Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Bromo-*N*-(3,4,5-trimethoxybenzylidene)aniline

Aliakbar Dehno Khalaji,[a]‡ Matthias Weil,[b] Kazuma Gotoh[c] and Hiroyuki Ishida[c]*

[a]Department of Chemistry, Faculty of Science, Golestan University, Gorgan, Iran, [b]Institute for Chemical Technology and Analytics, Division of Structural Chemistry, Vienna University of Technology, Getreidemarkt 9/164-SC, A-1060 Vienna, Austria, and [c]Department of Chemistry, Faculty of Science, Okayama University, Okayama 700-8530, Japan
Correspondence e-mail: ishidah@cc.okayama-u.ac.jp

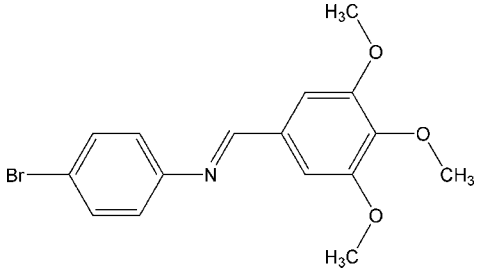
Received 26 January 2009; accepted 28 January 2009

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.025; wR factor = 0.065; data-to-parameter ratio = 18.1.

The title compound, $C_{16}H_{16}BrNO_3$, adopts an *E* configuration with respect to the imine C=N bond. The two benzene rings are twisted with respect to each other at an angle of 38.3 (1)°. In the crystal structure, molecules are connected by weak bifurcated C—H···(O, O) hydrogen bonds, forming a helical chain along the *b* axis.

Related literature

The structure of the isotypic 4-chloro compound was reported by Khalaji *et al.* (2009). For structures containing a 4-bromoaniline unit, see: Khalaji *et al.* (2007); Khalaji & Harrison (2008).



Experimental

Crystal data

$C_{16}H_{16}BrNO_3$ M_r = 350.21
Monoclinic, $P2_1$
a = 7.1951 (4) Å
b = 8.3722 (5) Å
c = 13.2882 (8) Å
β = 104.413 (3)°
V = 775.27 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.66 mm^{-1}
T = 296 (2) K
0.40 × 0.30 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
T_{min} = 0.403, T_{max} = 0.671
18229 measured reflections
3497 independent reflections
3064 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.065
S = 1.09
3497 reflections
193 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}
Absolute structure: Flack (1983), 1511 Friedel pairs
Flack parameter: 0.012 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1^{i}	0.93	2.63	3.272 (2)	127
C7—H7···O2^{i}	0.93	2.63	3.553 (3)	172

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2308).

References

Bruker (2006). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Khalaji, A. D., Asghari, J., Fejfarová, K. & Dušek, M. (2009). *Acta Cryst.* E**65**, o253.
Khalaji, A. D. & Harrison, W. T. A. (2008). *Anal. Sci.* **24**, x3–x4.
Khalaji, A. D., Slawin, A. M. Z. & Woollins, J. D. (2007). *Acta Cryst.* E**63**, o4257.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

‡ Additional correspondence author, e-mail: alidkhalaji@yahoo.com.

supporting information

Acta Cryst. (2009). E**65**, o436 [doi:10.1107/S1600536809003432]

4-Bromo-*N*-(3,4,5-trimethoxybenzylidene)aniline

Aliakbar Dehno Khalaji, Matthias Weil, Kazuma Gotoh and Hiroyuki Ishida

S1. Comment

Recently, we reported two Schiff-base compounds with 4-bromoaniline units that have been structurally characterized (Khalaji *et al.*, 2007; Khalaji & Harrison, 2008). In continuation of these studies, the title compound was prepared and its structure has been determined.

An *ORTEP* plot, with the atomic numbering scheme is depicted in Fig. 1. The two benzene rings are twisted with respect to each other at an angle of 38.3 (1)°. In the crystal structure, the molecules are connected by weak bifurcated C—H···(O, O) hydrogen bonds, forming a helical chain along the *b* axis.

The C7═N1 bond length of 1.268 (3) Å conforms to the value for a double bond, and is slightly shorter than the corresponding bond length in *N*-(2-benzylidenepropylidene)-4-bromoaniline [C23═N23 1.288 (6) Å; Khalaji *et al.*, 2007] and *β*-phenylcinnamaldehyde-4-bromoaniline [C7═N1 1.277 (4) Å; Khalaji & Harrison, 2008]. The C4—N1 bond length of 1.421 (2) Å conforms to the value for a single bond, and, in turn, is slightly longer than the corresponding bond length in *N*-(2-benzylidenepropylidene)-4-bromoaniline [C24—N23 1.411 (7) Å] and *β*-phenylcinnamaldehyde-4-bromoaniline [C6—N1 1.407 (4) Å]. All other bond lengths in the three related Schiff-base compounds are quite similar. For the title compound, the torsion angle, C8—C7—N1—C4, is 179.20 (18)°, indicating a virtually planar *E*-configuration with respect to the imine C═N bond (Khalaji *et al.*, 2007; Khalaji & Harrison, 2008).

In comparison with the isotypic structure of $C_{16}H_{16}ClNO_3$ (Dehno Khalaji *et al.*, 2009), all interatomic distances and angles (except those involving the halogen atom) are very similar.

S2. Experimental

The title compound was prepared by the reaction of 3,4,5-trimethoxybenzaldehyde (1 mmol, 0.196 g) and 4-bromoaniline (1 mmol, 0.172 g), which were dissolved in methanol (10 ml). The mixture was stirred at room temperature for 30 min. Colourless single crystals suitable for X-ray structure analysis were obtained by recrystallization from a methanol/chloroform (1:1 *v*/*v*) solution.

S3. Refinement

H atoms were positioned geometrically (C—H = 0.93 or 0.96 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C), allowing for free rotation of the methyl groups.

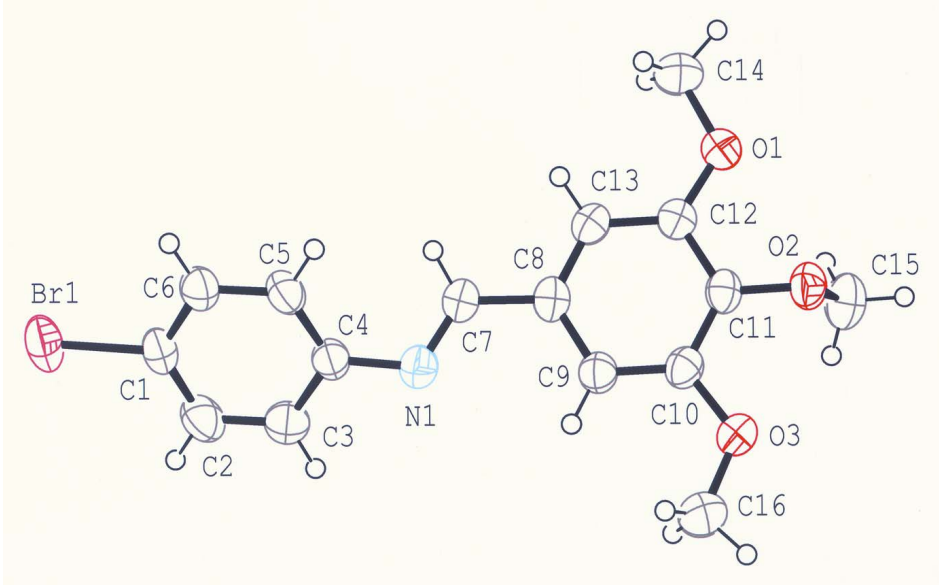


Figure 1

The molecular structure of the title compound, with the atom-labelling. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. Hydrogen atoms are denoted by spheres of arbitrary radius.

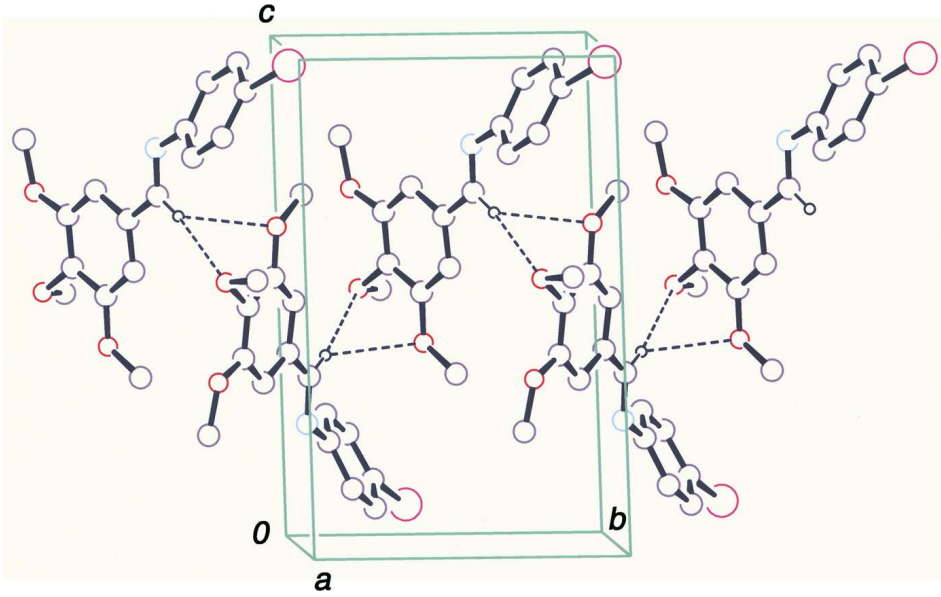


Figure 2

A partial packing diagram, viewed along the *a* axis. H atoms not involved in the C—H···O hydrogen bonds have been omitted.

4-Bromo-*N*-(3,4,5-trimethoxybenzylidene)aniline

Crystal data

$C_{16}H_{16}BrNO_3$
M_r = 350.21
Monoclinic, $P2_1$

Hall symbol: P 2yb
a = 7.1951 (4) Å
b = 8.3722 (5) Å

c = 13.2882 (8) Å
β = 104.413 (3)°
V = 775.27 (8) Å^3
Z = 2
F(000) = 356
D_x = 1.500 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 9957 reflections
θ = 2.9–29.0°
μ = 2.66 mm^{-1}
T = 296 K
Block, colourless
0.40 × 0.30 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
T_{min} = 0.403, T_{max} = 0.671

18229 measured reflections
3497 independent reflections
3064 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 28.0°, θ_{min} = 2.9°
h = −9→9
k = −10→10
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.065
S = 1.09
3497 reflections
193 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0339P)^2 + 0.0173P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}
Absolute structure: Flack (1983), 1511 Friedel pairs
Absolute structure parameter: 0.012 (6)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.99402 (3)	0.96811 (5)	0.988948 (16)	0.06694 (9)
O1	−0.26888 (18)	0.4772 (2)	0.36904 (9)	0.0522 (3)
O2	−0.46171 (19)	0.29368 (19)	0.46988 (11)	0.0498 (3)
O3	−0.3668 (2)	0.2687 (2)	0.67538 (12)	0.0600 (4)
N1	0.2656 (2)	0.6014 (2)	0.79047 (14)	0.0502 (4)
C1	0.7715 (3)	0.8470 (2)	0.92769 (15)	0.0450 (4)
C2	0.6245 (3)	0.8386 (3)	0.97682 (18)	0.0595 (6)
H2	0.6355	0.8891	1.0404	0.071*

C3	0.4614 (4)	0.7546 (3)	0.9308 (2)	0.0604 (6)
H3	0.3634	0.7458	0.9648	0.072*
C4	0.4402 (3)	0.6827 (2)	0.83465 (15)	0.0436 (4)
C5	0.5939 (3)	0.6886 (3)	0.78856 (16)	0.0478 (5)
H5	0.5850	0.6368	0.7256	0.057*
C6	0.7594 (3)	0.7705 (3)	0.83532 (17)	0.0498 (5)
H6	0.8618	0.7736	0.8043	0.060*
C7	0.2013 (3)	0.6071 (2)	0.69260 (16)	0.0427 (4)
H7	0.2700	0.6651	0.6542	0.051*
C8	0.0246 (3)	0.5272 (2)	0.63686 (16)	0.0411 (4)
C9	−0.0833 (3)	0.4364 (2)	0.69019 (15)	0.0432 (5)
H9	−0.0454	0.4266	0.7621	0.052*
C10	−0.2484 (3)	0.3611 (2)	0.63335 (16)	0.0430 (4)
C11	−0.3059 (3)	0.3758 (2)	0.52599 (16)	0.0411 (4)
C12	−0.1991 (2)	0.4695 (3)	0.47377 (12)	0.0403 (3)
C13	−0.0336 (3)	0.5450 (2)	0.53043 (16)	0.0417 (4)
H13	0.0381	0.6078	0.4963	0.050*
C14	−0.1666 (4)	0.5679 (4)	0.3108 (2)	0.0785 (8)
H14A	−0.1641	0.6779	0.3316	0.118*
H14B	−0.2287	0.5591	0.2382	0.118*
H14C	−0.0377	0.5284	0.3232	0.118*
C15	−0.6378 (3)	0.3769 (4)	0.4588 (2)	0.0673 (7)
H15A	−0.6588	0.3972	0.5262	0.101*
H15B	−0.7412	0.3133	0.4189	0.101*
H15C	−0.6324	0.4765	0.4238	0.101*
C16	−0.3111 (5)	0.2366 (4)	0.7828 (2)	0.0761 (8)
H16A	−0.1813	0.1983	0.8010	0.114*
H16B	−0.3944	0.1569	0.7997	0.114*
H16C	−0.3198	0.3327	0.8208	0.114*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.05738 (13)	0.06441 (15)	0.06520 (14)	−0.01188 (12)	−0.01083 (9)	−0.00513 (15)
O1	0.0556 (7)	0.0567 (8)	0.0431 (6)	−0.0113 (9)	0.0100 (5)	−0.0013 (9)
O2	0.0463 (7)	0.0467 (8)	0.0547 (8)	−0.0096 (7)	0.0092 (6)	−0.0106 (7)
O3	0.0588 (9)	0.0704 (10)	0.0506 (9)	−0.0236 (7)	0.0135 (7)	0.0033 (7)
N1	0.0478 (9)	0.0533 (10)	0.0498 (11)	−0.0107 (8)	0.0126 (8)	−0.0056 (8)
C1	0.0423 (9)	0.0395 (11)	0.0456 (11)	−0.0019 (8)	−0.0036 (8)	−0.0007 (8)
C2	0.0630 (13)	0.0664 (15)	0.0468 (12)	−0.0018 (11)	0.0094 (10)	−0.0168 (10)
C3	0.0582 (13)	0.0769 (18)	0.0500 (14)	−0.0086 (11)	0.0208 (11)	−0.0114 (10)
C4	0.0425 (9)	0.0441 (12)	0.0418 (10)	−0.0030 (9)	0.0060 (8)	−0.0009 (8)
C5	0.0463 (10)	0.0538 (14)	0.0400 (11)	−0.0014 (10)	0.0044 (8)	−0.0099 (9)
C6	0.0408 (10)	0.0595 (14)	0.0466 (12)	−0.0002 (9)	0.0060 (9)	−0.0020 (10)
C7	0.0376 (9)	0.0393 (10)	0.0504 (12)	−0.0019 (8)	0.0095 (8)	−0.0013 (8)
C8	0.0355 (9)	0.0337 (9)	0.0524 (11)	0.0014 (7)	0.0076 (8)	−0.0044 (7)
C9	0.0418 (9)	0.0433 (13)	0.0442 (9)	−0.0024 (7)	0.0101 (7)	−0.0026 (7)
C10	0.0434 (10)	0.0377 (11)	0.0497 (11)	−0.0038 (8)	0.0148 (8)	−0.0041 (8)

C11	0.0378 (9)	0.0345 (10)	0.0503 (11)	−0.0006 (8)	0.0097 (8)	−0.0080 (8)
C12	0.0412 (8)	0.0356 (8)	0.0449 (8)	0.0025 (11)	0.0122 (6)	−0.0028 (11)
C13	0.0397 (9)	0.0371 (10)	0.0502 (11)	−0.0020 (7)	0.0148 (8)	−0.0011 (8)
C14	0.0817 (17)	0.102 (2)	0.0505 (14)	−0.0239 (16)	0.0142 (13)	0.0169 (14)
C15	0.0429 (11)	0.0708 (17)	0.0828 (18)	−0.0044 (12)	0.0056 (11)	−0.0027 (13)
C16	0.0790 (17)	0.086 (2)	0.0611 (18)	−0.0228 (16)	0.0134 (15)	0.0180 (13)

Geometric parameters (Å, º)

Br1—C1	1.8992 (19)	C7—C8	1.464 (3)
O1—C12	1.358 (2)	C7—H7	0.9300
O1—C14	1.414 (3)	C8—C9	1.399 (3)
O2—C11	1.367 (2)	C8—C13	1.379 (3)
O2—C15	1.421 (3)	C9—H9	0.9300
O3—C16	1.409 (3)	C10—C9	1.390 (3)
O3—C10	1.368 (2)	C11—C10	1.388 (3)
C1—C2	1.376 (3)	C12—C13	1.392 (3)
C1—C6	1.368 (3)	C12—C11	1.397 (3)
C2—H2	0.9300	C13—H13	0.9300
C3—C2	1.375 (3)	C14—H14A	0.9600
C3—H3	0.9300	C14—H14B	0.9600
C4—N1	1.421 (2)	C14—H14C	0.9600
C4—C3	1.386 (3)	C15—H15A	0.9600
C4—C5	1.392 (3)	C15—H15B	0.9600
C5—C6	1.380 (3)	C15—H15C	0.9600
C5—H5	0.9300	C16—H16A	0.9600
C6—H6	0.9300	C16—H16B	0.9600
C7—N1	1.268 (3)	C16—H16C	0.9600
O1—C12—C11	115.25 (16)	C6—C1—Br1	119.56 (16)
O1—C12—C13	125.52 (17)	C6—C1—C2	121.14 (18)
O1—C14—H14A	109.5	C6—C5—C4	120.69 (19)
O1—C14—H14B	109.5	C6—C5—H5	119.7
O1—C14—H14C	109.5	C7—N1—C4	117.70 (17)
O2—C11—C10	120.65 (17)	C8—C7—H7	118.3
O2—C11—C12	119.28 (17)	C8—C9—H9	120.7
O2—C15—H15A	109.5	C8—C13—C12	120.43 (18)
O2—C15—H15B	109.5	C8—C13—H13	119.8
O2—C15—H15C	109.5	C9—C8—C7	120.90 (18)
O3—C10—C9	124.70 (18)	C10—O3—C16	118.18 (19)
O3—C10—C11	114.39 (17)	C10—C9—C8	118.58 (18)
O3—C16—H16A	109.5	C10—C9—H9	120.7
O3—C16—H16B	109.5	C10—C11—C12	120.00 (17)
O3—C16—H16C	109.5	C11—O2—C15	113.49 (18)
N1—C7—C8	123.41 (18)	C11—C10—C9	120.91 (17)
N1—C7—H7	118.3	C12—O1—C14	118.41 (17)
C1—C2—H2	120.4	C13—C8—C9	120.83 (17)
C1—C6—C5	119.45 (19)	C13—C8—C7	118.27 (17)

C1—C6—H6	120.3	C13—C12—C11	119.23 (16)
C2—C1—Br1	119.30 (15)	C12—C13—H13	119.8
C2—C3—C4	121.2 (2)	H14A—C14—H14B	109.5
C2—C3—H3	119.4	H14A—C14—H14C	109.5
C3—C2—C1	119.1 (2)	H14B—C14—H14C	109.5
C3—C2—H2	120.4	H15A—C15—H15B	109.5
C3—C4—N1	118.17 (18)	H15A—C15—H15C	109.5
C3—C4—C5	118.22 (19)	H15B—C15—H15C	109.5
C4—C3—H3	119.4	H16A—C16—H16B	109.5
C4—C5—H5	119.7	H16A—C16—H16C	109.5
C5—C4—N1	123.56 (17)	H16B—C16—H16C	109.5
C5—C6—H6	120.3		

Br1—C1—C2—C3	178.2 (2)	C6—C1—C2—C3	−1.4 (4)
Br1—C1—C6—C5	−177.04 (17)	C7—C8—C9—C10	−179.05 (17)
O1—C12—C11—O2	3.6 (3)	C7—C8—C13—C12	179.05 (18)
O1—C12—C11—C10	−179.35 (19)	C8—C7—N1—C4	179.19 (18)
O1—C12—C13—C8	−179.36 (19)	C9—C10—O3—C16	−5.2 (3)
O2—C11—C10—O3	−4.2 (3)	C9—C8—C13—C12	−1.5 (3)
O2—C11—C10—C9	175.96 (17)	C11—C10—O3—C16	174.9 (2)
O3—C10—C9—C8	179.93 (18)	C11—C12—C13—C8	0.2 (3)
N1—C4—C3—C2	−178.3 (2)	C11—C10—C9—C8	−0.2 (3)
N1—C4—C5—C6	179.6 (2)	C12—C11—C10—O3	178.83 (19)
N1—C7—C8—C13	178.51 (19)	C12—C11—C10—C9	−1.0 (3)
N1—C7—C8—C9	−0.9 (3)	C13—C8—C9—C10	1.5 (3)
C2—C1—C6—C5	2.6 (3)	C13—C12—C11—O2	−175.99 (19)
C3—C4—N1—C7	144.8 (2)	C13—C12—C11—C10	1.0 (3)
C3—C4—C5—C6	−3.0 (3)	C14—O1—C12—C11	−179.1 (2)
C4—C3—C2—C1	−2.0 (4)	C14—O1—C12—C13	0.5 (3)
C4—C5—C6—C1	−0.3 (4)	C15—O2—C11—C10	88.3 (2)
C5—C4—C3—C2	4.2 (4)	C15—O2—C11—C12	−94.7 (2)
C5—C4—N1—C7	−37.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O1^{i}	0.93	2.63	3.272 (2)	127
C7—H7···O2^{i}	0.93	2.63	3.553 (3)	172

Symmetry code: (i) −*x*, *y*+1/2, −*z*+1.