organic compounds

Hydrogen-bonding patterns in 2-amino-4,6-dimethoxypyrimidine–phthalic acid (1/1)

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In the title cocrystal, $C_6H_9N_3O_2 \cdot C_8H_6O_4$, both carboxylic acid groups of phthalic acid form an $R_2^2(8)$ ring motif (through N—H···O and O—H···N hydrogen bonds) on either side of the 2-amino-4,6-dimethoxypyrimidine molecule, generating a helical chain along the b axis. This chain is interpenetrated by a centrosymmetrically related chain to which it is linked by π–π stacking [perpendicular separation 3.332 Å, centroid–centroid distance 3.6424 (7) Å].

Related literature

For related literature, see: Baker & Santi (1965); Bernstein *et al.* (1995); Chinnakali *et al.* (1999); Etter (1990); Hunt *et al.* (1980); Hunter (1994); Low *et al.* (2002); Lynch & Jones (2004); Muthiah *et al.* (2006); Schwalbe & Williams (1982); Thanigaimani *et al.* (2006); Van Schalkwyk (1954).
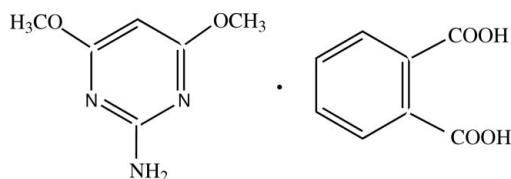
Experimental

Crystal data

$C_6H_9N_3O_2 \cdot C_8H_6O_4$
$M_r = 321.29$
Monoclinic, $P2_1/c$
$a = 11.2175$ (3) Å
$b = 7.3323$ (2) Å
$c = 17.7651$ (4) Å
$\beta = 90.776$ (2)°

$V = 1461.05$ (6) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.12$ mm^{-1}
$T = 120$ K
$0.50 \times 0.40 \times 0.25$ mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
Absorption correction: none
17905 measured reflections

3328 independent reflections
2748 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.114$
$S = 1.08$
3328 reflections

211 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O5^i	0.86	2.06	2.8704 (15)	156
N2—H2B···O4	0.86	2.01	2.8609 (15)	173
O3—H3···N1	0.82	1.87	2.6881 (14)	171
O6—H6···N3^{ii}	0.82	1.86	2.6738 (14)	172
C8—H8B···O5^{iii}	0.96	2.56	3.2199 (17)	126

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

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; 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: LW2035).

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supplementary materials

Hydrogen-bonding patterns in 2-amino-4,6-dimethoxypyrimidine-phthalic acid (1/1)

K. Thanigaimani, P. T. Muthiah and D. E. Lynch

Comment

Aminopyrimidine-carboxylate interactions are of fundamental important since they are involved in protein-nucleic acids recognition and protein-drug binding (Hunt *et al.*, 1980; Baker & Santi, 1965). The adducts of carboxylic acid with 2-amino heterocylic ring system have a graph-set motif $[R_2^2(8)]$ (Lynch & Jones, 2004). This motif is very robust in aminopyrimidine-carboxylic acid/ carboxylate systems. The crystal structures of aminopyrimidine derivatives (Schwalbe & Williams, 1982), aminopyrimidine carboxylates (Muthiah *et al.*, 2006) and cocrystals (Chinnakali *et al.*, 1999) have been reported. The crystal structure of 2-amino-4,6-dimethoxy pyrimidine (Low *et al.*, 2002) and 2-amino-4,6-dimethoxypyrimidine-4-aminobenzoic acid (1/1) (Thanigaimani *et al.*, 2006) have also been reported. The crystal structure of phthalic acid (Van Schalkwyk, 1954) is known. The present study investigates the hydrogen bonding patterns in 2-amino-4,6-dimethoxy pyrimidine: phthalic acid (1/1) cocrystal(I).

The asymmetric unit (Fig 1) contains one 2-amino-4,6-dimethoxypyrimidine molecule and one phthalic acid molecule, which are linked by N—H···O and O—H···N intermolecular hydrogen bonds involving the nitrogen (N1) atom and 2-amino (NH$_2$) group of the pyrimidine ring and carboxyl oxygen atoms (O3 and O4) to form a eight membered ring motif of graph-set notation of $[R_2^2(8)]$ (Etter, 1990; Bernstein *et al.*, 1995). The another nitrogen atom (N3) and 2-amino (NH$_2$) group of the pyrimidine ring also interact with carboxyl oxygen atoms (O5 and O6) to form a similar eight membered ring motif, generating a helical chain (made up of alternating pyrimidine and phthalic acid molecules) along the *b* axis (Fig 2). This chain is interpenetrated by a centrosymmetrically related chain (Fig 3) to which it is linked by π-π stacking of pyrimidines with a perpendicular separation of 3.332 Å, a centroid-centroid distance of 3.6424 (7) Å, ring offset of 1.471Å and a slip angle (the angle between the centroid vector and the normal to the plane) of 23.81° These are typical aromatic stacking values (Hunter, 1994).

Experimental

A hot methanolic solution (20 ml) of 2-amino-4,6-dimethoxy pyrimidine (38 mg, Aldrich) and phthalic acid (41 mg, Loba chemie) was warmed for half an hour over a water bath. The mixture was cooled slowly and kept at room temperature. After a few days colourless plate-like crystals were obtained.

Refinement

All H atoms were positioned geometrically and were refined using a riding model. The C—H, O—H and N—H bond lengths are 0.93–0.96, 0.82 and 0.86 Å, respectively [U_{iso} (H)=1.2 U_{eq}(parent atom)].
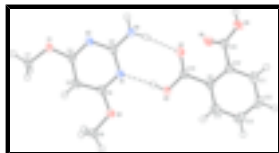
supplementary materials

Figures

Fig. 1. An *ORTEP* view of the asymmetric unit of (I) showing 50% probability displacement ellipsoids.
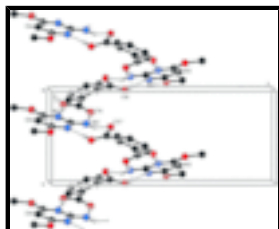

Fig. 2. Helical chain containing alternative pyrimidine and phthalic acid molecules (I) [symmetry code: (i) $-x + 2, y + 1/2, -z + 1/2$ (ii) $-x + 2, y - 1/2, -z + 1/2$.
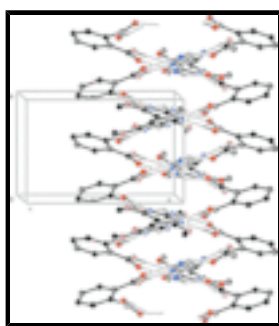
Fig. 3. Two interpenetrated centrosymmetrically related helical chain along the *b* axis (I).

2-amino-4,6-dimethoxypyrimidine–phthalic acid (1/1)

Crystal data

$C_6H_9N_3O_2 \cdot C_8H_6O_4$	$F_{000} = 672$
$M_r = 321.29$	$D_x = 1.461$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
$a = 11.2175$ (3) Å	$\theta = 2.9–27.5°$
$b = 7.3323$ (2) Å	$\mu = 0.12$ mm^{-1}
$c = 17.7651$ (4) Å	$T = 120$ K
$\beta = 90.776$ (2)°	Plate, colourless
$V = 1461.05$ (6) Å^3	$0.50 \times 0.40 \times 0.25$ mm
$Z = 4$	

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer	2748 reflections with $I > 2\sigma(I)$
Radiation source: Bruker–Nonius FR591 rotating anode	$R_{int} = 0.029$
Monochromator: graphite	$\theta_{max} = 27.5°$
$T = 120$ K	$\theta_{min} = 3.5°$

φ and ω scans	$h = -14 \rightarrow 14$
Absorption correction: none	$k = -9 \rightarrow 9$
17905 measured reflections	$l = -23 \rightarrow 23$
3328 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.041$	$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.4007P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.114$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.08$	$\Delta\rho_{max} = 0.33$ e Å^{-3}
3328 reflections	$\Delta\rho_{min} = -0.30$ e Å^{-3}
211 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $FC^* = KFC[1+0.001XFC^2\Lambda^3/SIN(2\Theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.034 (4)
Secondary atom site location: difference Fourier map	

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of F^2 > 2sigma(F^2) is used only for calculating –*R*-factor-obs *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	1.33166 (8)	0.41207 (13)	−0.01434 (5)	0.0234 (3)
O2	0.94377 (8)	0.19097 (15)	−0.08929 (5)	0.0270 (3)
N1	0.99024 (9)	0.26729 (16)	0.02820 (6)	0.0207 (3)
N2	1.03481 (10)	0.33885 (18)	0.15133 (6)	0.0275 (4)
N3	1.18347 (9)	0.37441 (15)	0.06527 (6)	0.0206 (3)
C2	1.07043 (11)	0.32599 (18)	0.08002 (7)	0.0202 (4)
C4	1.21785 (11)	0.35975 (17)	−0.00621 (7)	0.0190 (3)
C5	1.14427 (11)	0.29757 (18)	−0.06394 (7)	0.0205 (3)
C6	1.02900 (11)	0.25317 (18)	−0.04249 (7)	0.0203 (4)
C7	1.38345 (12)	0.39831 (19)	−0.08761 (7)	0.0248 (4)
C8	0.97333 (13)	0.1688 (2)	−0.16733 (7)	0.0313 (5)
O3	0.76763 (8)	0.15122 (14)	0.05538 (5)	0.0255 (3)

O4	0.79349 (9)	0.23059 (17)	0.17615 (5)	0.0361 (4)
O5	0.76904 (9)	−0.13437 (16)	0.24381 (5)	0.0335 (3)
O6	0.65842 (8)	−0.00257 (14)	0.33283 (5)	0.0266 (3)
C9	0.61256 (11)	0.08770 (17)	0.13920 (7)	0.0189 (3)
C10	0.58606 (11)	0.00391 (17)	0.20794 (7)	0.0191 (3)
C11	0.47029 (11)	−0.05260 (19)	0.22163 (8)	0.0246 (4)
C12	0.38160 (12)	−0.0300 (2)	0.16694 (9)	0.0293 (4)
C13	0.40797 (12)	0.0508 (2)	0.09912 (8)	0.0283 (4)
C14	0.52267 (11)	0.11137 (19)	0.08528 (7)	0.0230 (4)
C15	0.73442 (11)	0.16274 (18)	0.12583 (7)	0.0202 (4)
C16	0.68190 (11)	−0.04810 (18)	0.26337 (7)	0.0206 (4)
H2A	1.08350	0.37690	0.18570	0.0330*
H2B	0.96320	0.30910	0.16290	0.0330*
H5	1.16990	0.28630	−0.11330	0.0250*
H7A	1.46440	0.44060	−0.08530	0.0370*
H7B	1.33860	0.47170	−0.12260	0.0370*
H7C	1.38200	0.27340	−0.10380	0.0370*
H8A	0.90510	0.12350	−0.19460	0.0470*
H8B	1.03790	0.08370	−0.17160	0.0470*
H8C	0.99670	0.28430	−0.18780	0.0470*
H3	0.83520	0.19250	0.05140	0.0380*
H6	0.71170	−0.04000	0.36090	0.0400*
H11	0.45190	−0.10580	0.26760	0.0300*
H12	0.30440	−0.06950	0.17610	0.0350*
H13	0.34860	0.06480	0.06250	0.0340*
H14	0.53960	0.16800	0.03980	0.0280*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0198 (4)	0.0317 (5)	0.0188 (4)	−0.0030 (4)	0.0051 (3)	−0.0006 (4)
O2	0.0199 (5)	0.0452 (6)	0.0160 (4)	−0.0030 (4)	0.0022 (3)	−0.0057 (4)
N1	0.0188 (5)	0.0277 (6)	0.0156 (5)	−0.0002 (4)	0.0027 (4)	0.0000 (4)
N2	0.0186 (5)	0.0486 (8)	0.0155 (5)	−0.0066 (5)	0.0037 (4)	−0.0022 (5)
N3	0.0189 (5)	0.0263 (6)	0.0165 (5)	−0.0003 (4)	0.0022 (4)	0.0002 (4)
C2	0.0193 (6)	0.0232 (7)	0.0181 (6)	0.0013 (5)	0.0015 (5)	0.0014 (5)
C4	0.0187 (6)	0.0196 (6)	0.0188 (6)	0.0025 (5)	0.0035 (5)	0.0022 (5)
C5	0.0224 (6)	0.0247 (7)	0.0145 (5)	0.0017 (5)	0.0037 (5)	0.0003 (5)
C6	0.0209 (6)	0.0228 (7)	0.0173 (6)	0.0027 (5)	0.0007 (5)	0.0002 (5)
C7	0.0242 (7)	0.0273 (7)	0.0231 (6)	−0.0019 (5)	0.0099 (5)	−0.0009 (6)
C8	0.0248 (7)	0.0522 (10)	0.0170 (6)	−0.0034 (6)	0.0021 (5)	−0.0083 (6)
O3	0.0210 (5)	0.0382 (6)	0.0174 (5)	−0.0054 (4)	0.0041 (4)	−0.0006 (4)
O4	0.0277 (5)	0.0595 (8)	0.0212 (5)	−0.0182 (5)	0.0044 (4)	−0.0079 (5)
O5	0.0236 (5)	0.0565 (7)	0.0204 (5)	0.0149 (5)	0.0017 (4)	−0.0024 (5)
O6	0.0273 (5)	0.0363 (6)	0.0162 (4)	0.0104 (4)	0.0020 (4)	−0.0001 (4)
C9	0.0197 (6)	0.0190 (6)	0.0180 (6)	0.0009 (5)	0.0011 (5)	−0.0018 (5)
C10	0.0192 (6)	0.0185 (6)	0.0198 (6)	0.0023 (5)	0.0030 (5)	−0.0011 (5)
C11	0.0225 (6)	0.0233 (7)	0.0282 (7)	0.0008 (5)	0.0071 (5)	0.0016 (6)

C12	0.0164 (6)	0.0305 (8)	0.0411 (8)	−0.0022 (5)	0.0036 (6)	−0.0023 (6)
C13	0.0218 (7)	0.0295 (8)	0.0334 (8)	0.0043 (5)	−0.0062 (5)	−0.0011 (6)
C14	0.0242 (6)	0.0225 (7)	0.0223 (6)	0.0021 (5)	−0.0017 (5)	0.0008 (5)
C15	0.0208 (6)	0.0225 (7)	0.0173 (6)	0.0011 (5)	0.0024 (5)	0.0028 (5)
C16	0.0201 (6)	0.0238 (7)	0.0180 (6)	−0.0001 (5)	0.0039 (5)	0.0016 (5)

Geometric parameters (Å, °)

O1—C4	1.3427 (15)	C5—H5	0.9300
O1—C7	1.4361 (15)	C7—H7B	0.9600
O2—C6	1.3386 (16)	C7—H7A	0.9600
O2—C8	1.4391 (15)	C7—H7C	0.9600
O3—C15	1.3134 (15)	C8—H8C	0.9600
O4—C15	1.2123 (16)	C8—H8A	0.9600
O5—C16	1.2188 (16)	C8—H8B	0.9600
O6—C16	1.3086 (15)	C9—C10	1.4025 (18)
O3—H3	0.8200	C9—C15	1.4955 (18)
O6—H6	0.8200	C9—C14	1.3922 (18)
N1—C2	1.3489 (16)	C10—C16	1.4974 (18)
N1—C6	1.3385 (16)	C10—C11	1.3878 (18)
N2—C2	1.3368 (16)	C11—C12	1.391 (2)
N3—C4	1.3364 (16)	C12—C13	1.378 (2)
N3—C2	1.3460 (16)	C13—C14	1.3863 (18)
N2—H2B	0.8600	C11—H11	0.9300
N2—H2A	0.8600	C12—H12	0.9300
C4—C5	1.3849 (18)	C13—H13	0.9300
C5—C6	1.3916 (18)	C14—H14	0.9300
O3···N1	2.6881 (14)	C13···H14^{v}	3.0100
O4···C16	2.8629 (17)	C14···H14^{v}	3.1000
O4···N2	2.8609 (15)	C14···H7C^{ii}	3.0300
O4···O5	2.9479 (16)	C15···H7B^{vi}	2.8000
O5···O4	2.9479 (16)	C15···H2B	2.8500
O5···N2^{i}	2.8704 (15)	C16···H2A^{i}	2.8300
O5···C8^{ii}	3.2199 (17)	H2A···C16^{iii}	2.8300
O5···C15	3.0443 (17)	H2A···O5^{iii}	2.0600
O6···N3^{i}	2.6738 (14)	H2A···H6^{iii}	2.5300
O1···H6^{iii}	2.8000	H2A···C8^{vii}	2.9200
O1···H13^{iv}	2.8900	H2B···O4	2.0100
O2···H3	2.8000	H2B···C8^{vii}	3.0200
O3···H14	2.5700	H2B···H3	2.5800
O3···H13^{v}	2.9200	H2B···C15	2.8500
O4···H7B^{vi}	2.8000	H3···C2	2.8500
O4···H2B	2.0100	H3···N1	1.8700
O4···H8A^{vii}	2.8100	H3···O2	2.8000
O5···H2A^{i}	2.0600	H3···C6	2.7900
O5···H8B^{ii}	2.5600	H3···H2B	2.5800

O5···H5[ii]	2.6700	H5···C7	2.5700
O6···H11	2.6900	H5···O5[ii]	2.6700
O6···H7A[viii]	2.6700	H5···H8C	2.3400
N1···O3	2.6881 (14)	H5···C8	2.5400
N2···O5[iii]	2.8704 (15)	H5···H7B	2.3400
N2···O4	2.8609 (15)	H5···H7C	2.3800
N2···C8[vii]	3.3041 (17)	H5···H8B	2.3300
N3···O6[iii]	2.6738 (14)	H6···C4[i]	2.7900
N3···C16[iii]	3.4273 (16)	H6···H2A[i]	2.5300
N1···H3	1.8700	H6···C2[i]	2.8200
N2···H8C[vi]	2.8600	H6···N3[i]	1.8600
N3···H6[iii]	1.8600	H6···O1[i]	2.8000
N3···H13[iv]	2.9300	H7A···O6[xii]	2.6700
C2···C6[vi]	3.3452 (19)	H7B···C5	2.7400
C6···C2[vi]	3.3452 (19)	H7B···C15[vi]	2.8000
C7···C15[vi]	3.5417 (19)	H7B···H11[xii]	2.5400
C8···O5[ii]	3.2199 (17)	H7B···O4[vi]	2.8000
C8···N2[ix]	3.3041 (17)	H7B···H5	2.3400
C10···C11[x]	3.5441 (19)	H7C···H5	2.3800
C11···C10[xi]	3.5441 (19)	H7C···C9[ii]	2.7200
C13···C14[v]	3.5805 (19)	H7C···C14[ii]	3.0300
C14···C14[v]	3.4735 (18)	H7C···C10[ii]	2.7800
C14···C13[v]	3.5805 (19)	H7C···C5	2.7700
C15···O5	3.0443 (17)	H8A···H12[v]	2.4100
C15···C7[vi]	3.5417 (19)	H8A···O4[ix]	2.8100
C16···O4	2.8629 (17)	H8B···H5	2.3300
C16···N3[i]	3.4273 (16)	H8B···O5[ii]	2.5600
C2···H6[iii]	2.8200	H8B···C5	2.7300
C2···H3	2.8500	H8C···N2[vi]	2.8600
C4···H6[iii]	2.7900	H8C···C5	2.7400
C4···H13[iv]	2.8800	H8C···H5	2.3400
C5···H7C	2.7700	H11···O6	2.6900
C5···H8B	2.7300	H11···C9[xi]	2.8900
C5···H8C	2.7400	H11···C10[xi]	2.9300
C5···H7B	2.7400	H11···C7[viii]	3.0900
C6···H3	2.7900	H11···H7B[viii]	2.5400
C7···H11[xii]	3.0900	H12···H8A[v]	2.4100
C7···H5	2.5700	H13···O1[xiii]	2.8900
C8···H2B[ix]	3.0200	H13···N3[xiii]	2.9300
C8···H2A[ix]	2.9200	H13···C4[xiii]	2.8800
C8···H5	2.5400	H13···O3[v]	2.9200

C9···H7C^{ii}	2.7200	H14···O3	2.5700
C9···H11^{x}	2.8900	H14···C13^{v}	3.0100
C10···H7C^{ii}	2.7800	H14···C14^{v}	3.1000
C10···H11^{x}	2.9300		
C4—O1—C7	118.31 (10)	H8A—C8—H8C	110.00
C6—O2—C8	117.70 (10)	H8B—C8—H8C	109.00
C15—O3—H3	109.00	O2—C8—H8A	109.00
C16—O6—H6	109.00	O2—C8—H8B	109.00
C2—N1—C6	116.29 (11)	C10—C9—C14	119.58 (11)
C2—N3—C4	116.65 (11)	C14—C9—C15	120.00 (11)
C2—N2—H2B	120.00	C10—C9—C15	120.29 (11)
H2A—N2—H2B	120.00	C9—C10—C16	121.70 (11)
C2—N2—H2A	120.00	C11—C10—C16	118.29 (11)
N1—C2—N3	124.93 (11)	C9—C10—C11	119.56 (12)
N2—C2—N3	117.41 (11)	C10—C11—C12	120.28 (13)
N1—C2—N2	117.65 (11)	C11—C12—C13	120.12 (13)
O1—C4—C5	124.92 (11)	C12—C13—C14	120.23 (13)
N3—C4—C5	123.60 (11)	C9—C14—C13	120.22 (12)
O1—C4—N3	111.49 (11)	O3—C15—C9	113.54 (11)
C4—C5—C6	114.89 (11)	O4—C15—C9	121.70 (11)
O2—C6—C5	124.62 (11)	O3—C15—O4	124.74 (12)
N1—C6—C5	123.62 (11)	O6—C16—C10	113.78 (11)
O2—C6—N1	111.76 (11)	O5—C16—O6	125.01 (12)
C4—C5—H5	123.00	O5—C16—C10	121.07 (11)
C6—C5—H5	123.00	C10—C11—H11	120.00
O1—C7—H7B	109.00	C12—C11—H11	120.00
O1—C7—H7C	109.00	C11—C12—H12	120.00
H7A—C7—H7C	109.00	C13—C12—H12	120.00
H7B—C7—H7C	110.00	C12—C13—H13	120.00
H7A—C7—H7B	110.00	C14—C13—H13	120.00
O1—C7—H7A	109.00	C9—C14—H14	120.00
O2—C8—H8C	109.00	C13—C14—H14	120.00
H8A—C8—H8B	109.00		
C7—O1—C4—N3	177.86 (11)	C15—C9—C10—C11	175.14 (12)
C7—O1—C4—C5	−1.95 (18)	C15—C9—C10—C16	−12.75 (18)
C8—O2—C6—N1	−179.91 (11)	C10—C9—C14—C13	−0.7 (2)
C8—O2—C6—C5	−0.14 (19)	C15—C9—C14—C13	−176.50 (12)
C6—N1—C2—N2	−179.02 (12)	C10—C9—C15—O3	147.33 (12)
C6—N1—C2—N3	1.7 (2)	C10—C9—C15—O4	−34.25 (19)
C2—N1—C6—O2	178.89 (11)	C14—C9—C15—O3	−36.88 (17)
C2—N1—C6—C5	−0.89 (19)	C14—C9—C15—O4	141.55 (14)
C4—N3—C2—N1	−1.06 (19)	C9—C10—C11—C12	1.4 (2)
C4—N3—C2—N2	179.63 (12)	C16—C10—C11—C12	−170.97 (13)
C2—N3—C4—O1	179.80 (11)	C9—C10—C16—O5	−49.60 (19)
C2—N3—C4—C5	−0.39 (19)	C9—C10—C16—O6	134.45 (13)
O1—C4—C5—C6	−179.18 (12)	C11—C10—C16—O5	122.61 (15)
N3—C4—C5—C6	1.04 (19)	C11—C10—C16—O6	−53.35 (16)
C4—C5—C6—O2	179.89 (12)	C10—C11—C12—C13	−0.8 (2)

C4—C5—C6—N1	−0.36 (19)	C11—C12—C13—C14	−0.6 (2)
C14—C9—C10—C11	−0.67 (19)	C12—C13—C14—C9	1.3 (2)
C14—C9—C10—C16	171.44 (12)		

Symmetry codes: (i) −x+2, y−1/2, −z+1/2; (ii) −x+2, −y, −z; (iii) −x+2, y+1/2, −z+1/2; (iv) x+1, y, z; (v) −x+1, −y, −z; (vi) −x+2, −y+1, −z; (vii) x, −y+1/2, z+1/2; (viii) x−1, −y+1/2, z+1/2; (ix) x, −y+1/2, z−1/2; (x) −x+1, y+1/2, −z+1/2; (xi) −x+1, y−1/2, −z+1/2; (xii) x+1, −y+1/2, z−1/2; (xiii) x−1, y, z.

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O5[iii]	0.86	2.06	2.8704 (15)	156
N2—H2B···O4	0.86	2.01	2.8609 (15)	173
O3—H3···N1	0.82	1.87	2.6881 (14)	171
O6—H6···N3[i]	0.82	1.86	2.6738 (14)	172
C8—H8B···O5[ii]	0.96	2.56	3.2199 (17)	126

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

Fig. 1

Fig. 2

Fig. 3