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5,6-Dichloro-2-(2-fluorophenyl)isoindoline-1,3-dione

Orhan Büyükgüngör[a]* and Mustafa Odabaşoğlu[b]

[a]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139 Kurupelit Samsun, Turkey, and [b]Department of Chemistry, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139 Kurupelit Samsun, Turkey
Correspondence e-mail: orhanb@omu.edu.tr

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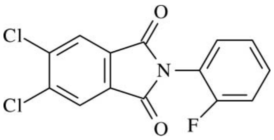
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.032; wR factor = 0.085; data-to-parameter ratio = 12.4.

The crystal structure of the title compound, $C_{14}H_6Cl_2FNO_2$, exhibits C—H···π and π–π interactions, which generate C(3) chains in the [100] direction. The π–π interaction occurs between the aromatic rings of isoindoline units, with a centroid–centroid distance of 3.672 Å and an interplanar separation of 3.528 Å. The isoindoline unit is planar and inclined at an angle of 58.63 (18)° to the substituent benzene ring. The F atom is disordered over two positions, with refined occupancies of 0.669 (3) and 0.331 (3).

Related literature

For general background, see: Hall *et al.* (1987); Abdel-Hafez (2004); Sena *et al.* (2007). For related literature, see: Loudon (2002).



Experimental

Crystal data

$C_{14}H_6Cl_2FNO_2$
M_r = 310.11
Orthorhombic, $Pbca$
a = 8.0078 (3) Å
b = 27.3570 (9) Å
c = 11.5563 (5) Å
V = 2531.63 (17) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.52 mm^{-1}
T = 296 K
0.76 × 0.50 × 0.20 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.703, T_{max} = 0.907
31181 measured reflections
2384 independent reflections
1956 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.085
S = 1.03
2384 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···$Cg1^i$	0.93	2.99	3.844 (4)	153

Symmetry code: (i) $x + \frac{1}{2}, y, -z + \frac{3}{2}$. $Cg1$ is the centroid of atoms C2–C7.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2693).

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supporting information

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5,6-Dichloro-2-(2-fluorophenyl)isoindoline-1,3-dione

Orhan Büyükgüngör and Mustafa Odabaşoğlu

S1. Comment

Phthalimide derivatives have various biological activities (Hall *et al.*, 1987; 1987; Abdel-Hafez, 2004; Sena *et al.* 2007). In view of the importance of the *N*-arylphthalimides, we herein report the results of title compound 5,6-dichloro-2-(2-fluorophenyl)isoindoline-1,3-dione, (I).

The molecule of (I) is built up from a 5,6-dichlorophthalimide unit connected to a *o*-fluorophenyl group through an nitrogen atom (Fig. 1). The isoindoline ring (atoms N1/C1–C8) is almost planar the largest deviation from the mean plane being 0.027 (2) Å for atom C1. The dihedral angle between the fluorophenyl ring and the mean plane of the isoindoline part is 58.63 (18)°. In (I), the crystal packing is stabilized by C6—H6···π (Table 1) interactions. The C1—N1 and C8—N1 bonds are 1.406 (2) and 1.394 (2)Å, respectively. These C—N bond lengths are shorter than C—N single bond (C—N = 1.47 Å; Loudon, 2002). This reflects both the sp^2 hybridization of the adjacent carbon and the overlap of unshared electrons on nitrogen with π-electron system of carbonyl groups (Fig. 3). The π–π interaction occurs between the aromatic rings (C2–C7) of isoindoline moieties at (x; y; z) and ($1 - x$; $1 - y$; $1 - z$) sites with the centroid-centroid distance of 3.672 Å and an interplanar separation of 3.528 Å.

S2. Experimental

A mixture of 4,5-dichlorophthalic acid (1.175 g, 0.005 mol) and 2-fluoroaniline (0.56 g, 0.005 mol) in DMF (1.5 ml) was heated at boiling temperature 15 min. The reaction mixture added in 50 ml e thanol (95%) and crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of this mixture at room temperature (yield 80%).

S3. Refinement

The F atom is disordered over two *ortho* positions with refined occupancies of 0.669 (3) and 0.331 (3). H atoms were positioned geometrically, with C—H = 0.93 Å for aromatic H, and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C)$.

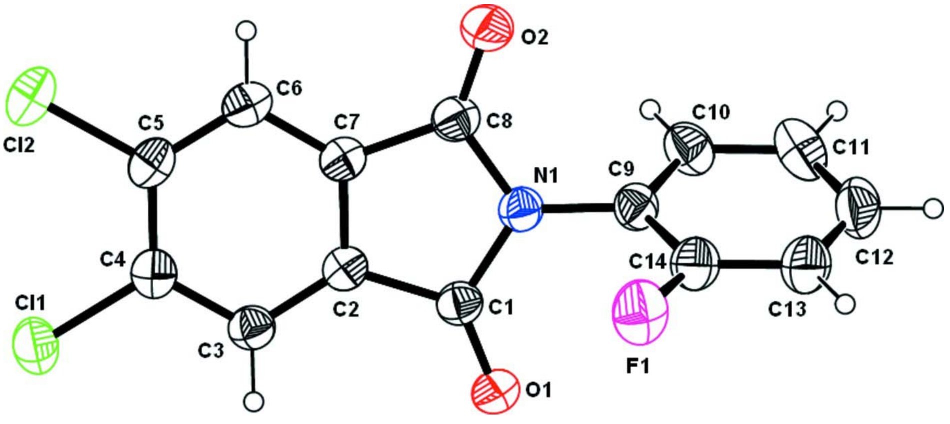

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Only the higher occupied of the disordered sites is shown.

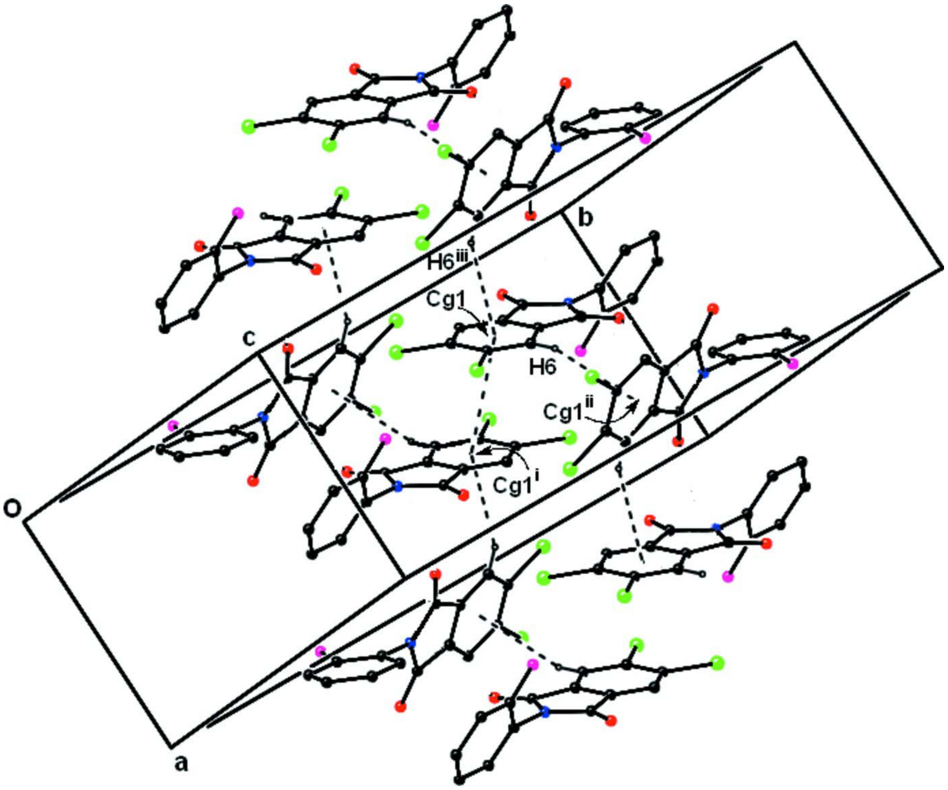

Figure 2

A partial packing diagram of (I), showing the formation of C(3) chain and π–π interactions. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) 1 - x,1 - y,1 - z; (ii) 1/2 + x, y, 3/2 - z; (iii) x - 1/2, y, 3/2 - z].

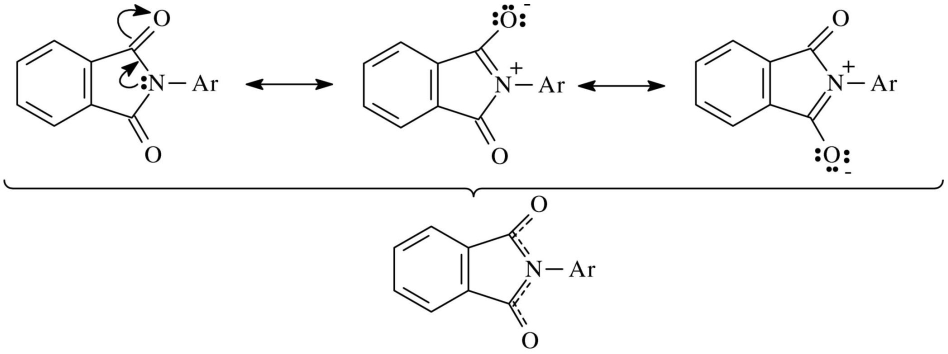

Figure 3

The *sp*² hybridization of the adjacent carbon and the overlap of unshared electrons on nitrogen with π-electron system of carbonyl groups.

5,6-Dichloro-2-(2-fluorophenyl)isoindoline-1,3-dione

Crystal data

$C_{14}H_6Cl_2FNO_2$
$M_r = 310.11$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 8.0078$ (3) Å
$b = 27.3570$ (9) Å
$c = 11.5563$ (5) Å
$V = 2531.63$ (17) Å^3
$Z = 8$
$F(000) = 1248$
$D_x = 1.622$ Mg m^{-3}
Mo *Kα* radiation, $\lambda = 0.71073$ Å
Cell parameters from 31181 reflections
$\theta = 1.5$–$26.2°$
$\mu = 0.52$ mm^{-1}
$T = 296$ K
Prism, colorless
0.76 × 0.50 × 0.20 mm

Data collection

Stoe IPDSII diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
ω scan rotation method
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.703$, $T_{max} = 0.907$
31181 measured reflections
2384 independent reflections
1956 reflections with $I > 2\sigma(I)$
$R_{int} = 0.063$
$\theta_{max} = 25.6°$, $\theta_{min} = 1.5°$
$h = -9 \rightarrow 9$
$k = -33 \rightarrow 33$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.085$
$S = 1.03$
2384 reflections
192 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
$w = 1/[\sigma^2(F_o^2) + (0.0471P)^2 + 0.3058P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0107 (9)

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	$U_{\rm iso}$*/$U_{\rm eq}$	Occ. (<1)
C1	0.28581 (19)	0.61572 (6)	0.53527 (14)	0.0525 (4)	
C2	0.30531 (19)	0.56310 (6)	0.56377 (13)	0.0503 (4)	
C3	0.2424 (2)	0.52248 (6)	0.50902 (14)	0.0548 (4)	
H3	0.1761	0.5254	0.4433	0.066*	
C4	0.2809 (2)	0.47693 (6)	0.55490 (15)	0.0554 (4)	
C5	0.3825 (2)	0.47286 (6)	0.65266 (15)	0.0578 (4)	
C6	0.4455 (2)	0.51427 (7)	0.70638 (15)	0.0585 (4)	
H6	0.5133	0.5118	0.7715	0.070*	
C7	0.40453 (19)	0.55905 (6)	0.66043 (14)	0.0516 (4)	
C8	0.4518 (2)	0.60897 (7)	0.69910 (14)	0.0569 (4)	
C9	0.3879 (2)	0.69277 (6)	0.62605 (15)	0.0557 (4)	
C10	0.3223 (3)	0.71816 (7)	0.71829 (17)	0.0702 (5)	
C11	0.3314 (3)	0.76849 (8)	0.7209 (2)	0.0850 (6)	
H11	0.2870	0.7857	0.7831	0.102*	
C12	0.4067 (3)	0.79318 (8)	0.6309 (2)	0.0867 (7)	
H12	0.4126	0.8271	0.6326	0.104*	
C13	0.4725 (3)	0.76831 (8)	0.5395 (2)	0.0867 (7)	
H13	0.5230	0.7851	0.4790	0.104*	
C14	0.4637 (2)	0.71824 (7)	0.53769 (17)	0.0687 (5)	
N1	0.37841 (17)	0.64082 (5)	0.61972 (11)	0.0540 (3)	
O1	0.20978 (16)	0.63434 (4)	0.45785 (10)	0.0670 (3)	
O2	0.53535 (19)	0.62065 (5)	0.78115 (12)	0.0813 (4)	
Cl1	0.20110 (7)	0.425557 (17)	0.48901 (4)	0.07471 (19)	
Cl2	0.43176 (8)	0.416515 (19)	0.70844 (5)	0.0847 (2)	
F1	0.5355 (3)	0.69169 (7)	0.45401 (16)	0.0918 (7)	0.669 (3)
F2	0.2785 (5)	0.68818 (15)	0.8105 (3)	0.0846 (14)	0.331 (3)
H10	0.2721	0.7019	0.7812	0.102*	0.669 (3)
H14	0.5077	0.7027	0.4724	0.102*	0.331 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0502 (9)	0.0555 (9)	0.0519 (8)	0.0034 (7)	−0.0010 (7)	0.0013 (7)
C2	0.0466 (8)	0.0536 (9)	0.0508 (8)	0.0033 (7)	0.0030 (7)	0.0024 (7)
C3	0.0548 (9)	0.0580 (9)	0.0517 (9)	0.0044 (7)	−0.0005 (7)	0.0001 (7)

C4	0.0535 (9)	0.0547 (9)	0.0579 (9)	0.0009 (7)	0.0110 (8)	−0.0015 (7)
C5	0.0549 (9)	0.0571 (10)	0.0613 (9)	0.0070 (7)	0.0107 (8)	0.0102 (8)
C6	0.0530 (9)	0.0664 (11)	0.0559 (9)	0.0022 (8)	−0.0015 (7)	0.0100 (8)
C7	0.0451 (8)	0.0571 (9)	0.0527 (8)	0.0007 (7)	0.0012 (7)	0.0051 (7)
C8	0.0490 (9)	0.0633 (11)	0.0583 (9)	−0.0074 (8)	−0.0035 (8)	0.0073 (8)
C9	0.0495 (9)	0.0536 (9)	0.0642 (10)	−0.0071 (7)	−0.0033 (8)	0.0033 (8)
C10	0.0716 (12)	0.0697 (12)	0.0694 (12)	−0.0167 (10)	0.0057 (10)	−0.0074 (9)
C11	0.0756 (14)	0.0754 (13)	0.1040 (17)	−0.0095 (11)	0.0001 (12)	−0.0284 (12)
C12	0.0862 (15)	0.0553 (11)	0.1186 (19)	−0.0149 (10)	−0.0175 (14)	0.0007 (12)
C13	0.0981 (17)	0.0699 (13)	0.0921 (15)	−0.0261 (12)	−0.0062 (13)	0.0163 (11)
C14	0.0676 (12)	0.0668 (11)	0.0717 (11)	−0.0108 (9)	0.0053 (10)	0.0052 (9)
N1	0.0530 (8)	0.0533 (7)	0.0556 (7)	−0.0044 (6)	−0.0038 (6)	0.0048 (6)
O1	0.0792 (9)	0.0590 (7)	0.0629 (7)	0.0081 (6)	−0.0172 (6)	0.0035 (5)
O2	0.0848 (10)	0.0764 (9)	0.0826 (9)	−0.0197 (7)	−0.0339 (8)	0.0115 (7)
Cl1	0.0859 (4)	0.0561 (3)	0.0822 (3)	−0.0074 (2)	0.0026 (3)	−0.0062 (2)
Cl2	0.0989 (4)	0.0610 (3)	0.0943 (4)	0.0118 (3)	−0.0031 (3)	0.0214 (2)
F1	0.1037 (15)	0.0859 (13)	0.0857 (12)	−0.0125 (10)	0.0384 (11)	0.0034 (10)
F2	0.086 (3)	0.095 (3)	0.073 (2)	−0.018 (2)	0.0152 (18)	−0.0176 (18)

Geometric parameters (Å, º)

C1—O1	1.1961 (19)	C9—C10	1.376 (3)
C1—N1	1.405 (2)	C9—C14	1.377 (2)
C1—C2	1.485 (2)	C9—N1	1.425 (2)
C2—C3	1.374 (2)	C10—C11	1.379 (3)
C2—C7	1.375 (2)	C10—F2	1.390 (4)
C3—C4	1.389 (2)	C10—H10	0.942 (2)
C3—H3	0.9300	C11—C12	1.379 (3)
C4—C5	1.396 (3)	C11—H11	0.9300
C4—Cl1	1.7213 (17)	C12—C13	1.362 (3)
C5—C6	1.387 (3)	C12—H12	0.9300
C5—Cl2	1.7169 (17)	C13—C14	1.372 (3)
C6—C7	1.375 (2)	C13—H13	0.9300
C6—H6	0.9300	C14—F1	1.339 (3)
C7—C8	1.486 (2)	C14—H14	0.935 (2)
C8—O2	1.204 (2)	F1—H14	0.4311 (19)
C8—N1	1.395 (2)	F2—H10	0.509 (4)
O1—C1—N1	125.46 (15)	C10—C9—N1	121.51 (15)
O1—C1—C2	129.22 (15)	C14—C9—N1	119.33 (16)
N1—C1—C2	105.32 (13)	C9—C10—C11	120.04 (19)
C3—C2—C7	121.37 (15)	C9—C10—F2	113.1 (2)
C3—C2—C1	130.02 (15)	C11—C10—F2	125.8 (2)
C7—C2—C1	108.60 (14)	C9—C10—H10	121.51 (19)
C2—C3—C4	117.92 (16)	C11—C10—H10	118.4 (2)
C2—C3—H3	121.0	C10—C11—C12	119.7 (2)
C4—C3—H3	121.0	C10—C11—H11	120.1
C3—C4—C5	120.63 (16)	C12—C11—H11	120.1

C3—C4—Cl1	118.77 (14)	C13—C12—C11	120.6 (2)
C5—C4—Cl1	120.61 (13)	C13—C12—H12	119.7
C6—C5—C4	120.61 (15)	C11—C12—H12	119.7
C6—C5—Cl2	118.79 (14)	C12—C13—C14	119.4 (2)
C4—C5—Cl2	120.60 (14)	C12—C13—H13	120.3
C7—C6—C5	117.91 (16)	C14—C13—H13	120.3
C7—C6—H6	121.0	F1—C14—C13	122.1 (2)
C5—C6—H6	121.0	F1—C14—C9	116.75 (17)
C6—C7—C2	121.55 (16)	C13—C14—C9	121.1 (2)
C6—C7—C8	129.94 (15)	C13—C14—H14	116.5 (2)
C2—C7—C8	108.50 (14)	C9—C14—H14	122.33 (19)
O2—C8—N1	125.90 (17)	C8—N1—C1	111.95 (13)
O2—C8—C7	128.49 (16)	C8—N1—C9	124.52 (14)
N1—C8—C7	105.61 (14)	C1—N1—C9	123.45 (14)
C10—C9—C14	119.16 (17)		

O1—C1—C2—C3	0.3 (3)	N1—C9—C10—C11	−179.04 (18)
N1—C1—C2—C3	−179.36 (16)	C14—C9—C10—F2	−168.5 (2)
O1—C1—C2—C7	179.79 (17)	N1—C9—C10—F2	11.8 (3)
N1—C1—C2—C7	0.13 (17)	C9—C10—C11—C12	−0.1 (3)
C7—C2—C3—C4	0.5 (2)	F2—C10—C11—C12	167.6 (3)
C1—C2—C3—C4	179.97 (15)	C10—C11—C12—C13	−0.2 (4)
C2—C3—C4—C5	−0.9 (2)	C11—C12—C13—C14	−0.1 (4)
C2—C3—C4—Cl1	179.09 (12)	C12—C13—C14—F1	−175.6 (2)
C3—C4—C5—C6	0.6 (3)	C12—C13—C14—C9	0.7 (3)
Cl1—C4—C5—C6	−179.39 (13)	C10—C9—C14—F1	175.55 (19)
C3—C4—C5—Cl2	−179.33 (13)	N1—C9—C14—F1	−4.7 (3)
Cl1—C4—C5—Cl2	0.7 (2)	C10—C9—C14—C13	−1.0 (3)
C4—C5—C6—C7	0.1 (2)	N1—C9—C14—C13	178.76 (19)
Cl2—C5—C6—C7	−179.98 (12)	O2—C8—N1—C1	−178.75 (17)
C5—C6—C7—C2	−0.5 (2)	C7—C8—N1—C1	1.21 (18)
C5—C6—C7—C8	179.55 (16)	O2—C8—N1—C9	−1.9 (3)
C3—C2—C7—C6	0.2 (2)	C7—C8—N1—C9	178.08 (14)
C1—C2—C7—C6	−179.39 (14)	O1—C1—N1—C8	179.45 (16)
C3—C2—C7—C8	−179.87 (15)	C2—C1—N1—C8	−0.86 (17)
C1—C2—C7—C8	0.59 (18)	O1—C1—N1—C9	2.5 (3)
C6—C7—C8—O2	−1.2 (3)	C2—C1—N1—C9	−177.77 (14)
C2—C7—C8—O2	178.87 (17)	C10—C9—N1—C8	−62.0 (2)
C6—C7—C8—N1	178.88 (16)	C14—C9—N1—C8	118.29 (19)
C2—C7—C8—N1	−1.10 (18)	C10—C9—N1—C1	114.5 (2)
C14—C9—C10—C11	0.7 (3)	C14—C9—N1—C1	−65.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···$Cg1^{\mathrm{i}}$	0.93	2.99	3.844 (4)	153

Symmetry code: (i) $x+1/2$, y, $-z+3/2$.