 81.124$ (13)°
 $\gamma = 73.441$ (9)°
 $V = 709.3$ (2) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 0.12$ mm⁻¹
 $T = 98$ K
 $0.55 \times 0.30 \times 0.25$ mm

Data collection

 Rigaku AFC12 Kappa/
 SATURN724 diffractometer
 Absorption correction: multi-scan
 (*ABSCOR*; Higashi, 1995)
 $T_{\text{min}} = 0.838$, $T_{\text{max}} = 1$

 4935 measured reflections
 3213 independent reflections
 3006 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 Standard reflections: 0

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.120$
 $S = 1.04$
 3213 reflections
 218 parameters
 2 restraints

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.37$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O}2-\text{H}1\text{O} \cdots \text{N}3^i$	0.86 (1)	1.83 (1)	2.6774 (17)	171 (2)
$\text{O}4-\text{H}2\text{O} \cdots \text{O}5$	0.84 (2)	1.84 (2)	2.6571 (15)	162 (2)
$\text{C}12-\text{H}12\text{B} \cdots \text{O}6^{\text{ii}}$	0.98	2.30	3.239 (2)	159

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

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP II* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5340).

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supporting information

Acta Cryst. (2013). E69, o1443 [doi:10.1107/S1600536813022563]

Benzene-1,2,4,5-tetracarboxylic acid bis(1,3,7-trimethyl-2,3,6,7-tetrahydro-1H-purine-2,6-dione)

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S1. Comment

The title co-crystal was investigated in continuation of recent structural studies of the products obtained from the co-crystallization of benzene-1,2,4,5-tetracarboxylic acid (LH_4) with various pyridyl-containing molecules (Arman & Tiekink, 2013).

The asymmetric unit comprises half a molecule of LH_4 , being disposed about a centre of inversion, and a molecule of caffeine in a general position, Fig. 1. Twists are evident in LH_4 as seen in the dihedral angles of 44.22 (7) and 49.74 (7)° formed, respectively, between the O1- and O3-carboxylic acids and the benzene ring to which they are attached. The 14 non-hydrogen atoms of the caffeine molecule are co-planar with an r.m.s. deviation = 0.040 Å.

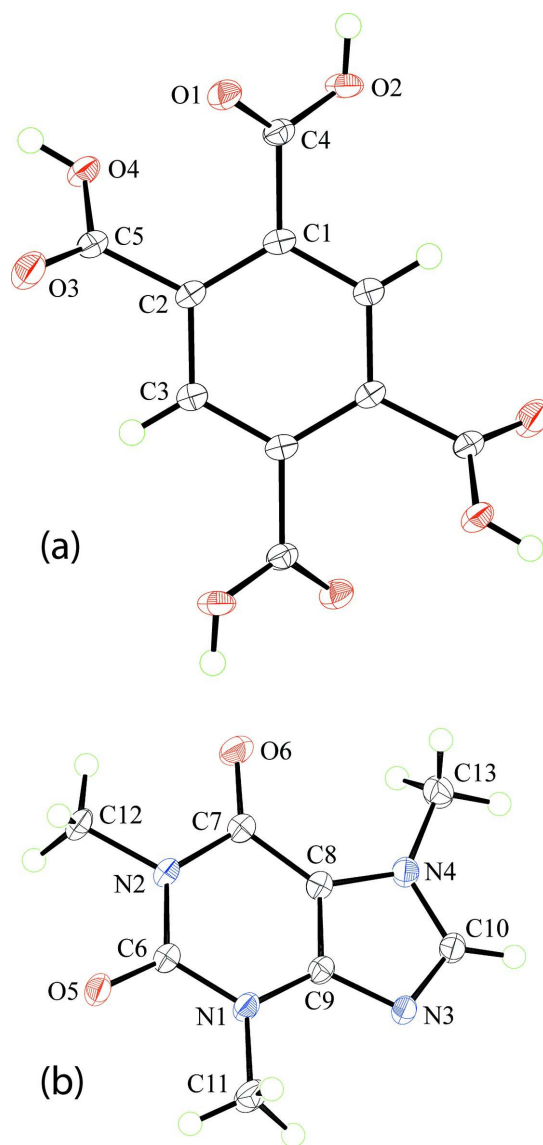
In the crystal packing, the HL_4 molecule forms two O2—H···O5-carbonyl and two O4—H···N3-imidazole hydrogen bonds to form a supramolecular chain constructed about centrosymmetric 26-membered $\{\cdots HO-C_4OH\cdots OCNCN\}_2$ synthons, Table 1. Chains are connected into a layer approximately parallel to $(\bar{1}10)$ by methyl-C12—H···O6(carbonyl) interactions *via* centrosymmetric and 10-membered $\{\cdots OCNCH\}_2$ synthons, Fig. 2. Layers are connected into a three-dimensional architecture by π - π interactions between the benzene and imidazolyl rings [inter-centroid distance = 3.4503 (10) Å, angle of inclination = 9.54 (7)° for symmetry operation $x, y, 1 + z$], Fig. 3.

S2. Experimental

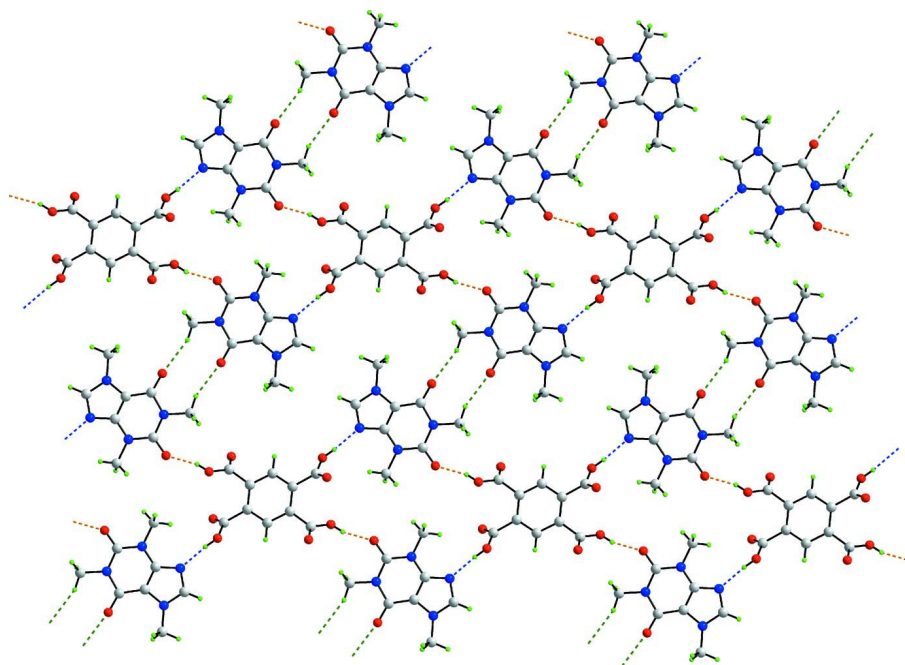
Crystals of (I) were obtained by the co-crystallization of caffeine (Sigma–Aldrich, 0.08 mmol) and benzene-1,2,4,5-tetracarboxylic acid (Sigma–Aldrich, 0.09 mmol) in acetone solution. Crystals were obtained by slow evaporation.

S3. Refinement

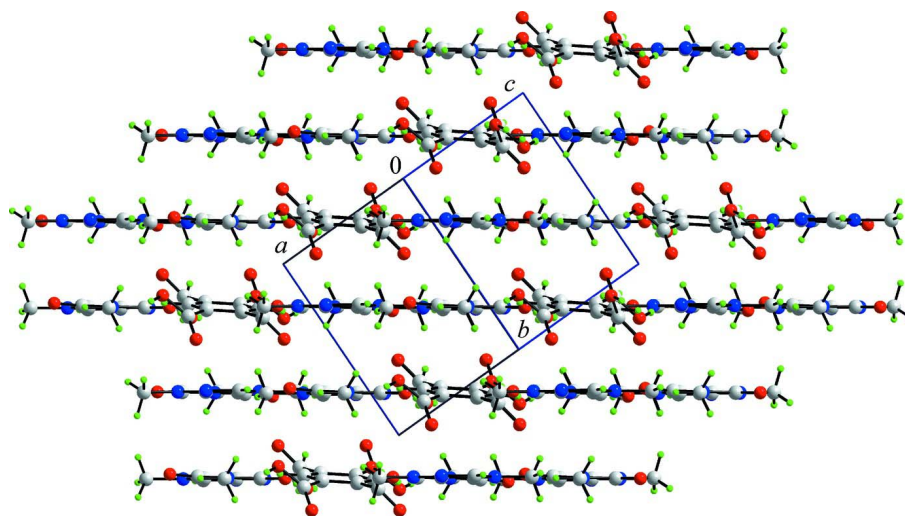
C-bound H atoms were placed in calculated positions (C—H = 0.95–0.98 Å) and were included in the refinement in the riding-model approximation, with $U_{iso}(H)$ set to 1.2–1.5 $U_{eq}(C)$. The O- and N-bound H atoms were located in a difference Fourier map and were refined with distance restraints of O—H = 0.84 (1) Å and N—H = 0.88 (1) Å, and with $U_{iso}(H) = 1.2U_{eq}(N)$ and 1.5 $U_{eq}(O)$.

**Figure 1**

Molecular structures of the components of (I), showing atom-labelling scheme and displacement ellipsoids at the 50% probability level. The unlabelled atoms of HL_4 (a) are generated by the symmetry operation $1 - x, 2 - y, 2 - z$.

**Figure 2**

Views of the supramolecular layer in (I). The O—H...O (orange), O—H...N (blue) and C—H...O (green) interactions are shown as dashed lines.

**Figure 3**

Unit-cell contents in (I) highlighting the stacking of layers shown in Fig. 2.

Benzene-1,2,4,5-tetracarboxylic acid bis(1,3,7-trimethyl-2,3,6,7-tetrahydro-1*H*-purine-2,6-dione)

Crystal data

$C_{10}H_6O_8 \cdot 2C_8H_{10}N_4O_2$

$M_r = 642.55$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.4570$ (15) Å

$b = 9.0490$ (15) Å

$c = 11.782$ (2) Å

$\alpha = 68.800$ (11)°

$\beta = 81.124$ (13)°

$\gamma = 73.441$ (9)°

$V = 709.3 (2) \text{ \AA}^3$
 $Z = 1$
 $F(000) = 334$
 $D_x = 1.504 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 2842 reflections

$\theta = 3.3\text{--}40.4^\circ$
 $\mu = 0.12 \text{ mm}^{-1}$
 $T = 98 \text{ K}$
 Block, colourless
 $0.55 \times 0.30 \times 0.25 \text{ mm}$

Data collection

Rigaku AFC12 Kappa/SATURN724
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.838, T_{\max} = 1$

4935 measured reflections
 3213 independent reflections
 3006 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\max} = 27.5^\circ, \theta_{\min} = 1.9^\circ$
 $h = -9 \rightarrow 9$
 $k = -11 \rightarrow 11$
 $l = -14 \rightarrow 15$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.120$
 $S = 1.04$
 3213 reflections
 218 parameters
 2 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
 $w = 1/[\sigma^2(F_o^2) + (0.0679P)^2 + 0.3025P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.37 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.33 \text{ e \AA}^{-3}$

Special details

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.10476 (13)	0.94888 (12)	0.88177 (9)	0.0215 (2)
O2	0.05750 (13)	1.21613 (12)	0.85531 (9)	0.0216 (2)
H1O	-0.0516 (16)	1.226 (2)	0.8350 (17)	0.032*
O3	0.56478 (16)	0.70162 (12)	0.84529 (9)	0.0283 (3)
O4	0.43724 (14)	0.95115 (12)	0.71641 (8)	0.0202 (2)
H2O	0.430 (3)	0.902 (2)	0.6695 (17)	0.051 (6)*
O5	0.35494 (13)	0.85084 (12)	0.54930 (8)	0.0225 (2)
O6	0.84545 (14)	0.50918 (14)	0.40808 (10)	0.0283 (2)
N1	0.30711 (15)	0.79348 (14)	0.38575 (10)	0.0189 (2)
N2	0.60054 (15)	0.68600 (13)	0.47529 (10)	0.0175 (2)

N3	0.29341 (15)	0.71840 (13)	0.20867 (10)	0.0180 (2)
N4	0.58825 (16)	0.56529 (14)	0.20875 (10)	0.0191 (2)
C1	0.33603 (17)	1.03685 (15)	0.94094 (11)	0.0162 (3)
C2	0.49837 (17)	0.92882 (15)	0.91321 (11)	0.0159 (2)
C3	0.66122 (18)	0.89199 (15)	0.97280 (11)	0.0168 (3)
H3	0.7709	0.8179	0.9544	0.020*
C4	0.15397 (18)	1.06201 (16)	0.88843 (11)	0.0174 (3)
C5	0.50294 (18)	0.84709 (16)	0.82237 (11)	0.0179 (3)
C6	0.41768 (18)	0.78078 (16)	0.47369 (11)	0.0178 (3)
C7	0.68657 (18)	0.59678 (16)	0.39564 (12)	0.0187 (3)
C8	0.56313 (18)	0.62252 (15)	0.30563 (11)	0.0174 (3)
C9	0.38190 (18)	0.71494 (15)	0.30277 (11)	0.0167 (3)
C10	0.42496 (19)	0.62623 (16)	0.15447 (12)	0.0193 (3)
H10	0.4046	0.6060	0.0844	0.023*
C11	0.1111 (2)	0.8869 (2)	0.38356 (14)	0.0312 (3)
H11A	0.0737	0.9370	0.2991	0.047*
H11B	0.0969	0.9727	0.4189	0.047*
H11C	0.0316	0.8135	0.4312	0.047*
C12	0.71544 (19)	0.68114 (17)	0.56767 (12)	0.0217 (3)
H12A	0.7238	0.7922	0.5549	0.033*
H12B	0.8414	0.6118	0.5607	0.033*
H12C	0.6579	0.6362	0.6491	0.033*
C13	0.7597 (2)	0.46116 (18)	0.17183 (13)	0.0251 (3)
H13A	0.7855	0.3524	0.2344	0.038*
H13B	0.8650	0.5102	0.1623	0.038*
H13C	0.7433	0.4514	0.0941	0.038*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0199 (5)	0.0252 (5)	0.0241 (5)	-0.0084 (4)	-0.0032 (4)	-0.0109 (4)
O2	0.0180 (5)	0.0209 (5)	0.0262 (5)	-0.0038 (4)	-0.0101 (4)	-0.0055 (4)
O3	0.0414 (6)	0.0210 (5)	0.0249 (5)	-0.0036 (4)	-0.0113 (4)	-0.0099 (4)
O4	0.0250 (5)	0.0225 (5)	0.0154 (4)	-0.0064 (4)	-0.0052 (3)	-0.0072 (4)
O5	0.0203 (5)	0.0306 (5)	0.0201 (5)	-0.0043 (4)	-0.0040 (4)	-0.0129 (4)
O6	0.0192 (5)	0.0350 (6)	0.0301 (5)	0.0028 (4)	-0.0086 (4)	-0.0147 (4)
N1	0.0148 (5)	0.0249 (6)	0.0188 (5)	-0.0036 (4)	-0.0036 (4)	-0.0093 (4)
N2	0.0159 (5)	0.0215 (5)	0.0163 (5)	-0.0054 (4)	-0.0039 (4)	-0.0059 (4)
N3	0.0194 (5)	0.0191 (5)	0.0172 (5)	-0.0051 (4)	-0.0042 (4)	-0.0065 (4)
N4	0.0196 (5)	0.0205 (5)	0.0193 (5)	-0.0056 (4)	-0.0017 (4)	-0.0083 (4)
C1	0.0166 (6)	0.0174 (6)	0.0148 (5)	-0.0062 (5)	-0.0038 (4)	-0.0030 (4)
C2	0.0186 (6)	0.0170 (5)	0.0140 (5)	-0.0067 (5)	-0.0030 (4)	-0.0048 (4)
C3	0.0173 (6)	0.0175 (5)	0.0163 (5)	-0.0050 (4)	-0.0029 (4)	-0.0049 (4)
C4	0.0167 (6)	0.0227 (6)	0.0139 (5)	-0.0064 (5)	-0.0019 (4)	-0.0060 (5)
C5	0.0184 (6)	0.0206 (6)	0.0174 (6)	-0.0061 (5)	-0.0036 (4)	-0.0075 (5)
C6	0.0175 (6)	0.0205 (6)	0.0163 (6)	-0.0067 (5)	-0.0023 (4)	-0.0050 (5)
C7	0.0180 (6)	0.0203 (6)	0.0180 (6)	-0.0056 (5)	-0.0022 (5)	-0.0057 (5)
C8	0.0178 (6)	0.0187 (6)	0.0161 (6)	-0.0052 (5)	-0.0015 (5)	-0.0056 (5)

C9	0.0163 (6)	0.0183 (6)	0.0164 (5)	-0.0064 (5)	-0.0022 (4)	-0.0046 (4)
C10	0.0228 (6)	0.0189 (6)	0.0176 (6)	-0.0064 (5)	-0.0042 (5)	-0.0058 (5)
C11	0.0177 (7)	0.0470 (9)	0.0312 (7)	0.0037 (6)	-0.0071 (5)	-0.0225 (7)
C12	0.0188 (6)	0.0291 (7)	0.0198 (6)	-0.0061 (5)	-0.0065 (5)	-0.0090 (5)
C13	0.0213 (6)	0.0279 (7)	0.0291 (7)	-0.0035 (5)	-0.0007 (5)	-0.0154 (6)

Geometric parameters (Å, °)

O1—C4	1.2123 (16)	C1—C3 ⁱ	1.3911 (17)
O2—C4	1.3176 (16)	C1—C2	1.4008 (18)
O2—H10	0.855 (9)	C1—C4	1.5030 (17)
O3—C5	1.2057 (17)	C2—C3	1.3935 (17)
O4—C5	1.3270 (15)	C2—C5	1.4968 (17)
O4—H2O	0.843 (9)	C3—C1 ⁱ	1.3911 (17)
O5—C6	1.2349 (16)	C3—H3	0.9500
O6—C7	1.2197 (17)	C7—C8	1.4236 (17)
N1—C9	1.3713 (17)	C8—C9	1.3698 (18)
N1—C6	1.3747 (16)	C10—H10	0.9500
N1—C11	1.4638 (17)	C11—H11A	0.9800
N2—C6	1.3875 (17)	C11—H11B	0.9800
N2—C7	1.4181 (17)	C11—H11C	0.9800
N2—C12	1.4671 (15)	C12—H12A	0.9800
N3—C10	1.3384 (17)	C12—H12B	0.9800
N3—C9	1.3618 (16)	C12—H12C	0.9800
N4—C10	1.3410 (17)	C13—H13A	0.9800
N4—C8	1.3829 (16)	C13—H13B	0.9800
N4—C13	1.4664 (17)	C13—H13C	0.9800
C4—O2—H10	110.9 (13)	O6—C7—N2	121.91 (12)
C5—O4—H2O	111.5 (16)	O6—C7—C8	126.89 (13)
C9—N1—C6	119.13 (11)	N2—C7—C8	111.20 (11)
C9—N1—C11	121.00 (11)	C9—C8—N4	105.48 (11)
C6—N1—C11	119.85 (11)	C9—C8—C7	123.20 (12)
C6—N2—C7	126.45 (11)	N4—C8—C7	131.32 (12)
C6—N2—C12	116.05 (11)	N3—C9—C8	111.54 (11)
C7—N2—C12	117.50 (11)	N3—C9—N1	126.48 (12)
C10—N3—C9	103.47 (11)	C8—C9—N1	121.97 (11)
C10—N4—C8	106.08 (11)	N3—C10—N4	113.43 (11)
C10—N4—C13	127.01 (11)	N3—C10—H10	123.3
C8—N4—C13	126.90 (11)	N4—C10—H10	123.3
C3 ⁱ —C1—C2	119.74 (12)	N1—C11—H11A	109.5
C3 ⁱ —C1—C4	119.61 (11)	N1—C11—H11B	109.5
C2—C1—C4	120.31 (11)	H11A—C11—H11B	109.5
C3—C2—C1	119.94 (11)	N1—C11—H11C	109.5
C3—C2—C5	117.97 (11)	H11A—C11—H11C	109.5
C1—C2—C5	122.07 (11)	H11B—C11—H11C	109.5
C1 ⁱ —C3—C2	120.31 (12)	N2—C12—H12A	109.5
C1 ⁱ —C3—H3	119.8	N2—C12—H12B	109.5

C2—C3—H3	119.8	H12A—C12—H12B	109.5
O1—C4—O2	125.76 (12)	N2—C12—H12C	109.5
O1—C4—C1	121.79 (12)	H12A—C12—H12C	109.5
O2—C4—C1	112.42 (11)	H12B—C12—H12C	109.5
O3—C5—O4	125.01 (12)	N4—C13—H13A	109.5
O3—C5—C2	121.87 (11)	N4—C13—H13B	109.5
O4—C5—C2	113.10 (11)	H13A—C13—H13B	109.5
O5—C6—N1	120.74 (12)	N4—C13—H13C	109.5
O5—C6—N2	121.29 (11)	H13A—C13—H13C	109.5
N1—C6—N2	117.97 (11)	H13B—C13—H13C	109.5
C3 ⁱ —C1—C2—C3	0.6 (2)	C6—N2—C7—C8	3.16 (18)
C4—C1—C2—C3	-172.74 (11)	C12—N2—C7—C8	-175.68 (10)
C3 ⁱ —C1—C2—C5	179.23 (11)	C10—N4—C8—C9	0.39 (14)
C4—C1—C2—C5	5.92 (18)	C13—N4—C8—C9	179.52 (12)
C1—C2—C3—C1 ⁱ	-0.6 (2)	C10—N4—C8—C7	179.29 (13)
C5—C2—C3—C1 ⁱ	-179.28 (11)	C13—N4—C8—C7	-1.6 (2)
C3 ⁱ —C1—C4—O1	-131.94 (13)	O6—C7—C8—C9	175.61 (13)
C2—C1—C4—O1	41.37 (18)	N2—C7—C8—C9	-3.34 (18)
C3 ⁱ —C1—C4—O2	46.39 (15)	O6—C7—C8—N4	-3.1 (2)
C2—C1—C4—O2	-140.30 (12)	N2—C7—C8—N4	177.92 (12)
C3—C2—C5—O3	48.27 (18)	C10—N3—C9—C8	-0.07 (14)
C1—C2—C5—O3	-130.41 (14)	C10—N3—C9—N1	179.02 (12)
C3—C2—C5—O4	-130.22 (12)	N4—C8—C9—N3	-0.20 (14)
C1—C2—C5—O4	51.09 (16)	C7—C8—C9—N3	-179.22 (11)
C9—N1—C6—O5	179.55 (11)	N4—C8—C9—N1	-179.34 (11)
C11—N1—C6—O5	-1.85 (19)	C7—C8—C9—N1	1.6 (2)
C9—N1—C6—N2	-1.00 (18)	C6—N1—C9—N3	-178.25 (12)
C11—N1—C6—N2	177.60 (12)	C11—N1—C9—N3	3.2 (2)
C7—N2—C6—O5	178.31 (12)	C6—N1—C9—C8	0.75 (19)
C12—N2—C6—O5	-2.83 (18)	C11—N1—C9—C8	-177.83 (13)
C7—N2—C6—N1	-1.14 (19)	C9—N3—C10—N4	0.33 (15)
C12—N2—C6—N1	177.72 (11)	C8—N4—C10—N3	-0.47 (15)
C6—N2—C7—O6	-175.85 (12)	C13—N4—C10—N3	-179.60 (12)
C12—N2—C7—O6	5.31 (19)		

Symmetry code: (i) $-x+1, -y+2, -z+2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H1O \cdots N3 ⁱⁱ	0.86 (1)	1.83 (1)	2.6774 (17)	171 (2)
O4—H2O \cdots O5	0.84 (2)	1.84 (2)	2.6571 (15)	162 (2)
C12—H12B \cdots O6 ⁱⁱⁱ	0.98	2.30	3.239 (2)	159

Symmetry codes: (ii) $-x, -y+2, -z+1$; (iii) $-x+2, -y+1, -z+1$.