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

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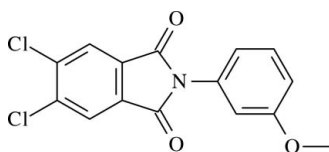
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 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.071; wR factor = 0.170; data-to-parameter ratio = 14.0.

The title compound, $\text{C}_{15}\text{H}_9\text{Cl}_2\text{NO}_3$, crystallizes as an inversion twin, the ratio of the twin components being 0.43 (13):0.57 (13). The isoindoline group is planar and inclined by $77.63(3)^\circ$ to the aromatic ring substituent. The crystal structure is stabilized by aromatic $\pi-\pi$ stacking interactions involving the benzene rings of adjacent isoindoline groups, with a centroid-centroid distance of $3.664(7)$ Å and an interplanar separation of 3.409 Å.

Related literature

For general background, see: Chapman *et al.* (1979); Hall *et al.*, (1983; 1987); Srivastava *et al.* (2001); Abdel-Hafez (2004); Sena *et al.* (2007).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_9\text{Cl}_2\text{NO}_3$
 $M_r = 322.13$

 Orthorhombic, $P2_12_12_1$
 $a = 6.8689(5)$ Å

 $b = 9.7362(10)$ Å
 $c = 20.4271(14)$ Å
 $V = 1366.1(2)$ Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 0.48$ mm⁻¹
 $T = 296$ K
 $0.65 \times 0.51 \times 0.23$ mm

Data collection

 Stoe IPDSII diffractometer
 Absorption correction: integration
 ($X\text{-RED32}$; Stoe & Cie, 2002)
 $T_{\min} = 0.685$, $T_{\max} = 0.888$

 6123 measured reflections
 2687 independent reflections
 2076 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.117$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.071$
 $wR(F^2) = 0.169$
 $S = 1.00$
 2687 reflections
 192 parameters
 H-atom parameters constrained

 $\Delta\rho_{\max} = 0.63$ e Å⁻³
 $\Delta\rho_{\min} = -0.57$ e Å⁻³
 Absolute structure: Flack (1983),
 1114 Friedel pairs
 Flack parameter: 0.43 (13)

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: RZ2205).

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supporting information

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

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S1. Comment

The present work is part of a structural study of derivatives of *N*-arylphthalimides (Abdel-Hafez, 2004; Chapman *et al.*, 1979; Hall *et al.*, 1983; Hall *et al.*, 1987; Sena *et al.*, 2007; Srivastava *et al.*, 2001). We report here the crystal structure of 5,6-dichloro-2-(3-methoxyphenyl)isoindoline-1,3-dione.

The molecule of the title compound consist of a 5,6-dichlorophthalimide unit connected to a *m*-methoxyphenyl group through the nitrogen atom (Fig. 1). The isoindoline ring (atoms N1/C1–C8) is almost planar, the largest deviation from the mean plane being 0.051 (3) Å for atom C5. The dihedral angle between the methoxyphenyl ring and the mean plane of the isoindoline group is 77.63 (3)°. The crystal packing is stabilized by aromatic $\pi\cdots\pi$ stacking interactions (Fig. 2) occurring between the aromatic rings of isoindoline groups at (x, y, z) and $(-1/2+x; 3/2 - y; 1-z)$, with a centroid-centroid distance of 3.664 (7) Å and a plane-plane separations of 3.409 Å.

S2. Experimental

A mixture of 4,5-dichlorophthalic acid (1.175 g, 0.005 mol) and 2-aminophenol (0.545 g, 0.02 mol) in DMF (1.5 ml) was heated at boiling temperature for 15 min, then ethanol (95%, 50 ml) was added. Crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of this mixture at room temperature (yield 80%; mp. 546–548 K).

S3. Refinement

H atoms were positioned geometrically, with C—H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms. The rather high Rint value is ascribed to the poor nature of the available crystals.

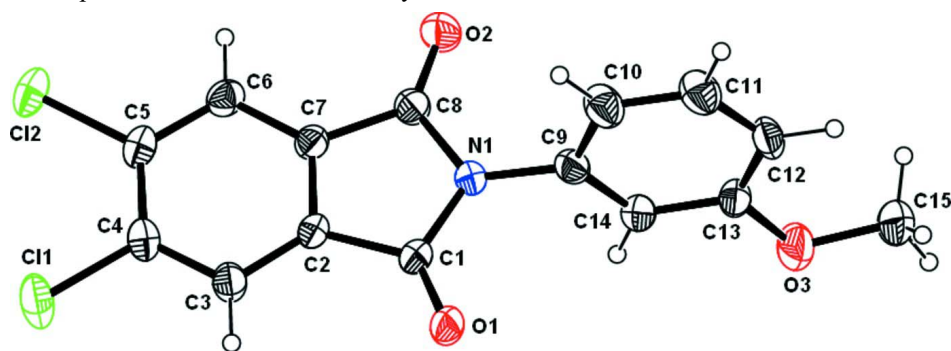
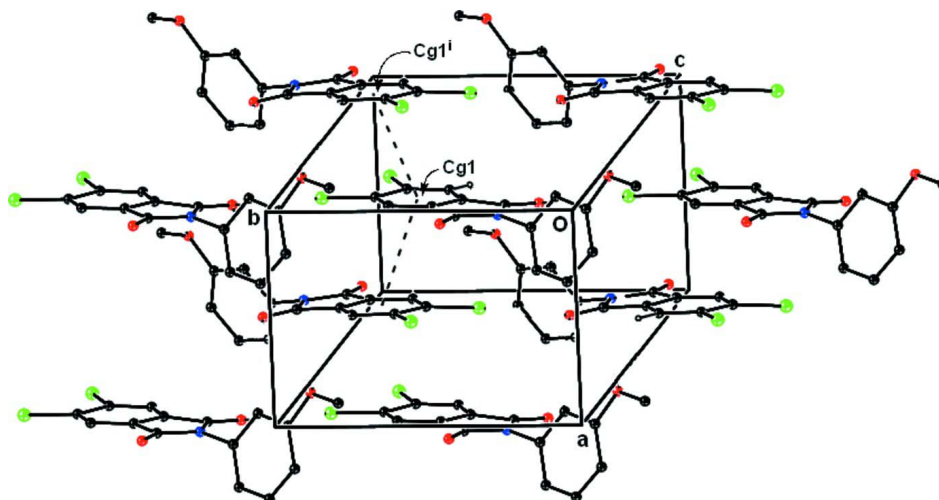


Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

**Figure 2**

A partial packing diagram of the title compound, showing the formation of $\pi\cdots\pi$ stacking interactions. H atoms are omitted for clarity. Cg1 and Cg1¹ are the centroids of the benzene rings of the isoindoline groups. [Symmetry code: (i) $-1/2+x, 3/2-y, 1-z$].

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

Crystal data

$C_{15}H_9Cl_2NO_3$

$M_r = 322.13$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 6.8689$ (5) Å

$b = 9.7362$ (10) Å

$c = 20.4271$ (14) Å

$V = 1366.1$ (2) Å³

$Z = 4$

$F(000) = 656$

$D_x = 1.566$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 6123 reflections

$\theta = 2.0$ – 27.3°

$\mu = 0.48$ mm⁻¹

$T = 296$ K

Prism, colourless

$0.65 \times 0.51 \times 0.23$ 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)

$T_{\min} = 0.685$, $T_{\max} = 0.888$

6123 measured reflections

2687 independent reflections

2076 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.117$

$\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 2.0^\circ$

$h = -8 \rightarrow 8$

$k = -12 \rightarrow 10$

$l = -23 \rightarrow 25$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.071$

$wR(F^2) = 0.169$

$S = 1.01$

2687 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

$$w = 1/[\sigma^2(F_o^2) + (0.1011P)^2]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.63 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.57 \text{ e } \text{\AA}^{-3}$$

Extinction correction: *SHELXL97* (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.054 (7)

Absolute structure: Flack (1983), 1114 Friedel pairs

Absolute structure parameter: 0.43 (13)

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2767 (6)	0.3976 (4)	0.4834 (2)	0.0408 (9)
C2	0.2607 (6)	0.5458 (4)	0.5011 (2)	0.0380 (8)
C3	0.2552 (7)	0.6055 (4)	0.5626 (2)	0.0474 (9)
H3	0.2551	0.5526	0.6005	0.057*
C4	0.2499 (6)	0.7459 (4)	0.5652 (2)	0.0458 (9)
C5	0.2520 (6)	0.8278 (4)	0.5080 (2)	0.0444 (9)
C6	0.2545 (7)	0.7642 (4)	0.4471 (2)	0.0463 (9)
H6	0.2538	0.8157	0.4088	0.056*
C7	0.2579 (6)	0.6225 (4)	0.44509 (19)	0.0395 (8)
C8	0.2677 (6)	0.5285 (4)	0.3883 (2)	0.0438 (9)
C9	0.3137 (6)	0.2765 (5)	0.3758 (2)	0.0436 (10)
C10	0.4978 (6)	0.2535 (6)	0.3505 (3)	0.0598 (13)
H10	0.5993	0.3135	0.3599	0.072*
C11	0.5282 (7)	0.1425 (6)	0.3118 (3)	0.0624 (14)
H11	0.6517	0.1277	0.2946	0.075*
C12	0.3806 (7)	0.0500 (5)	0.2972 (2)	0.0527 (12)
H12	0.4041	-0.0263	0.2710	0.063*
C13	0.1967 (7)	0.0752 (5)	0.3229 (2)	0.0477 (11)
C14	0.1630 (6)	0.1861 (4)	0.3621 (2)	0.0466 (10)
H14	0.0397	0.2011	0.3795	0.056*
C15	0.0552 (10)	-0.1131 (6)	0.2632 (3)	0.0743 (16)
H15A	-0.0652	-0.1629	0.2605	0.112*
H15B	0.1584	-0.1749	0.2749	0.112*
H15C	0.0834	-0.0720	0.2216	0.112*
N1	0.2788 (5)	0.3963 (4)	0.41436 (16)	0.0412 (8)
O1	0.2907 (5)	0.2994 (3)	0.51795 (15)	0.0550 (8)
O2	0.2669 (6)	0.5562 (3)	0.33082 (15)	0.0616 (9)
O3	0.0389 (5)	-0.0085 (4)	0.3116 (2)	0.0749 (11)
Cl1	0.2389 (2)	0.82582 (13)	0.64049 (6)	0.0730 (4)

C12	0.25735 (16)	1.00342 (11)	0.51245 (6)	0.0578 (4)
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Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.047 (2)	0.0320 (19)	0.044 (2)	-0.0019 (18)	0.0005 (19)	0.0014 (16)
C2	0.0407 (16)	0.0300 (17)	0.043 (2)	-0.0036 (16)	-0.0006 (19)	-0.0017 (14)
C3	0.058 (2)	0.041 (2)	0.043 (2)	0.004 (2)	-0.004 (2)	-0.0045 (17)
C4	0.0455 (19)	0.044 (2)	0.047 (2)	0.003 (2)	-0.006 (2)	-0.0117 (17)
C5	0.0383 (15)	0.0338 (19)	0.061 (2)	0.0010 (17)	-0.006 (2)	-0.0052 (18)
C6	0.0487 (19)	0.037 (2)	0.053 (2)	0.006 (2)	0.001 (2)	0.0073 (17)
C7	0.0399 (17)	0.0360 (19)	0.043 (2)	-0.0027 (18)	0.001 (2)	0.0000 (15)
C8	0.049 (2)	0.041 (2)	0.042 (2)	0.004 (2)	0.0003 (18)	0.0012 (16)
C9	0.054 (2)	0.043 (2)	0.034 (2)	0.0034 (19)	0.0016 (15)	0.0006 (18)
C10	0.050 (2)	0.072 (3)	0.058 (3)	-0.006 (2)	0.007 (2)	-0.016 (3)
C11	0.057 (3)	0.069 (4)	0.061 (3)	0.007 (3)	0.010 (2)	-0.010 (3)
C12	0.075 (3)	0.043 (3)	0.040 (3)	0.012 (2)	0.002 (2)	-0.011 (2)
C13	0.066 (3)	0.035 (2)	0.042 (2)	-0.0022 (19)	-0.0012 (18)	-0.0046 (18)
C14	0.054 (2)	0.040 (2)	0.046 (2)	0.0013 (19)	0.0058 (18)	-0.001 (2)
C15	0.108 (4)	0.059 (3)	0.055 (3)	-0.021 (3)	-0.001 (3)	-0.013 (3)
N1	0.0523 (19)	0.0362 (17)	0.0351 (18)	-0.0028 (15)	0.0025 (15)	-0.0036 (13)
O1	0.087 (2)	0.0336 (15)	0.0445 (17)	0.0030 (16)	-0.0010 (15)	0.0009 (12)
O2	0.090 (2)	0.0525 (18)	0.0425 (18)	0.004 (2)	0.0015 (18)	0.0047 (14)
O3	0.081 (2)	0.067 (2)	0.076 (3)	-0.026 (2)	0.0145 (19)	-0.027 (2)
Cl1	0.1007 (9)	0.0574 (7)	0.0609 (8)	0.0176 (8)	-0.0121 (8)	-0.0238 (6)
Cl2	0.0536 (5)	0.0314 (5)	0.0883 (8)	0.0032 (5)	-0.0054 (6)	-0.0070 (5)

Geometric parameters (Å, °)

C1—O1	1.192 (5)	C9—C10	1.384 (6)
C1—N1	1.410 (5)	C9—C14	1.387 (6)
C1—C2	1.491 (5)	C9—N1	1.428 (5)
C2—C7	1.367 (5)	C10—C11	1.355 (7)
C2—C3	1.384 (6)	C10—H10	0.9300
C3—C4	1.368 (6)	C11—C12	1.388 (7)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.415 (6)	C12—C13	1.390 (6)
C4—C11	1.725 (4)	C12—H12	0.9300
C5—C6	1.389 (6)	C13—C14	1.364 (6)
C5—C12	1.713 (4)	C13—O3	1.376 (5)
C6—C7	1.381 (6)	C14—H14	0.9300
C6—H6	0.9300	C15—O3	1.424 (6)
C7—C8	1.479 (6)	C15—H15A	0.9600
C8—O2	1.205 (5)	C15—H15B	0.9600
C8—N1	1.395 (5)	C15—H15C	0.9600
O1—C1—N1	125.7 (4)	C14—C9—N1	120.2 (3)
O1—C1—C2	129.6 (4)	C11—C10—C9	119.2 (4)

N1—C1—C2	104.6 (3)	C11—C10—H10	120.4
C7—C2—C3	122.0 (4)	C9—C10—H10	120.4
C7—C2—C1	109.0 (3)	C10—C11—C12	122.0 (4)
C3—C2—C1	129.0 (4)	C10—C11—H11	119.0
C4—C3—C2	117.1 (4)	C12—C11—H11	119.0
C4—C3—H3	121.5	C11—C12—C13	117.9 (4)
C2—C3—H3	121.5	C11—C12—H12	121.1
C3—C4—C5	122.0 (4)	C13—C12—H12	121.1
C3—C4—C11	119.1 (3)	C14—C13—O3	115.7 (4)
C5—C4—C11	118.9 (3)	C14—C13—C12	121.1 (4)
C6—C5—C4	119.2 (4)	O3—C13—C12	123.2 (4)
C6—C5—C12	119.5 (3)	C13—C14—C9	119.6 (4)
C4—C5—C12	121.2 (3)	C13—C14—H14	120.2
C7—C6—C5	118.2 (4)	C9—C14—H14	120.2
C7—C6—H6	120.9	O3—C15—H15A	109.5
C5—C6—H6	120.9	O3—C15—H15B	109.5
C2—C7—C6	121.4 (4)	H15A—C15—H15B	109.5
C2—C7—C8	108.6 (3)	O3—C15—H15C	109.5
C6—C7—C8	130.0 (4)	H15A—C15—H15C	109.5
O2—C8—N1	125.4 (4)	H15B—C15—H15C	109.5
O2—C8—C7	128.7 (4)	C8—N1—C1	111.9 (3)
N1—C8—C7	105.9 (3)	C8—N1—C9	123.5 (3)
C10—C9—C14	120.3 (4)	C1—N1—C9	124.1 (3)
C10—C9—N1	119.5 (4)	C13—O3—C15	118.6 (4)
O1—C1—C2—C7	176.9 (5)	C14—C9—C10—C11	0.5 (8)
N1—C1—C2—C7	-1.1 (5)	N1—C9—C10—C11	-177.4 (4)
O1—C1—C2—C3	-1.1 (8)	C9—C10—C11—C12	-0.6 (9)
N1—C1—C2—C3	-179.1 (4)	C10—C11—C12—C13	0.8 (8)
C7—C2—C3—C4	-1.0 (7)	C11—C12—C13—C14	-0.9 (7)
C1—C2—C3—C4	176.7 (4)	C11—C12—C13—O3	179.7 (5)
C2—C3—C4—C5	-0.6 (8)	O3—C13—C14—C9	-179.7 (4)
C2—C3—C4—C11	178.9 (3)	C12—C13—C14—C9	0.9 (7)
C3—C4—C5—C6	1.7 (7)	C10—C9—C14—C13	-0.7 (7)
C11—C4—C5—C6	-177.9 (4)	N1—C9—C14—C13	177.2 (4)
C3—C4—C5—C12	-176.8 (4)	O2—C8—N1—C1	180.0 (5)
C11—C4—C5—C12	3.6 (5)	C7—C8—N1—C1	0.1 (5)
C4—C5—C6—C7	-1.0 (7)	O2—C8—N1—C9	7.6 (7)
C12—C5—C6—C7	177.4 (3)	C7—C8—N1—C9	-172.3 (4)
C3—C2—C7—C6	1.7 (7)	O1—C1—N1—C8	-177.5 (4)
C1—C2—C7—C6	-176.5 (4)	C2—C1—N1—C8	0.6 (5)
C3—C2—C7—C8	179.3 (4)	O1—C1—N1—C9	-5.2 (7)
C1—C2—C7—C8	1.2 (5)	C2—C1—N1—C9	172.9 (3)
C5—C6—C7—C2	-0.6 (7)	C10—C9—N1—C8	72.8 (6)
C5—C6—C7—C8	-177.7 (4)	C14—C9—N1—C8	-105.2 (5)
C2—C7—C8—O2	179.3 (5)	C10—C9—N1—C1	-98.6 (5)
C6—C7—C8—O2	-3.3 (9)	C14—C9—N1—C1	83.4 (5)
C2—C7—C8—N1	-0.8 (5)	C14—C13—O3—C15	170.2 (5)

C6—C7—C8—N1

176.6 (5)

C12—C13—O3—C15

−10.5 (8)
