Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Dimethyl 2-nitrobiphenyl-4,4′-dicarboxylate

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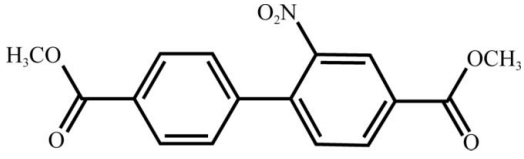
Received 17 February 2014; accepted 23 February 2014

Key indicators: single-crystal X-ray study; T = 90 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.097; data-to-parameter ratio = 13.9.

The title compound, $C_{16}H_{13}NO_6$, exhibits a biphenyl unit with a dihedral angle between the two aryl rings of 56.01 (5)°. The two ester groups vary slightly from planarity, with aryl–ester dihedral angles of 4.57 (5) and 16.73 (5)°. The nitro group is turned from the aromatic unit with an aryl–nitro dihedral angle of 48.66 (4)°. In the crystal, molecules are connected by weak C—H···O interactions, forming a three-dimensional network.

Related literature

For the synthesis of the title compound, see: Olkhovik *et al.* (2008). For coordination polymers featuring the 2-nitrobiphenyl-4,4′-dicarboxylate linker, see: Jing *et al.* (2012).



Experimental

Crystal data

$C_{16}H_{13}NO_6$
M_r = 315.27
Monoclinic, $C2/c$
a = 20.3958 (17) Å
b = 8.3334 (6) Å
c = 18.9386 (14) Å
β = 118.342 (7)°
V = 2833.1 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 90 K
0.25 × 0.20 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.972, T_{max} = 0.983
19059 measured reflections
2918 independent reflections
2360 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.097
S = 1.04
2918 reflections
210 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O6^{i}	0.95	2.50	3.3405 (16)	148
C13—H13A···O2^{ii}	0.95	2.39	3.2435 (16)	150
C14—H14A···O4^{iii}	0.95	2.59	3.3954 (16)	143

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

VCMV thanks the the UMass Dartmouth Office of Undergraduate Research Award and the Urban Massachusetts Louis Stokes Alliance for Minority Participation (UMLSAMP) for funding. DRM gratefully acknowledges support from the National Science Foundation (CHE-1229339).

Supporting information for this paper is available from the IUCr electronic archives (Reference: FF2127).

References

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Jing, X.-H., Yi, X.-C., Gao, E.-Q. & Blatov, V. A. (2012). *Dalton Trans.* **41**, 14316–14328.
Olkhovik, V. K., Vasilevskii, D. A., Pap, A. A., Kalechyts, G. V., Matveienko, Y. V., Baran, A. G., Halinouski, N. A. & Petushok, V. G. (2008). *ARKIVOC*, **ix**, 69–93.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2014). E**70**, o371 [doi:10.1107/S1600536814004218]

Dimethyl 2-nitrobiphenyl-4,4′-dicarboxylate

Vanessa C. M. Vieira, James A. Golen, Arnold L. Rheingold and David R. Manke

S1. Comment

Biphenyl-4,4′-dicarboxylate and its derivatives have become prevalent linkers in the preparation of metal-organic frameworks (MOFs). The ability to incorporate different functional groups into the pores of MOFs is one advantage of this class of materials. As a part of our efforts in this arena, we prepared the previously reported dimethyl 2-nitro-biphenyl-4,4′-dicarboxylate (Olkhovik *et al.* 2008) and report its structure herein.

The structure of the title compound is shown in Figure 1. The structure has a torsion angle of 56.01 (5)° between the two aryl rings. The ester groups vary slightly from the planes of the aromatic rings, with aryl-ester dihedral angles of 4.57 (5)° and 16.73 (5)°. The nitro group shows an aryl-nitro torsion angle of 48.66 (4)°. No π-π interactions were noted between the aromatic rings. The packing for the title compound is shown in Figure 2.

S2. Experimental

The compound was prepared by literature procedure (Olkhovik *et al.* 2008). Crystals suitable for single-crystal X-ray analysis were grown by slow evaporation of an ethanol solution.

S3. Refinement

Data corrected for absorption with *SADABS* (Bruker, 2005) and structure solved by direct methods (*SHELXS*) and all non-hydrogen atoms refined anisotropically by full matrix least squares on F^2 (*SHELXL* (Sheldrick, 2008)). All hydrogen atoms were placed in calculated positions and then refined with riding model with C—H lengths of 0.95 Å for (CH) and 0.98 Å for (CH_3) and with isotropic displacement parameters set to 1.20 and 1.50 times U_{eq} of the parent C atom.

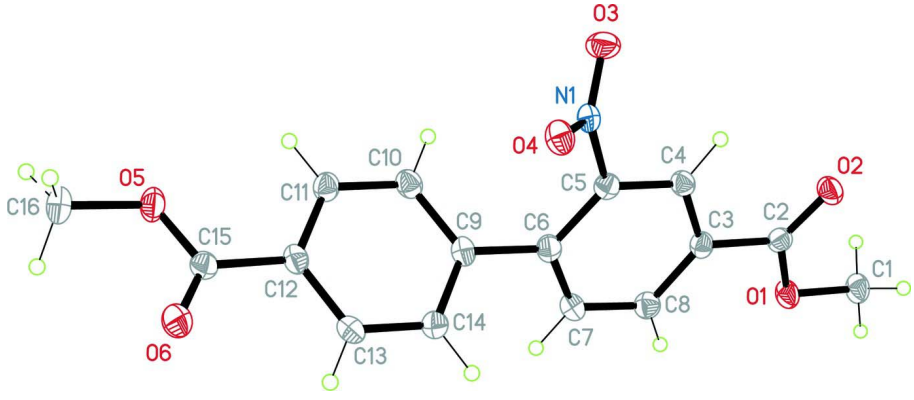


Figure 1

Molecular structure of the title compound, showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as spheres of arbitrary radius.

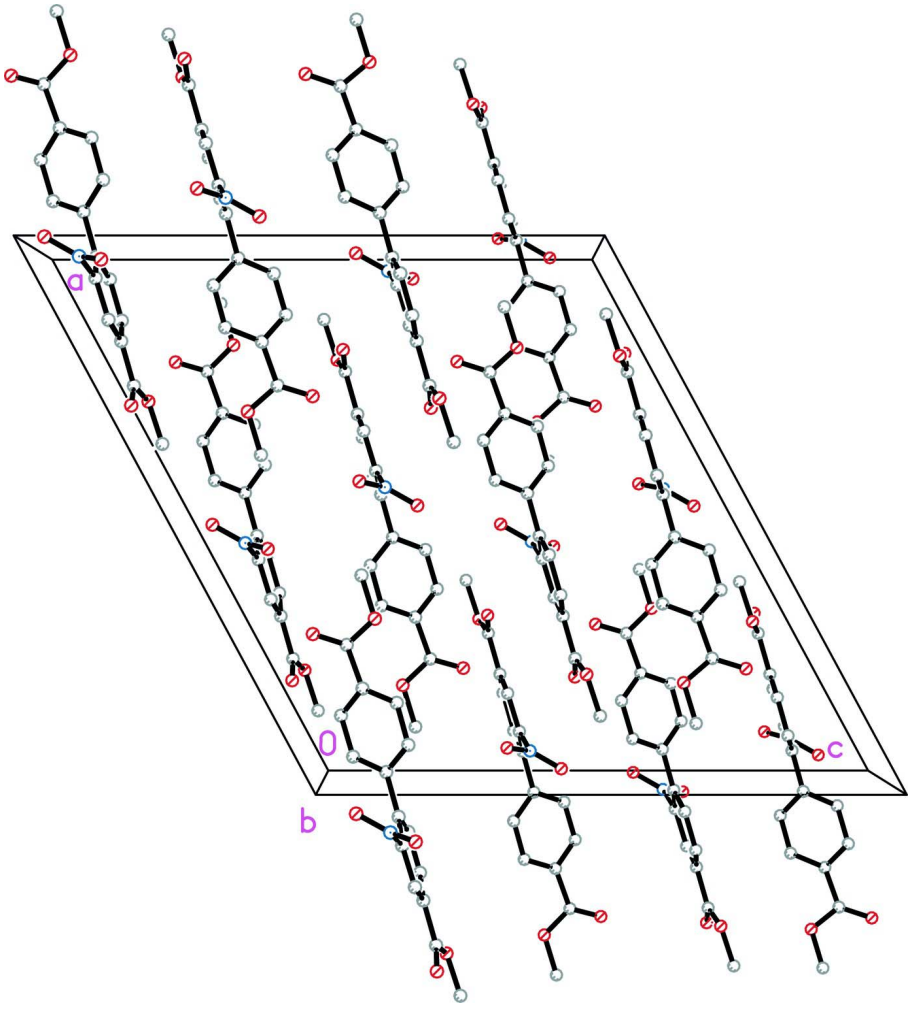


Figure 2

Molecular packing of the title compound.

Dimethyl 2-nitrobiphenyl-4,4′-dicarboxylate

Crystal data

$C_{16}H_{13}NO_6$
$M_r = 315.27$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 20.3958\ (17)$ Å
$b = 8.3334\ (6)$ Å
$c = 18.9386\ (14)$ Å
$\beta = 118.342\ (7)°$
$V = 2833.1\ (4)$ Å^3
$Z = 8$

$F(000) = 1312$
$D_x = 1.478$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7800 reflections
$\theta = 2.4–26.4°$
$\mu = 0.12$ mm^{-1}
$T = 90$ K
Block, colorless
0.25 × 0.20 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube

Graphite monochromator
φ and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.972, T_{max} = 0.983
19059 measured reflections
2918 independent reflections
2360 reflections with $I > 2\sigma(I)$

R_{int} = 0.029
θ_{max} = 26.5°, θ_{min} = 2.7°
h = −25→25
k = −10→10
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.097
S = 1.04
2918 reflections
210 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 1.4779P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.20578 (5)	0.35494 (11)	0.07049 (6)	0.0225 (2)
O2	0.19389 (5)	0.61983 (11)	0.04893 (6)	0.0241 (2)
O3	0.43918 (5)	0.91646 (12)	0.14067 (6)	0.0294 (3)
O4	0.48248 (5)	0.82157 (11)	0.06479 (5)	0.0231 (2)
O5	0.81617 (5)	0.57857 (12)	0.26263 (6)	0.0250 (2)
O6	0.78697 (5)	0.47958 (13)	0.14129 (6)	0.0304 (3)
N1	0.44911 (6)	0.80702 (13)	0.10372 (6)	0.0197 (3)
C1	0.12899 (7)	0.35323 (17)	0.05505 (9)	0.0239 (3)
H1A	0.1125	0.2420	0.0527	0.036*
H1B	0.0974	0.4064	0.0038	0.036*
H1C	0.1251	0.4101	0.0982	0.036*
C2	0.23120 (7)	0.49999 (15)	0.06615 (8)	0.0186 (3)
C3	0.31111 (7)	0.49867 (15)	0.08548 (7)	0.0182 (3)
C4	0.34421 (7)	0.64598 (16)	0.08921 (7)	0.0179 (3)
H4A	0.3169	0.7428	0.0807	0.022*
C5	0.41786 (7)	0.64881 (16)	0.10550 (7)	0.0178 (3)
C6	0.46139 (7)	0.51142 (16)	0.12059 (8)	0.0191 (3)
C7	0.42667 (7)	0.36580 (16)	0.11802 (8)	0.0210 (3)
H7A	0.4546	0.2693	0.1289	0.025*

C8	0.35235 (7)	0.35825 (16)	0.10001 (8)	0.0206 (3)
H8A	0.3297	0.2573	0.0976	0.025*
C9	0.54141 (7)	0.51468 (16)	0.14053 (8)	0.0188 (3)
C10	0.59328 (7)	0.60408 (16)	0.20508 (8)	0.0206 (3)
H10A	0.5775	0.6654	0.2366	0.025*
C11	0.66769 (7)	0.60418 (16)	0.22374 (8)	0.0201 (3)
H11A	0.7030	0.6635	0.2685	0.024*
C12	0.69054 (7)	0.51723 (15)	0.17669 (8)	0.0189 (3)
C13	0.63915 (7)	0.42696 (16)	0.11247 (8)	0.0204 (3)
H13A	0.6548	0.3671	0.0804	0.024*
C14	0.56517 (7)	0.42431 (16)	0.09521 (8)	0.0197 (3)
H14A	0.5304	0.3604	0.0521	0.024*
C15	0.76862 (7)	0.52180 (16)	0.19011 (8)	0.0206 (3)
C16	0.89312 (7)	0.58733 (19)	0.27969 (9)	0.0289 (3)
H16A	0.9252	0.6008	0.3375	0.043*
H16B	0.8997	0.6789	0.2512	0.043*
H16C	0.9066	0.4882	0.2619	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0154 (5)	0.0204 (5)	0.0315 (5)	−0.0001 (4)	0.0110 (4)	0.0023 (4)
O2	0.0175 (5)	0.0207 (5)	0.0339 (5)	0.0015 (4)	0.0119 (4)	−0.0003 (4)
O3	0.0263 (5)	0.0246 (5)	0.0434 (6)	−0.0047 (4)	0.0214 (5)	−0.0125 (5)
O4	0.0193 (5)	0.0267 (5)	0.0272 (5)	0.0007 (4)	0.0142 (4)	0.0032 (4)
O5	0.0147 (4)	0.0331 (6)	0.0258 (5)	−0.0010 (4)	0.0084 (4)	−0.0031 (4)
O6	0.0215 (5)	0.0371 (6)	0.0365 (6)	−0.0022 (4)	0.0170 (5)	−0.0112 (5)
N1	0.0128 (5)	0.0225 (6)	0.0228 (6)	0.0013 (4)	0.0075 (4)	−0.0013 (5)
C1	0.0149 (6)	0.0256 (7)	0.0309 (7)	−0.0014 (5)	0.0107 (6)	0.0017 (6)
C2	0.0181 (6)	0.0199 (7)	0.0176 (6)	−0.0011 (5)	0.0082 (5)	−0.0019 (5)
C3	0.0160 (6)	0.0225 (7)	0.0158 (6)	0.0004 (5)	0.0073 (5)	−0.0002 (5)
C4	0.0176 (6)	0.0199 (7)	0.0163 (6)	0.0025 (5)	0.0080 (5)	0.0000 (5)
C5	0.0181 (6)	0.0205 (7)	0.0158 (6)	−0.0007 (5)	0.0089 (5)	−0.0010 (5)
C6	0.0173 (6)	0.0248 (7)	0.0152 (6)	0.0022 (5)	0.0076 (5)	−0.0008 (5)
C7	0.0192 (6)	0.0205 (7)	0.0230 (7)	0.0035 (5)	0.0096 (5)	0.0011 (5)
C8	0.0202 (6)	0.0203 (7)	0.0213 (7)	−0.0008 (5)	0.0099 (5)	0.0005 (5)
C9	0.0158 (6)	0.0204 (7)	0.0198 (7)	0.0026 (5)	0.0081 (5)	0.0036 (5)
C10	0.0201 (6)	0.0245 (7)	0.0184 (6)	0.0038 (5)	0.0100 (5)	0.0004 (5)
C11	0.0178 (6)	0.0228 (7)	0.0172 (6)	0.0009 (5)	0.0064 (5)	0.0007 (5)
C12	0.0160 (6)	0.0188 (7)	0.0214 (7)	0.0026 (5)	0.0086 (5)	0.0034 (5)
C13	0.0205 (6)	0.0189 (7)	0.0235 (7)	0.0036 (5)	0.0119 (5)	−0.0001 (5)
C14	0.0182 (6)	0.0193 (7)	0.0199 (6)	0.0006 (5)	0.0077 (5)	−0.0002 (5)
C15	0.0187 (6)	0.0164 (7)	0.0258 (7)	0.0006 (5)	0.0099 (6)	−0.0001 (5)
C16	0.0155 (6)	0.0345 (8)	0.0337 (8)	−0.0018 (6)	0.0094 (6)	−0.0025 (7)

Geometric parameters (Å, º)

O1—C2	1.3331 (16)	C6—C9	1.4906 (17)
O1—C1	1.4504 (14)	C7—C8	1.3883 (18)
O2—C2	1.2030 (16)	C7—H7A	0.9500
O3—N1	1.2235 (14)	C8—H8A	0.9500
O4—N1	1.2240 (14)	C9—C14	1.3905 (19)
O5—C15	1.3364 (16)	C9—C10	1.3932 (18)
O5—C16	1.4461 (15)	C10—C11	1.3853 (17)
O6—C15	1.2030 (17)	C10—H10A	0.9500
N1—C5	1.4716 (17)	C11—C12	1.3894 (18)
C1—H1A	0.9800	C11—H11A	0.9500
C1—H1B	0.9800	C12—C13	1.3905 (18)
C1—H1C	0.9800	C12—C15	1.4891 (18)
C2—C3	1.4913 (17)	C13—C14	1.3841 (17)
C3—C4	1.3866 (18)	C13—H13A	0.9500
C3—C8	1.3902 (18)	C14—H14A	0.9500
C4—C5	1.3828 (17)	C16—H16A	0.9800
C4—H4A	0.9500	C16—H16B	0.9800
C5—C6	1.3928 (18)	C16—H16C	0.9800
C6—C7	1.3942 (19)		
C2—O1—C1	114.26 (10)	C7—C8—H8A	120.1
C15—O5—C16	115.38 (11)	C3—C8—H8A	120.1
O3—N1—O4	124.13 (11)	C14—C9—C10	119.29 (12)
O3—N1—C5	117.62 (10)	C14—C9—C6	119.63 (11)
O4—N1—C5	118.25 (10)	C10—C9—C6	121.06 (12)
O1—C1—H1A	109.5	C11—C10—C9	120.51 (12)
O1—C1—H1B	109.5	C11—C10—H10A	119.7
H1A—C1—H1B	109.5	C9—C10—H10A	119.7
O1—C1—H1C	109.5	C10—C11—C12	119.77 (12)
H1A—C1—H1C	109.5	C10—C11—H11A	120.1
H1B—C1—H1C	109.5	C12—C11—H11A	120.1
O2—C2—O1	123.70 (11)	C11—C12—C13	120.00 (12)
O2—C2—C3	123.34 (12)	C11—C12—C15	122.29 (12)
O1—C2—C3	112.95 (11)	C13—C12—C15	117.66 (12)
C4—C3—C8	120.06 (12)	C14—C13—C12	119.98 (12)
C4—C3—C2	117.06 (11)	C14—C13—H13A	120.0
C8—C3—C2	122.87 (11)	C12—C13—H13A	120.0
C5—C4—C3	118.53 (11)	C13—C14—C9	120.40 (12)
C5—C4—H4A	120.7	C13—C14—H14A	119.8
C3—C4—H4A	120.7	C9—C14—H14A	119.8
C4—C5—C6	123.42 (12)	O6—C15—O5	123.68 (12)
C4—C5—N1	116.52 (11)	O6—C15—C12	123.98 (12)
C6—C5—N1	120.03 (11)	O5—C15—C12	112.34 (12)
C5—C6—C7	116.40 (12)	O5—C16—H16A	109.5
C5—C6—C9	123.47 (12)	O5—C16—H16B	109.5
C7—C6—C9	120.11 (11)	H16A—C16—H16B	109.5

C8—C7—C6	121.69 (12)	O5—C16—H16C	109.5
C8—C7—H7A	119.2	H16A—C16—H16C	109.5
C6—C7—H7A	119.2	H16B—C16—H16C	109.5
C7—C8—C3	119.86 (12)		

C1—O1—C2—O2	−1.86 (18)	C2—C3—C8—C7	−179.73 (11)
C1—O1—C2—C3	177.73 (10)	C5—C6—C9—C14	−125.48 (14)
O2—C2—C3—C4	4.93 (19)	C7—C6—C9—C14	55.96 (17)
O1—C2—C3—C4	−174.66 (11)	C5—C6—C9—C10	56.00 (18)
O2—C2—C3—C8	−175.54 (13)	C7—C6—C9—C10	−122.56 (14)
O1—C2—C3—C8	4.87 (18)	C14—C9—C10—C11	0.48 (19)
C8—C3—C4—C5	1.65 (19)	C6—C9—C10—C11	179.01 (12)
C2—C3—C4—C5	−178.80 (11)	C9—C10—C11—C12	1.38 (19)
C3—C4—C5—C6	−1.81 (19)	C10—C11—C12—C13	−1.77 (19)
C3—C4—C5—N1	176.20 (11)	C10—C11—C12—C15	175.57 (12)
O3—N1—C5—C4	48.64 (15)	C11—C12—C13—C14	0.28 (19)
O4—N1—C5—C4	−130.48 (12)	C15—C12—C13—C14	−177.17 (12)
O3—N1—C5—C6	−133.27 (12)	C12—C13—C14—C9	1.60 (19)
O4—N1—C5—C6	47.60 (16)	C10—C9—C14—C13	−1.98 (19)
C4—C5—C6—C7	0.46 (19)	C6—C9—C14—C13	179.47 (12)
N1—C5—C6—C7	−177.48 (11)	C16—O5—C15—O6	0.21 (19)
C4—C5—C6—C9	−178.14 (12)	C16—O5—C15—C12	−179.65 (11)
N1—C5—C6—C9	3.91 (19)	C11—C12—C15—O6	−162.42 (13)
C5—C6—C7—C8	1.06 (19)	C13—C12—C15—O6	15.0 (2)
C9—C6—C7—C8	179.72 (12)	C11—C12—C15—O5	17.44 (18)
C6—C7—C8—C3	−1.2 (2)	C13—C12—C15—O5	−165.16 (12)
C4—C3—C8—C7	−0.20 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4*A*···O6^{i}	0.95	2.50	3.3405 (16)	148
C13—H13*A*···O2^{ii}	0.95	2.39	3.2435 (16)	150
C14—H14*A*···O4^{iii}	0.95	2.59	3.3954 (16)	143

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