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3-Methoxy-2-[(E)-(4-methoxyphenyl)-iminomethyl]phenol

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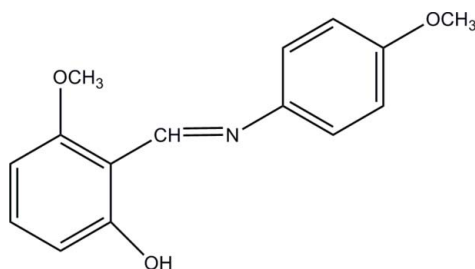
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.053; wR factor = 0.122; data-to-parameter ratio = 15.0.

The title compound, $\text{C}_{15}\text{H}_{15}\text{NO}_3$, adopts the enol-imine tautomeric form. The two rings are twisted with respect to each other, making a dihedral angle of $44.08(5)^\circ$. The 3-methoxy-2-[(E)-(4-methoxyphenyl)-iminomethyl]phenol unit is almost planar, the largest deviation from the mean plane being $0.047(2)$ Å. Such a planar conformation might be related to the occurrence of an intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond. In the crystal, intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into sheets parallel to (010). These sheets are interconnected by weak $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For background to the properties and uses of Schiff bases, see: Barton & Ollis (1979); Layer (1963); Ingold (1969); Cohen *et al.* (1964); Taggi *et al.* (2002). For hydrogen-bond motifs, see: Etter *et al.* (1990); Bernstein *et al.* (1995). For related structures, see: Özdemir Tari *et al.* (2010); Şahin *et al.* (2005).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{15}\text{NO}_3$
 $M_r = 257.28$
Monoclinic, $P2_1/c$

$a = 14.2658(8)$ Å
 $b = 14.1553(11)$ Å
 $c = 6.5893(17)$ Å

$\beta = 96.398(9)^\circ$
 $V = 1322.3(4)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.65 \times 0.32 \times 0.14$ mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration
(*X-RED32*; Stoe, 2002)
 $T_{\min} = 0.991$, $T_{\max} = 0.997$

7244 measured reflections
2585 independent reflections
1622 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.122$
 $S = 1.04$
2585 reflections

172 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.11$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C1–C6 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C13}-\text{H13}\cdots\text{O2}^{\text{i}}$	0.93	2.60	3.428 (3)	149
$\text{C14}-\text{H14A}\cdots\text{O1}^{\text{ii}}$	0.96	2.49	3.412 (3)	162
$\text{O2}-\text{H2A}\cdots\text{N1}$	0.82	1.87	2.590 (2)	146
$\text{C5}-\text{H5}\cdots\text{Cg1}^{\text{iii}}$	0.93	2.80	3.486 (2)	132

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

Data collection: *X-AREA* (Stoe, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-III* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2646).

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supporting information

Acta Cryst. (2011). E67, o343–o344 [doi:10.1107/S1600536811000596]

3-Methoxy-2-[(*E*)-(4-methoxyphenyl)iminomethyl]phenol

Gonca Özdemir Tari, Şamil Işık, Ramazan Özkan and Ayşen Alaman Ağar

S1. Comment

Schiff bases are used as starting materials in the synthesis of important drugs, such as antibiotics and antiallergic, antiphlogistic, and antitumor substances (Barton *et al.*, 1979; Layer, 1963; Ingold 1969). On the industrial scale, they have a wide range of applications, such as dyes and pigments (Taggi *et al.*, 2002). There are two characteristic properties of Schiff bases, *viz.* Photochromism and thermochromism (Cohen *et al.*, 1964). In general, Schiff bases display two possible tautomeric forms, the phenol-imine (OH) and the keto-amine (NH) forms. Depending on the tautomers, two types of intramolecular hydrogen bonds are observed in Schiff bases: O—H \cdots N in phenol-imine (Özdemir Tari *et al.*, 2010) and N—H \cdots O in keto-amine tautomers (Şahin *et al.*, 2005). Another form of the Schiff base compounds is also known as zwitterion having an ionic intramolecular hydrogen bond (N⁺—H \cdots O⁻) and this form is rarely seen in the solid state (Özdemir Tari *et al.*, 2010).

The title compound, C₁₅H₁₅O₃N₁, adopts the enol-imine tautomeric form. The C7=N1 [1.278 (3) Å] and C8=N1 [1.419 (2) Å] bond distances are of double-bond character, whereas, C6—O2 [1.356 (2) Å] distance is single bond. These distances are similar to that reported in the literature [1.269 (8) Å] and [1.397 (7) Å] for C=N and [1.332 (8) Å] for C—O respectively (Özdemir Tari *et al.*, 2010).

The two phenyl rings are twisted with respect to each other making dihedral angle of 44.08 (5)° (Fig. 1). The 4-methoxyphenylimino)phenol moiety is planar with the largest deviation from the mean plane being 0.047 (2)Å at C7. Such planar conformation might be related to the occurrence of the O—H \cdots N intramolecular hydrogen bond (Fig. 1, Table 1). This intramolecular N—H \cdots O hydrogen bond results in the formation of an S(6) ring (Etter *et al.*, 1990; Bernstein *et al.*, 1995).

Intermolecular C—H \cdots O hydrogen bonds link the molecules forming sheets parallel to the (0 1 0) plane (Fig. 2, Table 1). These sheets are interconnected by weak C—H \cdots π interactions (Table 1, Cg1 is the centroid of the C1—C6 phenyl ring).

S2. Experimental

(*E*)-3-methoxy-2-((4-methoxyphenylimino)methyl)phenol was prepared by refluxing a mixture of a solution containing 2-hydroxy-6-methoxybenzaldehyde (15.2 mg, 0.1 mmol) in ethanol (30 ml) and a solution containing 4-methoxyaniline (12.3 mg, 0.1 mmol) in ethanol (20 ml). The reaction mixture was stirred for 2 h under reflux. Single crystals of the title compound for *x*-ray analysis were obtained by slow evaporation of an ethanol solution (yield 72%; m.p 346–348 K).

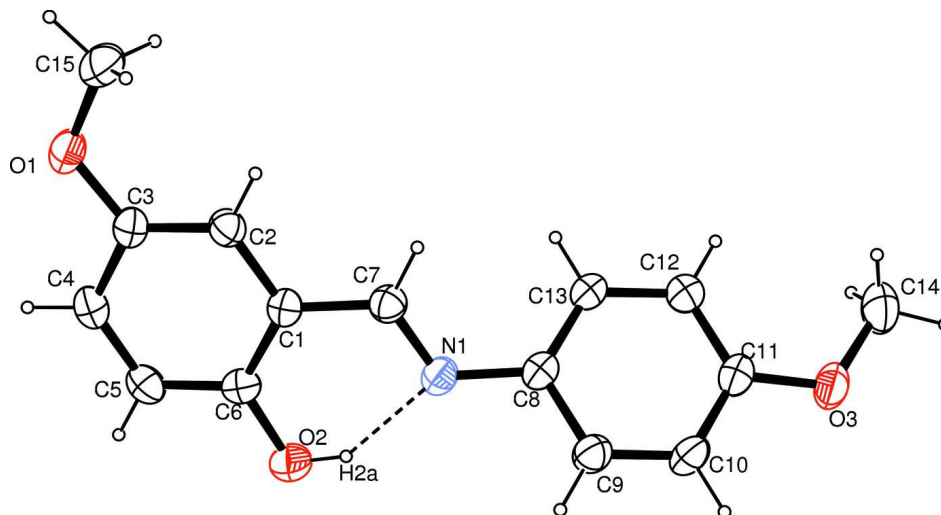


Figure 1

The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii. Intramolecular H bond is shown as dashed line.

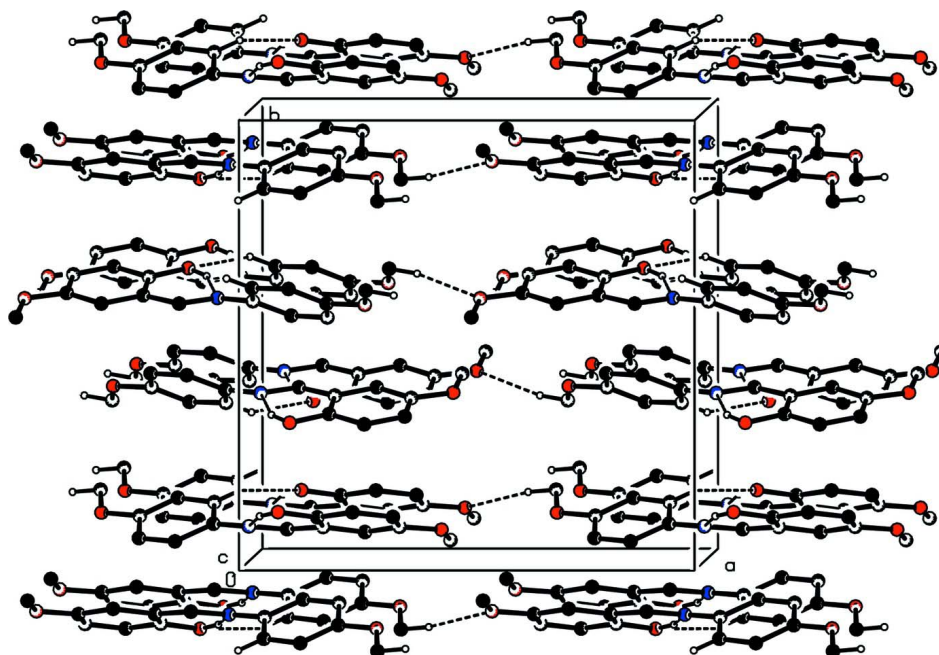


Figure 2

The crystal packing of the title compound showing the formation of sheets parallel to the (0 1 0) plane. H bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

3-Methoxy-2-[(*E*)-(4-methoxyphenyl)iminomethyl]phenol

Crystal data

$C_{15}H_{15}NO_3$
 $M_r = 257.28$

Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

$a = 14.2658 (8) \text{ \AA}$
 $b = 14.1553 (11) \text{ \AA}$
 $c = 6.5893 (17) \text{ \AA}$
 $\beta = 96.398 (9)^\circ$
 $V = 1322.3 (4) \text{ \AA}^3$
 $Z = 4$
 $F(000) = 544$
 $D_x = 1.292 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 12079 reflections
 $\theta = 1.4\text{--}27.2^\circ$
 $\mu = 0.09 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Prism, brown
 $0.65 \times 0.32 \times 0.14 \text{ mm}$

Data collection

Stoe IPDS 2
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: 6.67 pixels mm^{-1}
 φ scan rotation method
 Absorption correction: integration
 (*X-RED32*; Stoe, 2002)
 $T_{\min} = 0.991$, $T_{\max} = 0.997$

7244 measured reflections
 2585 independent reflections
 1622 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$
 $\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 1.4^\circ$
 $h = -16 \rightarrow 17$
 $k = -17 \rightarrow 17$
 $l = -8 \rightarrow 6$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.122$
 $S = 1.04$
 2585 reflections
 172 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
 $w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.1038P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.11 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e \AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.20529 (13)	0.11619 (13)	0.4013 (3)	0.0443 (5)
C2	0.29338 (14)	0.10131 (14)	0.3295 (3)	0.0483 (5)
H2	0.2959	0.0793	0.1972	0.058*
C3	0.37566 (14)	0.11898 (15)	0.4526 (3)	0.0516 (5)
C4	0.37125 (16)	0.15062 (15)	0.6515 (3)	0.0548 (6)
H4	0.4268	0.1626	0.7353	0.066*
C5	0.28635 (16)	0.16440 (15)	0.7254 (3)	0.0542 (6)
H5	0.2848	0.1854	0.8588	0.065*

C6	0.20294 (15)	0.14735 (14)	0.6034 (3)	0.0479 (5)
C7	0.11923 (14)	0.10451 (13)	0.2639 (3)	0.0481 (5)
H7	0.1229	0.0900	0.1274	0.058*
C8	-0.04619 (13)	0.11307 (14)	0.1930 (3)	0.0459 (5)
C9	-0.12676 (15)	0.07751 (15)	0.2668 (4)	0.0544 (6)
H9	-0.1226	0.0522	0.3977	0.065*
C10	-0.21198 (15)	0.07926 (15)	0.1495 (4)	0.0553 (6)
H10	-0.2648	0.0535	0.1995	0.066*
C11	-0.22005 (14)	0.11941 (14)	-0.0446 (4)	0.0512 (5)
C12	-0.14058 (15)	0.15559 (16)	-0.1200 (4)	0.0561 (6)
H12	-0.1452	0.1823	-0.2497	0.067*
C13	-0.05435 (15)	0.15172 (15)	-0.0012 (3)	0.0537 (6)
H13	-0.0010	0.1755	-0.0526	0.064*
C14	-0.3216 (2)	0.1629 (2)	-0.3428 (5)	0.0908 (9)
H14A	-0.3868	0.1590	-0.3970	0.136*
H14B	-0.2837	0.1297	-0.4310	0.136*
H14C	-0.3026	0.2280	-0.3338	0.136*
C15	0.47450 (18)	0.0772 (2)	0.1985 (4)	0.0881 (9)
H15A	0.5402	0.0742	0.1795	0.132*
H15B	0.4427	0.1196	0.1000	0.132*
H15C	0.4472	0.0153	0.1810	0.132*
N1	0.03849 (12)	0.11390 (12)	0.3283 (3)	0.0496 (4)
O1	0.46496 (10)	0.10987 (13)	0.3953 (3)	0.0752 (5)
O2	0.12069 (11)	0.16290 (11)	0.6830 (2)	0.0636 (5)
H2A	0.0763	0.1500	0.5975	0.095*
O3	-0.30930 (10)	0.12168 (12)	-0.1453 (3)	0.0662 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0417 (11)	0.0426 (10)	0.0480 (12)	-0.0004 (9)	0.0020 (9)	-0.0004 (9)
C2	0.0481 (12)	0.0525 (12)	0.0439 (12)	0.0043 (9)	0.0032 (10)	-0.0031 (9)
C3	0.0418 (11)	0.0593 (12)	0.0524 (13)	0.0040 (10)	0.0000 (10)	0.0011 (10)
C4	0.0537 (14)	0.0603 (13)	0.0475 (13)	-0.0011 (10)	-0.0073 (11)	-0.0002 (10)
C5	0.0630 (15)	0.0575 (13)	0.0412 (12)	0.0010 (10)	0.0017 (11)	-0.0042 (10)
C6	0.0494 (13)	0.0472 (11)	0.0487 (13)	0.0010 (9)	0.0117 (10)	0.0013 (9)
C7	0.0468 (12)	0.0484 (11)	0.0494 (12)	0.0000 (9)	0.0059 (10)	-0.0025 (9)
C8	0.0401 (11)	0.0430 (11)	0.0551 (13)	-0.0016 (9)	0.0075 (10)	-0.0027 (9)
C9	0.0480 (13)	0.0570 (13)	0.0586 (14)	-0.0036 (10)	0.0080 (11)	0.0085 (11)
C10	0.0406 (12)	0.0562 (13)	0.0706 (15)	-0.0078 (10)	0.0127 (11)	0.0063 (11)
C11	0.0404 (11)	0.0493 (11)	0.0632 (14)	-0.0021 (9)	0.0025 (10)	-0.0071 (11)
C12	0.0519 (13)	0.0645 (14)	0.0516 (13)	-0.0045 (10)	0.0046 (11)	0.0036 (10)
C13	0.0402 (12)	0.0667 (13)	0.0548 (14)	-0.0061 (10)	0.0086 (10)	0.0042 (11)
C14	0.0623 (17)	0.125 (3)	0.079 (2)	-0.0102 (16)	-0.0186 (15)	0.0173 (18)
C15	0.0519 (15)	0.142 (3)	0.0722 (18)	0.0053 (15)	0.0146 (14)	-0.0252 (18)
N1	0.0409 (10)	0.0518 (10)	0.0561 (11)	-0.0028 (8)	0.0057 (8)	0.0023 (8)
O1	0.0402 (9)	0.1206 (15)	0.0637 (11)	0.0052 (9)	0.0009 (8)	-0.0167 (10)
O2	0.0557 (9)	0.0812 (11)	0.0560 (10)	0.0037 (8)	0.0157 (8)	-0.0081 (8)

O3	0.0417 (9)	0.0795 (11)	0.0749 (11)	-0.0074 (7)	-0.0055 (8)	0.0016 (9)
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Geometric parameters (Å, °)

C1—C6	1.407 (3)	C9—H9	0.9300
C1—C2	1.407 (3)	C10—C11	1.393 (3)
C1—C7	1.451 (3)	C10—H10	0.9300
C2—C3	1.373 (3)	C11—O3	1.369 (2)
C2—H2	0.9300	C11—C12	1.386 (3)
C3—O1	1.375 (3)	C12—C13	1.384 (3)
C3—C4	1.393 (3)	C12—H12	0.9300
C4—C5	1.369 (3)	C13—H13	0.9300
C4—H4	0.9300	C14—O3	1.419 (3)
C5—C6	1.381 (3)	C14—H14A	0.9600
C5—H5	0.9300	C14—H14B	0.9600
C6—O2	1.356 (2)	C14—H14C	0.9600
C7—N1	1.278 (3)	C15—O1	1.397 (3)
C7—H7	0.9300	C15—H15A	0.9600
C8—C13	1.385 (3)	C15—H15B	0.9600
C8—C9	1.391 (3)	C15—H15C	0.9600
C8—N1	1.419 (2)	O2—H2A	0.8200
C9—C10	1.366 (3)		
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C6—C1—C2	118.79 (19)	C9—C10—H10	119.9
C6—C1—C7	121.21 (19)	C11—C10—H10	119.9
C2—C1—C7	119.91 (19)	O3—C11—C12	124.9 (2)
C3—C2—C1	120.7 (2)	O3—C11—C10	115.58 (18)
C3—C2—H2	119.6	C12—C11—C10	119.5 (2)
C1—C2—H2	119.6	C13—C12—C11	119.6 (2)
C2—C3—O1	125.3 (2)	C13—C12—H12	120.2
C2—C3—C4	119.3 (2)	C11—C12—H12	120.2
O1—C3—C4	115.40 (19)	C12—C13—C8	121.1 (2)
C5—C4—C3	121.0 (2)	C12—C13—H13	119.5
C5—C4—H4	119.5	C8—C13—H13	119.5
C3—C4—H4	119.5	O3—C14—H14A	109.5
C4—C5—C6	120.4 (2)	O3—C14—H14B	109.5
C4—C5—H5	119.8	H14A—C14—H14B	109.5
C6—C5—H5	119.8	O3—C14—H14C	109.5
O2—C6—C5	118.21 (18)	H14A—C14—H14C	109.5
O2—C6—C1	122.03 (19)	H14B—C14—H14C	109.5
C5—C6—C1	119.75 (19)	O1—C15—H15A	109.5
N1—C7—C1	120.8 (2)	O1—C15—H15B	109.5
N1—C7—H7	119.6	H15A—C15—H15B	109.5
C1—C7—H7	119.6	O1—C15—H15C	109.5
C13—C8—C9	118.6 (2)	H15A—C15—H15C	109.5
C13—C8—N1	123.71 (18)	H15B—C15—H15C	109.5
C9—C8—N1	117.54 (19)	C7—N1—C8	121.78 (19)
C10—C9—C8	120.9 (2)	C3—O1—C15	118.36 (18)

C10—C9—H9	119.6	C6—O2—H2A	109.5
C8—C9—H9	119.6	C11—O3—C14	117.85 (19)
C9—C10—C11	120.29 (19)		
C6—C1—C2—C3	-1.5 (3)	N1—C8—C9—C10	176.32 (19)
C7—C1—C2—C3	175.00 (19)	C8—C9—C10—C11	-1.9 (3)
C1—C2—C3—O1	-177.7 (2)	C9—C10—C11—O3	-176.91 (19)
C1—C2—C3—C4	1.0 (3)	C9—C10—C11—C12	1.5 (3)
C2—C3—C4—C5	-0.1 (3)	O3—C11—C12—C13	178.0 (2)
O1—C3—C4—C5	178.7 (2)	C10—C11—C12—C13	-0.3 (3)
C3—C4—C5—C6	-0.2 (3)	C11—C12—C13—C8	-0.6 (3)
C4—C5—C6—O2	-179.4 (2)	C9—C8—C13—C12	0.3 (3)
C4—C5—C6—C1	-0.4 (3)	N1—C8—C13—C12	-174.7 (2)
C2—C1—C6—O2	-179.81 (18)	C1—C7—N1—C8	172.99 (18)
C7—C1—C6—O2	3.7 (3)	C13—C8—N1—C7	-36.1 (3)
C2—C1—C6—C5	1.2 (3)	C9—C8—N1—C7	148.9 (2)
C7—C1—C6—C5	-175.24 (19)	C2—C3—O1—C15	-2.2 (4)
C6—C1—C7—N1	-6.4 (3)	C4—C3—O1—C15	179.1 (2)
C2—C1—C7—N1	177.11 (18)	C12—C11—O3—C14	0.9 (3)
C13—C8—C9—C10	1.0 (3)	C10—C11—O3—C14	179.2 (2)

Hydrogen-bond geometry (Å, °)

*Cg*1 is the centroid of the C1—C6 ring.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C13—H13...O2 ⁱ	0.93	2.60	3.428 (3)	149
C14—H14 <i>A</i> ...O1 ⁱⁱ	0.96	2.49	3.412 (3)	162
O2—H2 <i>A</i> ...N1	0.82	1.87	2.590 (2)	146
C5—H5... <i>Cg</i> 1 ⁱⁱⁱ	0.93	2.80	3.486 (2)	132

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