Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(3-Chloro-1,4-dioxo-1,4-dihydronaphthalen-2-yl)-*N*-propionylpropionamide

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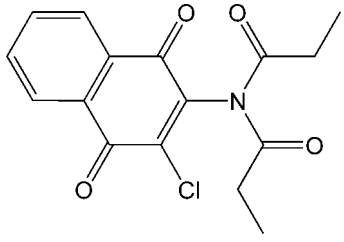
Received 4 December 2013; accepted 20 December 2013

Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.115; data-to-parameter ratio = 14.5.

In the title molecule, $C_{16}H_{14}ClNO_4$, the four essentially planar atoms of the imide group [r.m.s. deviation = 0.0286 (11) Å] form a dihedral angle of 77.36 (13)° with the naphthoquinone group [maximun deviation = 0.111 (2) Å for the carbonyl O atom in the naphthalene 1-position] and the two imide carbonyl groups are oriented *anti* with respect to each other. In the crystal, molecules are connected by weak C—H···O hydrogen bonds, as well as π–π stacking interactions [centroid–centroid distance = 3.888 (3) Å], forming a three-dimensional network.

Related literature

For the synthesis and biological evaluation of imido-substituted 1,4-naphthoquinone derivatives, see: Bakare *et al.* (2003); Berhe *et al.* (2008); Brandy *et al.* (2013). For the anticancer and antitrypanosomal activity of related compounds, see: Bakare *et al.* (2003); Berhe *et al.* (2008); Khraiwesh *et al.* (2012). For a related structure, see: Butcher *et al.* (2013).



Experimental

Crystal data

$C_{16}H_{14}ClNO_4$
M_r = 319.73
Triclinic, $P\overline{1}$
a = 8.1362 (9) Å
b = 8.2254 (9) Å
c = 12.4471 (11) Å
α = 98.105 (8)°
β = 92.297 (8)°
γ = 116.821 (11)°
V = 730.88 (15) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 2.48 mm^{-1}
T = 123 K
0.48 × 0.34 × 0.08 mm

Data collection

Agilent Xcalibur (Ruby, Gemini) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.396, T_{max} = 1.000
4648 measured reflections
2908 independent reflections
2419 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.115
S = 1.01
2908 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 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
C5—H5A···O3^{i}	0.95	2.59	3.294 (2)	131
C15—H15A···O1^{ii}	0.99	2.55	3.442 (2)	150
C16—H16B···O4^{iii}	0.98	2.54	3.425 (3)	150
C16—H16C···O3^{iv}	0.98	2.60	3.482 (3)	150

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

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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*.

This work was supported in part by grant No. 5-U54—CA914–31 (Howard University/Johns Hopkins Cancer Center Partnership), in part by grant G12MD007597 from the National Institute On Minority Health and Health Disparities of the National Institutes of Health, and in part by MRI grant No. CHE-1126533 from the NSF for the purchase of a GC-Mass spectrometer. RJB wishes to acknowledge the NSF–MRI program (grant CHE-0619278) for funds to purchase the diffractometer as well as the Howard University Nanoscience Facility for access to liquid nitrogen.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5677).

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supporting information

Acta Cryst. (2014). E**70**, o102 [doi:10.1107/S1600536813034302]

N-(3-Chloro-1,4-dioxo-1,4-dihydronaphthalen-2-yl)-*N*-propionylpropionamide

Nabil Idris, Ray J. Butcher and Oladapo Bakare

S1. Comment

Our group are involved in the synthesis and biological evaluation of some imido-substituted 1,4-naphthoquinone derivatives (Bakare *et al.*, 2003; Berhe *et al.*, 2008; Brandy *et al.*, 2013), and have previously reported that 2-chloro-3-dipropionylamino-1,4-naphthoquinone and some of its analogs possess inhibitory activities against certain protein kinases (Bakare *et al.*, 2003). This class of compounds have also been shown to possess anticancer (Bakare *et al.*, 2003; Berhe *et al.*, 2008) and anti-trypanosomal activities (Khraiwesh, *et al.*, 2012). As part of our studies (Butcher *et al.*, 2013) on the synthesis, properties, and structural characterization of this class of compounds, we herein present, the crystal structure of the title compound.

In the title molecule (Fig. 1), the naphthoquinone moiety deviates from planarity. The outer ring (C3-C8) is essentially planar (r.m.s. 0.004 (1) Å) while the inner ring (C1/C2/C3/C8/C9/C10) deviates slightly from planarity (r.m.s. 0.029 (1) Å) with a maximum deviation of 0.0437 (13) Å for C9. The imide group (N1/C14/O3/O4) is almost planar (r.m.s. 0.0286 (11) and the dihedral angle between this group and the whole naphthoquinone group (C1-C10/O1/O2) is 77.36 (13)°, with the two imide carbonyls oriented *anti* with respect to each other. In the crystal, molecules are linked by weak C—H···O hydrogen bonds as well as π—π interactions between the naphthoquinone rings with a centroid to centroid distance of 3.888 (3) Å between C1/C2/C3/C8/C9/C10 and C3/C4/C5/C6/C7/C8 in symmetry related rings (-*x*, 1 - *y*, 1 - *z*) forming a three-dimensional network (Fig. 2).

S2. Experimental

The title compound was synthesized by refluxing 2-amino-3-chloro-1,4-naphthoquinone in propionyl chloride as previously reported (Bakare *et al.* (2003)). The crude compound thus obtained was crystallized from ethanol to obtain yellow crystals suitable for X-ray studies.

S3. Refinement

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with a C—H distances of 0.93 and 0.97 Å U_{iso}(H) = 1.2U_{eq}(C) and 0.96 Å for CH_3 [U_{iso}(H) = 1.5U_{eq}(C)].

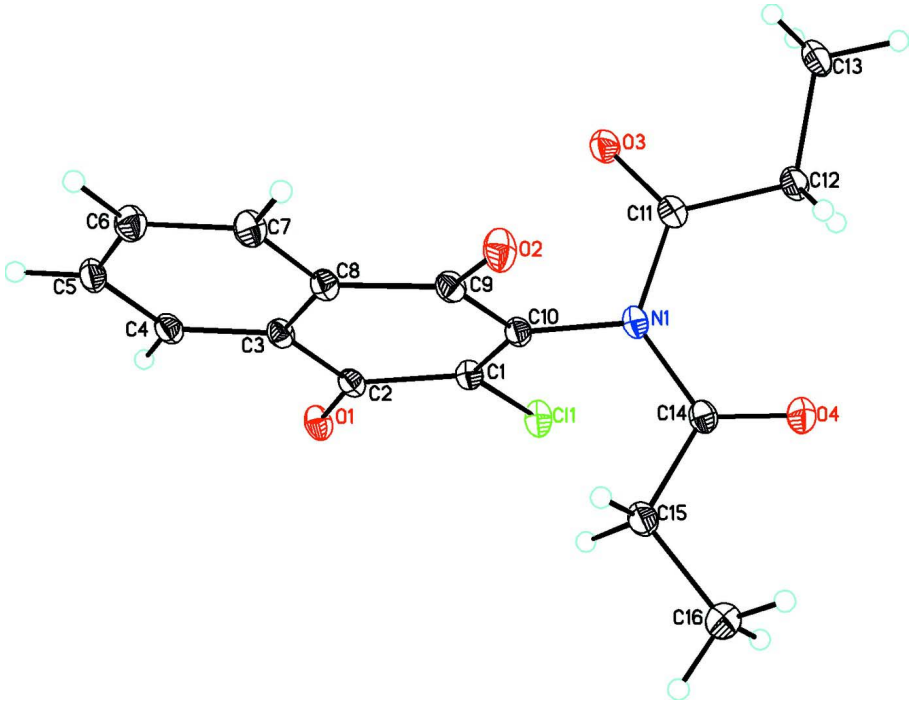

Figure 1

The molecular structure of the title compound with displacement parameters shown at the 30% probability level.

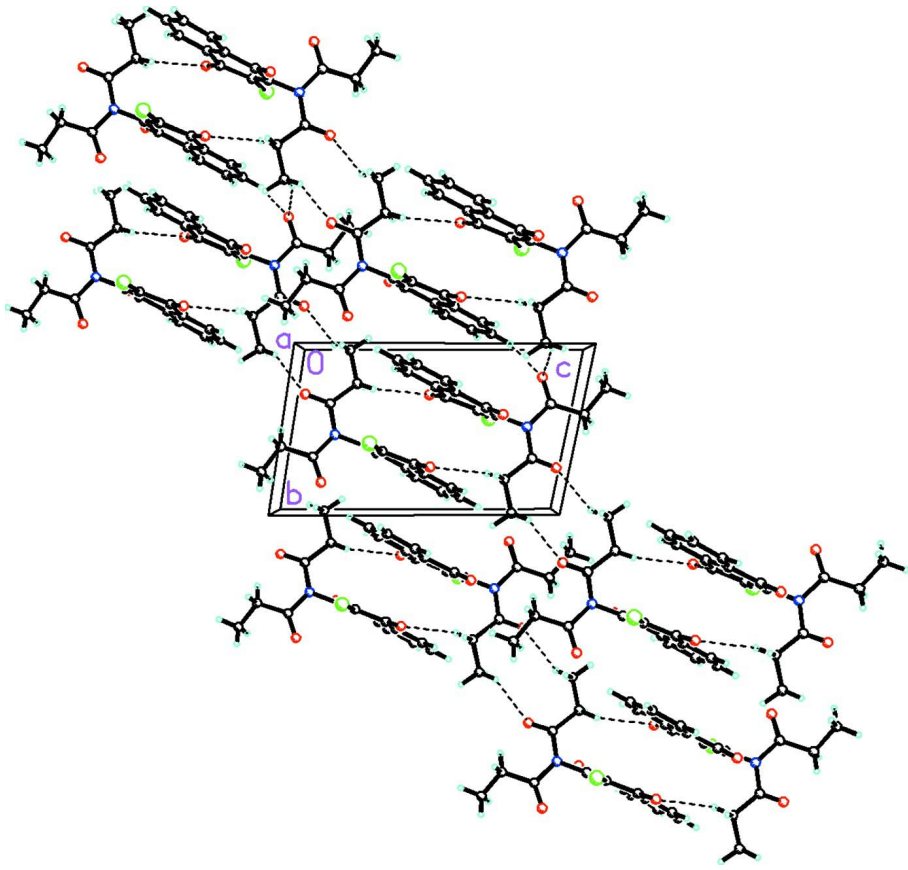

Figure 2

The crystal packing viewed along the *a* axis showing the weak C—H···O hydrogen bonds (as dashed lines) as well as the π–π stacking along the *b* axis.

N-(3-Chloro-1,4-dioxo-1,4-dihydronaphthalen-2-yl)-N-propionylpropionamide

Crystal data

$C_{16}H_{14}ClNO_4$
$M_r = 319.73$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.1362\ (9)$ Å
$b = 8.2254\ (9)$ Å
$c = 12.4471\ (11)$ Å
$\alpha = 98.105\ (8)°$
$\beta = 92.297\ (8)°$
$\gamma = 116.821\ (11)°$
$V = 730.88\ (15)$ Å^3
$Z = 2$
$F(000) = 332$
$D_x = 1.453$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 1754 reflections
$\theta = 3.6–75.0°$
$\mu = 2.48$ mm^{-1}
$T = 123$ K
Plate, colorless
0.48 × 0.34 × 0.08 mm

Data collection

Agilent Xcalibur (Ruby, Gemini) diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 10.5081 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
$T_{min} = 0.396$, $T_{max} = 1.000$
4648 measured reflections
2908 independent reflections
2419 reflections with $I > 2\sigma(I)$

R_{int} = 0.030
θ_{max} = 75.2°, θ_{min} = 3.6°
h = −10→7
k = −8→10
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.115
S = 1.01
2908 reflections
201 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
$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 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}
Cl1	0.34840 (7)	0.42387 (7)	0.70172 (4)	0.02795 (15)
O1	0.3577 (2)	0.2807 (2)	0.47615 (11)	0.0272 (3)
O2	0.9546 (2)	0.4037 (2)	0.73820 (12)	0.0297 (3)
O3	0.6017 (2)	0.1818 (2)	0.85542 (12)	0.0298 (3)
O4	0.7362 (2)	0.7230 (2)	0.96429 (12)	0.0313 (3)
N1	0.6911 (2)	0.4761 (2)	0.83472 (13)	0.0217 (3)
C1	0.5193 (3)	0.3762 (3)	0.65414 (16)	0.0210 (4)
C2	0.4900 (3)	0.2978 (3)	0.53419 (15)	0.0212 (4)
C3	0.6266 (3)	0.2389 (3)	0.49340 (15)	0.0208 (4)
C4	0.5978 (3)	0.1483 (3)	0.38574 (15)	0.0236 (4)
H4A	0.4934	0.1283	0.3385	0.028*
C5	0.7225 (3)	0.0869 (3)	0.34745 (16)	0.0254 (4)
H5A	0.7026	0.0243	0.2742	0.031*
C6	0.8766 (3)	0.1177 (3)	0.41683 (17)	0.0264 (4)
H6A	0.9610	0.0751	0.3907	0.032*
C7	0.9074 (3)	0.2096 (3)	0.52347 (16)	0.0253 (4)
H7A	1.0137	0.2320	0.5699	0.030*
C8	0.7822 (3)	0.2692 (3)	0.56247 (15)	0.0211 (4)
C9	0.8146 (3)	0.3624 (3)	0.67823 (15)	0.0215 (4)
C10	0.6669 (3)	0.4052 (3)	0.72075 (15)	0.0205 (4)
C11	0.6645 (3)	0.3405 (3)	0.90111 (15)	0.0227 (4)
C12	0.7214 (3)	0.4027 (3)	1.02245 (15)	0.0255 (4)

H12A	0.6392	0.4507	1.0547	0.031*
H12B	0.8498	0.5049	1.0359	0.031*
C13	0.7112 (4)	0.2455 (3)	1.07811 (17)	0.0386 (6)
H13A	0.7554	0.2926	1.1560	0.058*
H13B	0.7892	0.1949	1.0446	0.058*
H13C	0.5825	0.1479	1.0695	0.058*
C14	0.7498 (3)	0.6667 (3)	0.87202 (15)	0.0223 (4)
C15	0.8322 (3)	0.7895 (3)	0.78881 (16)	0.0268 (4)
H15A	0.7356	0.7554	0.7270	0.032*
H15B	0.9332	0.7671	0.7600	0.032*
C16	0.9083 (4)	0.9933 (3)	0.83514 (19)	0.0380 (5)
H16A	0.9591	1.0659	0.7777	0.057*
H16B	1.0067	1.0290	0.8950	0.057*
H16C	0.8086	1.0170	0.8627	0.057*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0231 (2)	0.0398 (3)	0.0226 (2)	0.0178 (2)	0.00071 (17)	−0.00015 (18)
O1	0.0246 (7)	0.0381 (8)	0.0195 (7)	0.0158 (7)	−0.0031 (5)	0.0034 (6)
O2	0.0244 (7)	0.0406 (9)	0.0231 (7)	0.0168 (7)	−0.0043 (6)	−0.0023 (6)
O3	0.0371 (8)	0.0241 (7)	0.0218 (7)	0.0096 (6)	−0.0017 (6)	0.0026 (6)
O4	0.0405 (9)	0.0310 (8)	0.0207 (7)	0.0158 (7)	0.0053 (6)	0.0020 (6)
N1	0.0220 (8)	0.0260 (9)	0.0156 (7)	0.0105 (7)	−0.0002 (6)	0.0018 (6)
C1	0.0184 (9)	0.0242 (9)	0.0206 (9)	0.0103 (8)	0.0026 (7)	0.0032 (7)
C2	0.0210 (9)	0.0223 (9)	0.0174 (9)	0.0072 (8)	0.0009 (7)	0.0054 (7)
C3	0.0199 (9)	0.0210 (9)	0.0182 (9)	0.0063 (7)	0.0028 (7)	0.0049 (7)
C4	0.0265 (10)	0.0235 (10)	0.0173 (9)	0.0085 (8)	0.0001 (7)	0.0045 (7)
C5	0.0317 (11)	0.0244 (10)	0.0165 (9)	0.0101 (9)	0.0040 (8)	0.0021 (7)
C6	0.0262 (10)	0.0295 (10)	0.0249 (10)	0.0134 (9)	0.0066 (8)	0.0054 (8)
C7	0.0222 (9)	0.0289 (10)	0.0229 (10)	0.0103 (8)	0.0011 (7)	0.0038 (8)
C8	0.0207 (9)	0.0223 (9)	0.0182 (9)	0.0080 (8)	0.0015 (7)	0.0037 (7)
C9	0.0198 (9)	0.0231 (9)	0.0197 (9)	0.0084 (8)	−0.0003 (7)	0.0042 (7)
C10	0.0219 (9)	0.0203 (9)	0.0168 (9)	0.0078 (8)	0.0016 (7)	0.0030 (7)
C11	0.0206 (9)	0.0267 (10)	0.0200 (9)	0.0099 (8)	0.0025 (7)	0.0053 (8)
C12	0.0280 (10)	0.0265 (10)	0.0180 (9)	0.0091 (8)	0.0009 (8)	0.0045 (7)
C13	0.0584 (15)	0.0346 (12)	0.0186 (10)	0.0182 (11)	−0.0043 (10)	0.0059 (8)
C14	0.0199 (9)	0.0270 (10)	0.0190 (9)	0.0104 (8)	−0.0003 (7)	0.0030 (7)
C15	0.0297 (10)	0.0287 (11)	0.0206 (9)	0.0122 (9)	0.0022 (8)	0.0048 (8)
C16	0.0511 (14)	0.0274 (12)	0.0303 (11)	0.0141 (11)	0.0047 (10)	0.0038 (9)

Geometric parameters (Å, º)

Cl1—C1	1.7117 (19)	C7—C8	1.392 (3)
O1—C2	1.214 (2)	C7—H7A	0.9500
O2—C9	1.217 (2)	C8—C9	1.486 (3)
O3—C11	1.206 (3)	C9—C10	1.492 (3)
O4—C14	1.205 (2)	C11—C12	1.507 (3)

N1—C14	1.416 (3)	C12—C13	1.523 (3)
N1—C11	1.425 (2)	C12—H12A	0.9900
N1—C10	1.425 (2)	C12—H12B	0.9900
C1—C10	1.339 (3)	C13—H13A	0.9800
C1—C2	1.504 (3)	C13—H13B	0.9800
C2—C3	1.480 (3)	C13—H13C	0.9800
C3—C4	1.394 (3)	C14—C15	1.510 (3)
C3—C8	1.404 (3)	C15—C16	1.512 (3)
C4—C5	1.394 (3)	C15—H15A	0.9900
C4—H4A	0.9500	C15—H15B	0.9900
C5—C6	1.395 (3)	C16—H16A	0.9800
C5—H5A	0.9500	C16—H16B	0.9800
C6—C7	1.384 (3)	C16—H16C	0.9800
C6—H6A	0.9500		

C14—N1—C11	126.40 (16)	N1—C10—C9	116.65 (16)
C14—N1—C10	120.34 (16)	O3—C11—N1	117.25 (17)
C11—N1—C10	113.08 (16)	O3—C11—C12	123.88 (18)
C10—C1—C2	123.02 (17)	N1—C11—C12	118.84 (17)
C10—C1—Cl1	121.46 (15)	C11—C12—C13	111.82 (17)
C2—C1—Cl1	115.52 (14)	C11—C12—H12A	109.3
O1—C2—C3	122.90 (17)	C13—C12—H12A	109.3
O1—C2—C1	120.67 (17)	C11—C12—H12B	109.3
C3—C2—C1	116.41 (16)	C13—C12—H12B	109.3
C4—C3—C8	119.68 (18)	H12A—C12—H12B	107.9
C4—C3—C2	119.45 (17)	C12—C13—H13A	109.5
C8—C3—C2	120.85 (17)	C12—C13—H13B	109.5
C3—C4—C5	119.94 (18)	H13A—C13—H13B	109.5
C3—C4—H4A	120.0	C12—C13—H13C	109.5
C5—C4—H4A	120.0	H13A—C13—H13C	109.5
C4—C5—C6	119.90 (18)	H13B—C13—H13C	109.5
C4—C5—H5A	120.1	O4—C14—N1	121.10 (18)
C6—C5—H5A	120.1	O4—C14—C15	123.97 (18)
C7—C6—C5	120.52 (18)	N1—C14—C15	114.92 (16)
C7—C6—H6A	119.7	C14—C15—C16	113.04 (17)
C5—C6—H6A	119.7	C14—C15—H15A	109.0
C6—C7—C8	119.80 (18)	C16—C15—H15A	109.0
C6—C7—H7A	120.1	C14—C15—H15B	109.0
C8—C7—H7A	120.1	C16—C15—H15B	109.0
C7—C8—C3	120.15 (18)	H15A—C15—H15B	107.8
C7—C8—C9	118.99 (17)	C15—C16—H16A	109.5
C3—C8—C9	120.86 (17)	C15—C16—H16B	109.5
O2—C9—C8	122.72 (18)	H16A—C16—H16B	109.5
O2—C9—C10	119.76 (18)	C15—C16—H16C	109.5
C8—C9—C10	117.51 (16)	H16A—C16—H16C	109.5
C1—C10—N1	122.45 (17)	H16B—C16—H16C	109.5
C1—C10—C9	120.90 (17)		

C10—C1—C2—O1	−177.09 (19)	Cl1—C1—C10—N1	0.3 (3)
Cl1—C1—C2—O1	3.8 (3)	C2—C1—C10—C9	0.8 (3)
C10—C1—C2—C3	4.7 (3)	Cl1—C1—C10—C9	179.78 (14)
Cl1—C1—C2—C3	−174.35 (14)	C14—N1—C10—C1	−76.6 (2)
O1—C2—C3—C4	−4.5 (3)	C11—N1—C10—C1	107.9 (2)
C1—C2—C3—C4	173.66 (17)	C14—N1—C10—C9	103.9 (2)
O1—C2—C3—C8	177.06 (19)	C11—N1—C10—C9	−71.6 (2)
C1—C2—C3—C8	−4.8 (3)	O2—C9—C10—C1	174.07 (19)
C8—C3—C4—C5	0.5 (3)	C8—C9—C10—C1	−6.1 (3)
C2—C3—C4—C5	−177.96 (17)	O2—C9—C10—N1	−6.5 (3)
C3—C4—C5—C6	−0.5 (3)	C8—C9—C10—N1	173.41 (16)
C4—C5—C6—C7	−0.3 (3)	C14—N1—C11—O3	175.03 (18)
C5—C6—C7—C8	1.1 (3)	C10—N1—C11—O3	−9.8 (2)
C6—C7—C8—C3	−1.0 (3)	C14—N1—C11—C12	−6.9 (3)
C6—C7—C8—C9	178.09 (18)	C10—N1—C11—C12	168.24 (17)
C4—C3—C8—C7	0.2 (3)	O3—C11—C12—C13	6.7 (3)
C2—C3—C8—C7	178.70 (18)	N1—C11—C12—C13	−171.24 (19)
C4—C3—C8—C9	−178.88 (17)	C11—N1—C14—O4	−19.9 (3)
C2—C3—C8—C9	−0.4 (3)	C10—N1—C14—O4	165.21 (18)
C7—C8—C9—O2	6.6 (3)	C11—N1—C14—C15	158.81 (18)
C3—C8—C9—O2	−174.25 (19)	C10—N1—C14—C15	−16.0 (2)
C7—C8—C9—C10	−173.25 (17)	O4—C14—C15—C16	3.9 (3)
C3—C8—C9—C10	5.9 (3)	N1—C14—C15—C16	−174.85 (18)
C2—C1—C10—N1	−178.69 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*A*···O3^{i}	0.95	2.59	3.294 (2)	131
C15—H15*A*···O1^{ii}	0.99	2.55	3.442 (2)	150
C16—H16*B*···O4^{iii}	0.98	2.54	3.425 (3)	150
C16—H16*C*···O3^{iv}	0.98	2.60	3.482 (3)	150

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