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3,4,5-Trihydroxy-*N'*-(1*H*-indol-3-yl-methylidene)benzohydrazide pentahydrate

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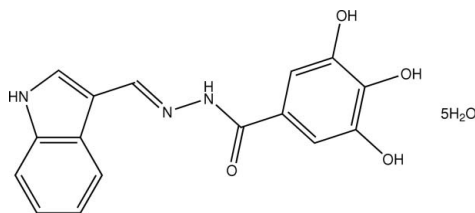
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å;
 R factor = 0.041; wR factor = 0.115; data-to-parameter ratio = 13.1.

The two aromatic parts of the title compound, $\text{C}_{16}\text{H}_{13}\text{N}_3\text{O}_4 \cdot 5\text{H}_2\text{O}$, are connected through a conjugated $-\text{CH}=\text{N}-\text{NH}-\text{C}(\text{O})-$ fragment, giving an almost planar molecule. The organic molecules and uncoordinated water molecules are linked by $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds into a three-dimensional network.

Related literature

For the structure of anhydrous *N'*-(1*H*-indol-3-ylmethylidene)-3,4,5-trihydroxybenzohydrazide, see: Khaledi *et al.* (2008).



Experimental

Crystal data

 $\text{C}_{16}\text{H}_{13}\text{N}_3\text{O}_4 \cdot 5\text{H}_2\text{O}$
 $M_r = 401.37$
 Triclinic, $P\bar{1}$
 $a = 7.4379$ (2) Å
 $b = 9.1178$ (2) Å
 $c = 14.1966$ (3) Å
 $\alpha = 103.814$ (1)°
 $\beta = 103.716$ (1)°

 $\gamma = 90.613$ (2)°
 $V = 905.95$ (4) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.12$ mm⁻¹
 $T = 100$ (2) K
 $0.30 \times 0.25 \times 0.04$ mm

Data collection

 Bruker SMART APEX
 diffractometer
 Absorption correction: none
 7524 measured reflections

 4096 independent reflections
 3285 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.115$
 $S = 1.05$
 4096 reflections
 313 parameters
 15 restraints

 H atoms treated by a mixture of
 independent and constrained
 refinement
 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1}-\text{H1} \cdots \text{O4}^{\text{i}}$	0.85 (1)	1.90 (1)	2.740 (2)	174 (2)
$\text{O2}-\text{H2} \cdots \text{O2}^{\text{w}}$	0.84 (1)	1.94 (1)	2.697 (2)	149 (2)
$\text{O3}-\text{H3} \cdots \text{O3}^{\text{w}}$	0.85 (1)	1.90 (1)	2.724 (2)	164 (2)
$\text{N1}-\text{H1}^{\text{n}} \cdots \text{O1}^{\text{w}^{\text{ii}}}$	0.88 (1)	2.11 (1)	2.978 (2)	169 (2)
$\text{N3}-\text{H3}^{\text{n}} \cdots \text{O4}^{\text{w}^{\text{iii}}}$	0.88 (1)	2.15 (1)	3.024 (2)	171 (2)
$\text{O1}^{\text{w}}-\text{H11} \cdots \text{O1}$	0.85 (1)	1.93 (1)	2.771 (2)	170 (3)
$\text{O1}^{\text{w}}-\text{H12} \cdots \text{O3}^{\text{w}^{\text{iv}}}$	0.86 (1)	1.94 (1)	2.760 (2)	161 (3)
$\text{O2}^{\text{w}}-\text{H21} \cdots \text{O3}^{\text{v}}$	0.84 (1)	2.29 (1)	3.123 (2)	172 (2)
$\text{O2}^{\text{w}}-\text{H22} \cdots \text{O5}^{\text{w}}$	0.85 (1)	1.94 (2)	2.753 (2)	161 (3)
$\text{O3}^{\text{w}}-\text{H31} \cdots \text{O5}^{\text{w}^{\text{vi}}}$	0.85 (1)	1.95 (1)	2.796 (2)	172 (3)
$\text{O3}^{\text{w}}-\text{H32} \cdots \text{O3}^{\text{vii}}$	0.85 (1)	2.06 (1)	2.890 (2)	165 (2)
$\text{O4}^{\text{w}}-\text{H41} \cdots \text{O1}^{\text{w}}$	0.85 (1)	1.97 (1)	2.803 (2)	169 (2)
$\text{O4}^{\text{w}}-\text{H42} \cdots \text{O4}^{\text{viii}}$	0.84 (1)	2.37 (2)	2.921 (2)	124 (2)
$\text{O4}^{\text{w}}-\text{H42} \cdots \text{N2}^{\text{viii}}$	0.84 (1)	2.39 (1)	3.211 (2)	166 (2)
$\text{O5}^{\text{w}}-\text{H51} \cdots \text{O4}^{\text{w}^{\text{ix}}}$	0.85 (1)	1.97 (1)	2.798 (2)	167 (2)
$\text{O5}^{\text{w}}-\text{H52} \cdots \text{O2}^{\text{w}^{\text{x}}}$	0.85 (1)	1.97 (1)	2.810 (2)	168 (3)

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $x, y + 1, z$; (iii) $-x, -y + 2, -z + 1$;
 (iv) $x, y - 1, z$; (v) $-x + 1, -y + 1, -z + 2$; (vi) $x - 1, y + 1, z$; (vii)
 $-x + 1, -y + 2, -z + 2$; (viii) $-x, -y + 1, -z + 1$; (ix) $x + 1, y, z$; (x)
 $-x + 2, -y + 1, -z + 2$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2*;
 data reduction: *SAINT* (Bruker, 2007); program(s) used to solve
 structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine
 structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-*
SEED (Barbour, 2001); software used to prepare material for
 publication: *pubCIF* (Westrip, 2008).

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 (Science Fund grants 12-02-03-2031 and 12-02-03-2051).

Supplementary data and figures for this paper are available from the
 IUCr electronic archives (Reference: TK2335).

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supplementary materials

Acta Cryst. (2008). E64, o2481 [doi:10.1107/S1600536808039342]

3,4,5-Trihydroxy-*N'*-(1*H*-indol-3-ylmethylidene)benzohydrazide pentahydrate

H. Khaledi, H. Mohd Ali and S. W. Ng

Comment

(type here to add)

Experimental

Indole-3-carbaldehyde (1.0 g, 7 mmol) and 3,4,5-trihydroxybenzoylhydrazine (1.29 g, 7 mmol) were heated in ethanol (60 ml) for 6 h. About 1 ml of acetic acid also added. The solution was set aside for the growth of crystals.

Refinement

Hydrogen atoms were placed at calculated positions (C–H 0.95 Å), and were treated as riding on their parent carbon atoms, with $U(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$. The nitrogen- and oxygen-bound H atoms were located in a difference Fourier map, and were refined with distance restraints of N–H 0.88 ± 0.01 and O–H 0.84 ± 0.01 Å.

Figures

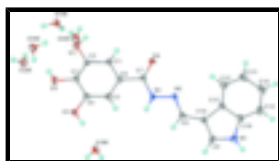


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $\text{C}_{16}\text{H}_{13}\text{N}_3\text{O}_4 \cdot 5\text{H}_2\text{O}$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

3,4,5-Trihydroxy-*N'*-(1*H*-indol-3-ylmethylidene)benzohydrazide pentahydrate

Crystal data

$\text{C}_{16}\text{H}_{13}\text{N}_3\text{O}_4 \cdot 5\text{H}_2\text{O}$

$M_r = 401.37$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.4379$ (2) Å

$b = 9.1178$ (2) Å

$c = 14.1966$ (3) Å

$\alpha = 103.814$ (1)°

$\beta = 103.716$ (1)°

$\gamma = 90.613$ (2)°

$V = 905.95$ (4) Å³

$Z = 2$

$F_{000} = 424$

$D_x = 1.471$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 2461 reflections

$\theta = 2.3$ – 28.0 °

$\mu = 0.12$ mm⁻¹

$T = 100$ (2) K

Plate, pale-yellow

$0.30 \times 0.25 \times 0.04$ mm

supplementary materials

Data collection

Bruker SMART APEX diffractometer	3285 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.023$
Monochromator: graphite	$\theta_{\text{max}} = 27.5^\circ$
$T = 100(2)$ K	$\theta_{\text{min}} = 2.3^\circ$
ω scans	$h = -9 \rightarrow 9$
Absorption correction: None	$k = -11 \rightarrow 11$
7524 measured reflections	$l = -18 \rightarrow 18$
4096 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.115$	$w = 1/[\sigma^2(F_o^2) + (0.0613P)^2 + 0.1789P]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
4096 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
313 parameters	$\Delta\rho_{\text{max}} = 0.35 \text{ e } \text{\AA}^{-3}$
15 restraints	$\Delta\rho_{\text{min}} = -0.25 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.59471 (16)	0.40029 (12)	0.72041 (8)	0.0184 (2)
O2	0.51512 (17)	0.56940 (13)	0.89648 (8)	0.0217 (3)
O3	0.35915 (16)	0.82334 (12)	0.90287 (8)	0.0180 (2)
O4	0.31349 (16)	0.68239 (12)	0.45992 (8)	0.0197 (3)
O1w	0.35758 (17)	0.17412 (13)	0.73409 (8)	0.0200 (3)
O2w	0.75855 (17)	0.38643 (13)	0.97416 (9)	0.0221 (3)
O3w	0.34432 (17)	1.12598 (13)	0.91713 (8)	0.0213 (3)
O4w	0.01511 (18)	0.30256 (13)	0.69710 (9)	0.0231 (3)
O5w	1.07168 (17)	0.33392 (13)	0.90316 (9)	0.0232 (3)
N1	0.29295 (19)	0.91884 (14)	0.55157 (9)	0.0162 (3)
N2	0.24067 (18)	0.97264 (14)	0.46592 (9)	0.0165 (3)
N3	0.08352 (19)	1.39294 (15)	0.34212 (10)	0.0183 (3)
C1	0.4530 (2)	0.58425 (16)	0.63391 (11)	0.0147 (3)
H1	0.4753	0.5274	0.5729	0.018*
C2	0.5040 (2)	0.53102 (16)	0.71918 (11)	0.0146 (3)
C3	0.4698 (2)	0.61229 (17)	0.80875 (11)	0.0151 (3)

C4	0.3855 (2)	0.74827 (16)	0.81103 (11)	0.0147 (3)
C5	0.3332 (2)	0.80233 (16)	0.72644 (11)	0.0148 (3)
H5	0.2733	0.8942	0.7290	0.018*
C6	0.3693 (2)	0.72070 (16)	0.63685 (10)	0.0140 (3)
C7	0.3236 (2)	0.77172 (16)	0.54225 (11)	0.0144 (3)
C8	0.2179 (2)	1.11562 (17)	0.48486 (11)	0.0168 (3)
H8	0.2354	1.1700	0.5528	0.020*
C9	0.1240 (2)	1.34618 (17)	0.42795 (11)	0.0175 (3)
H9	0.1226	1.4073	0.4921	0.021*
C10	0.1675 (2)	1.19684 (17)	0.40875 (11)	0.0158 (3)
C11	0.1553 (2)	1.14989 (17)	0.30295 (11)	0.0154 (3)
C12	0.1934 (2)	1.01736 (17)	0.23897 (11)	0.0194 (3)
H12A	0.2299	0.9310	0.2632	0.023*
C13	0.1763 (2)	1.01605 (19)	0.13982 (12)	0.0229 (4)
H13	0.2023	0.9275	0.0956	0.028*
C14	0.1213 (2)	1.1422 (2)	0.10270 (12)	0.0241 (4)
H14	0.1093	1.1367	0.0338	0.029*
C15	0.0845 (2)	1.27369 (19)	0.16400 (12)	0.0212 (3)
H15	0.0478	1.3594	0.1390	0.025*
C16	0.1033 (2)	1.27600 (17)	0.26438 (11)	0.0169 (3)
H1o	0.616 (3)	0.370 (2)	0.6634 (10)	0.046 (7)*
H2o	0.584 (3)	0.4971 (18)	0.8974 (16)	0.039 (6)*
H3o	0.333 (3)	0.9129 (14)	0.8998 (16)	0.038 (6)*
H1n	0.314 (3)	0.9850 (17)	0.6102 (9)	0.025 (5)*
H3n	0.052 (3)	1.4844 (15)	0.3374 (17)	0.046 (6)*
H11	0.438 (3)	0.235 (2)	0.7274 (19)	0.060 (8)*
H12	0.380 (4)	0.163 (3)	0.7939 (10)	0.059 (8)*
H21	0.719 (3)	0.336 (2)	1.0090 (16)	0.055 (7)*
H22	0.848 (3)	0.349 (4)	0.952 (2)	0.112 (13)*
H31	0.255 (3)	1.184 (3)	0.915 (2)	0.067 (9)*
H32	0.425 (2)	1.158 (2)	0.9721 (10)	0.037 (6)*
H41	0.117 (2)	0.260 (3)	0.7001 (18)	0.055 (8)*
H42	-0.066 (3)	0.242 (2)	0.6537 (15)	0.063 (8)*
H51	1.039 (3)	0.331 (3)	0.8414 (8)	0.055 (8)*
H52	1.124 (4)	0.4219 (19)	0.932 (2)	0.101 (12)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0248 (6)	0.0168 (5)	0.0172 (6)	0.0079 (5)	0.0091 (5)	0.0068 (4)
O2	0.0281 (7)	0.0247 (6)	0.0164 (6)	0.0119 (5)	0.0081 (5)	0.0099 (5)
O3	0.0241 (6)	0.0179 (6)	0.0123 (5)	0.0060 (5)	0.0060 (4)	0.0026 (4)
O4	0.0282 (6)	0.0169 (5)	0.0131 (5)	0.0037 (5)	0.0051 (5)	0.0021 (4)
O1w	0.0247 (6)	0.0202 (6)	0.0156 (6)	0.0008 (5)	0.0053 (5)	0.0051 (4)
O2w	0.0242 (7)	0.0240 (6)	0.0223 (6)	0.0054 (5)	0.0101 (5)	0.0094 (5)
O3w	0.0235 (6)	0.0233 (6)	0.0155 (6)	0.0042 (5)	0.0031 (5)	0.0032 (4)
O4w	0.0217 (6)	0.0249 (6)	0.0217 (6)	0.0056 (5)	0.0035 (5)	0.0057 (5)
O5w	0.0249 (7)	0.0234 (6)	0.0203 (6)	0.0028 (5)	0.0070 (5)	0.0023 (5)

supplementary materials

N1	0.0228 (7)	0.0147 (6)	0.0106 (6)	0.0027 (5)	0.0029 (5)	0.0037 (5)
N2	0.0201 (7)	0.0179 (6)	0.0127 (6)	0.0027 (5)	0.0041 (5)	0.0063 (5)
N3	0.0196 (7)	0.0150 (6)	0.0212 (7)	0.0033 (5)	0.0041 (5)	0.0069 (5)
C1	0.0158 (7)	0.0148 (7)	0.0129 (7)	0.0006 (6)	0.0040 (6)	0.0019 (5)
C2	0.0143 (7)	0.0127 (7)	0.0176 (7)	0.0014 (6)	0.0046 (6)	0.0043 (6)
C3	0.0144 (7)	0.0181 (7)	0.0138 (7)	0.0005 (6)	0.0034 (6)	0.0060 (6)
C4	0.0140 (7)	0.0166 (7)	0.0126 (7)	-0.0003 (6)	0.0046 (6)	0.0009 (5)
C5	0.0158 (7)	0.0131 (7)	0.0152 (7)	0.0019 (6)	0.0040 (6)	0.0028 (5)
C6	0.0136 (7)	0.0148 (7)	0.0125 (7)	-0.0014 (6)	0.0019 (5)	0.0030 (5)
C7	0.0142 (7)	0.0159 (7)	0.0132 (7)	0.0002 (6)	0.0033 (6)	0.0037 (5)
C8	0.0191 (8)	0.0175 (7)	0.0136 (7)	0.0022 (6)	0.0041 (6)	0.0032 (6)
C9	0.0177 (8)	0.0176 (7)	0.0169 (7)	0.0013 (6)	0.0038 (6)	0.0043 (6)
C10	0.0164 (7)	0.0154 (7)	0.0155 (7)	0.0012 (6)	0.0035 (6)	0.0042 (6)
C11	0.0153 (7)	0.0175 (7)	0.0134 (7)	-0.0011 (6)	0.0027 (6)	0.0046 (6)
C12	0.0202 (8)	0.0174 (7)	0.0194 (8)	-0.0010 (6)	0.0042 (6)	0.0031 (6)
C13	0.0249 (9)	0.0226 (8)	0.0191 (8)	-0.0037 (7)	0.0070 (7)	-0.0005 (6)
C14	0.0242 (9)	0.0322 (9)	0.0145 (7)	-0.0084 (7)	0.0028 (6)	0.0054 (6)
C15	0.0185 (8)	0.0264 (8)	0.0191 (8)	-0.0041 (7)	0.0002 (6)	0.0109 (6)
C16	0.0137 (7)	0.0184 (7)	0.0178 (7)	-0.0017 (6)	0.0016 (6)	0.0054 (6)

Geometric parameters (Å, °)

O1—C2	1.3777 (18)	C1—C2	1.383 (2)
O1—H1o	0.845 (9)	C1—C6	1.393 (2)
O2—C3	1.3615 (18)	C1—H1	0.9500
O2—H2o	0.839 (10)	C2—C3	1.392 (2)
O3—C4	1.3801 (17)	C3—C4	1.392 (2)
O3—H3o	0.851 (10)	C4—C5	1.379 (2)
O4—C7	1.2400 (17)	C5—C6	1.399 (2)
O1w—H11	0.850 (10)	C5—H5	0.9500
O1w—H12	0.855 (10)	C6—C7	1.490 (2)
O2w—H21	0.843 (10)	C8—C10	1.433 (2)
O2w—H22	0.846 (10)	C8—H8	0.9500
O3w—H31	0.854 (10)	C9—C10	1.381 (2)
O3w—H32	0.848 (10)	C9—H9	0.9500
O4w—H41	0.848 (10)	C10—C11	1.441 (2)
O4w—H42	0.839 (10)	C11—C12	1.404 (2)
O5w—H51	0.847 (10)	C11—C16	1.408 (2)
O5w—H52	0.850 (10)	C12—C13	1.380 (2)
N1—C7	1.3434 (19)	C12—H12A	0.9500
N1—N2	1.3910 (17)	C13—C14	1.402 (2)
N1—H1n	0.882 (9)	C13—H13	0.9500
N2—C8	1.288 (2)	C14—C15	1.375 (2)
N3—C9	1.354 (2)	C14—H14	0.9500
N3—C16	1.376 (2)	C15—C16	1.394 (2)
N3—H3n	0.882 (10)	C15—H15	0.9500
C2—O1—H1o	106.5 (16)	C5—C6—C7	123.54 (13)
C3—O2—H2o	115.8 (15)	O4—C7—N1	122.36 (13)
C4—O3—H3o	107.5 (14)	O4—C7—C6	121.46 (13)

H11—O1w—H12	112 (2)	N1—C7—C6	116.18 (13)
H21—O2w—H22	114 (3)	N2—C8—C10	123.34 (14)
H31—O3w—H32	109 (2)	N2—C8—H8	118.3
H41—O4w—H42	107 (2)	C10—C8—H8	118.3
H51—O5w—H52	106 (3)	N3—C9—C10	110.20 (14)
C7—N1—N2	119.24 (12)	N3—C9—H9	124.9
C7—N1—H1n	122.5 (12)	C10—C9—H9	124.9
N2—N1—H1n	118.0 (12)	C9—C10—C8	123.34 (14)
C8—N2—N1	113.26 (12)	C9—C10—C11	106.25 (13)
C9—N3—C16	109.03 (13)	C8—C10—C11	130.40 (14)
C9—N3—H3n	125.2 (15)	C12—C11—C16	119.25 (14)
C16—N3—H3n	125.7 (15)	C12—C11—C10	134.26 (14)
C2—C1—C6	120.49 (13)	C16—C11—C10	106.36 (13)
C2—C1—H1	119.8	C13—C12—C11	118.14 (15)
C6—C1—H1	119.8	C13—C12—H12A	120.9
O1—C2—C1	122.20 (13)	C11—C12—H12A	120.9
O1—C2—C3	117.48 (13)	C12—C13—C14	121.62 (15)
C1—C2—C3	120.29 (14)	C12—C13—H13	119.2
O2—C3—C4	116.52 (13)	C14—C13—H13	119.2
O2—C3—C2	124.59 (14)	C15—C14—C13	121.35 (15)
C4—C3—C2	118.89 (13)	C15—C14—H14	119.3
O3—C4—C5	123.17 (14)	C13—C14—H14	119.3
O3—C4—C3	115.42 (13)	C14—C15—C16	117.23 (15)
C5—C4—C3	121.41 (13)	C14—C15—H15	121.4
C4—C5—C6	119.40 (14)	C16—C15—H15	121.4
C4—C5—H5	120.3	N3—C16—C15	129.42 (14)
C6—C5—H5	120.3	N3—C16—C11	108.14 (13)
C1—C6—C5	119.50 (13)	C15—C16—C11	122.40 (15)
C1—C6—C7	116.96 (13)		
C7—N1—N2—C8	-179.15 (14)	N1—N2—C8—C10	178.78 (14)
C6—C1—C2—O1	-176.94 (13)	C16—N3—C9—C10	-1.24 (18)
C6—C1—C2—C3	0.9 (2)	N3—C9—C10—C8	179.52 (14)
O1—C2—C3—O2	-1.9 (2)	N3—C9—C10—C11	0.96 (18)
C1—C2—C3—O2	-179.82 (14)	N2—C8—C10—C9	172.92 (15)
O1—C2—C3—C4	177.27 (13)	N2—C8—C10—C11	-8.9 (3)
C1—C2—C3—C4	-0.7 (2)	C9—C10—C11—C12	175.33 (17)
O2—C3—C4—O3	0.3 (2)	C8—C10—C11—C12	-3.1 (3)
C2—C3—C4—O3	-178.94 (13)	C9—C10—C11—C16	-0.34 (17)
O2—C3—C4—C5	-179.82 (13)	C8—C10—C11—C16	-178.76 (16)
C2—C3—C4—C5	1.0 (2)	C16—C11—C12—C13	-0.6 (2)
O3—C4—C5—C6	178.43 (13)	C10—C11—C12—C13	-175.81 (16)
C3—C4—C5—C6	-1.5 (2)	C11—C12—C13—C14	-0.4 (2)
C2—C1—C6—C5	-1.4 (2)	C12—C13—C14—C15	0.9 (3)
C2—C1—C6—C7	178.38 (13)	C13—C14—C15—C16	-0.3 (2)
C4—C5—C6—C1	1.7 (2)	C9—N3—C16—C15	-176.86 (16)
C4—C5—C6—C7	-178.10 (13)	C9—N3—C16—C11	0.99 (17)
N2—N1—C7—O4	2.7 (2)	C14—C15—C16—N3	176.83 (16)
N2—N1—C7—C6	-176.87 (12)	C14—C15—C16—C11	-0.8 (2)
C1—C6—C7—O4	18.6 (2)	C12—C11—C16—N3	-176.83 (14)

supplementary materials

C5—C6—C7—O4	-161.64 (14)	C10—C11—C16—N3	-0.38 (17)
C1—C6—C7—N1	-161.86 (13)	C12—C11—C16—C15	1.2 (2)
C5—C6—C7—N1	17.9 (2)	C10—C11—C16—C15	177.65 (14)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1o \cdots O4 ⁱ	0.85 (1)	1.90 (1)	2.740 (2)	174 (2)
O2—H2o \cdots O2w	0.84 (1)	1.94 (1)	2.697 (2)	149 (2)
O3—H3o \cdots O3w	0.85 (1)	1.90 (1)	2.724 (2)	164 (2)
N1—H1n \cdots O1w ⁱⁱ	0.88 (1)	2.11 (1)	2.978 (2)	169 (2)
N3—H3n \cdots O4w ⁱⁱⁱ	0.88 (1)	2.15 (1)	3.024 (2)	171 (2)
O1w—H11 \cdots O1	0.85 (1)	1.93 (1)	2.771 (2)	170 (3)
O1w—H12 \cdots O3w ^{iv}	0.86 (1)	1.94 (1)	2.760 (2)	161 (3)
O2w—H21 \cdots O3 ^v	0.84 (1)	2.29 (1)	3.123 (2)	172 (2)
O2w—H22 \cdots O5w	0.85 (1)	1.94 (2)	2.753 (2)	161 (3)
O3w—H31 \cdots O5w ^{vi}	0.85 (1)	1.95 (1)	2.796 (2)	172 (3)
O3w—H32 \cdots O3 ^{vii}	0.85 (1)	2.06 (1)	2.890 (2)	165 (2)
O4w—H41 \cdots O1w	0.85 (1)	1.97 (1)	2.803 (2)	169 (2)
O4w—H42 \cdots O4 ^{viii}	0.84 (1)	2.37 (2)	2.921 (2)	124 (2)
O4w—H42 \cdots N2 ^{viii}	0.84 (1)	2.39 (1)	3.211 (2)	166 (2)
O5w—H51 \cdots O4w ^{ix}	0.85 (1)	1.97 (1)	2.798 (2)	167 (2)
O5w—H52 \cdots O2w ^x	0.85 (1)	1.97 (1)	2.810 (2)	168 (3)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x, y+1, z$; (iii) $-x, -y+2, -z+1$; (iv) $x, y-1, z$; (v) $-x+1, -y+1, -z+2$; (vi) $x-1, y+1, z$; (vii) $-x+1, -y+2, -z+2$; (viii) $-x, -y+1, -z+1$; (ix) $x+1, y, z$; (x) $-x+2, -y+1, -z+2$.

Fig. 1

