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Key indicators

Single-crystal X-ray study
 $T = 273$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.038
 wR factor = 0.118
 Data-to-parameter ratio = 18.1

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

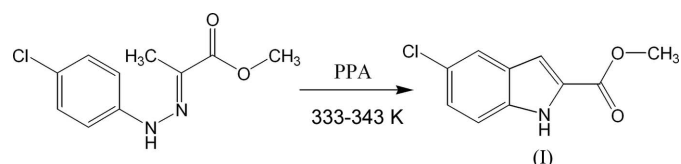
Methyl 5-chloro-1*H*-indole-2-carboxylate

In the molecule of the title compound, $\text{C}_{10}\text{H}_8\text{ClNO}_2$, the indole ring system is planar. In the crystal structure, the molecules are linked by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming centrosymmetric dimers.

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Comment

The synthesis of indole derivatives has long been a topic of fundamental interest to organic and medicinal chemists (Murphy *et al.*, 1997; Cavallini *et al.*, 1958). Indole derivatives are important pharmacologically, possessing anti-allergic (Shigenaga *et al.*, 1993), central-nervous-system depressant (Sen Gupta *et al.*, 1982), muscle relaxant (Butera *et al.*, 2001), and anti-cancer (Al-Soud *et al.*, 2004; Hu *et al.*, 2005; Preobrazhenskaya *et al.*, 1993) properties. The Fischer indole synthesis is the most widely used method for the preparation of indole derivatives (Robinson, 1982; Hughes, 1993). The chemistry of indoles and their synthetic applications have been published (Murakami, 1999). The crystal structure of methyl indole-3-carboxylate has been reported (Hu *et al.*, 2005), as have the structures of some esters of indole-2-carboxylic acid (Chandrakantha *et al.*, 1990, 1992, 1998). In view of the importance of the title compound, (I), its crystal structure is reported here.



In the molecule of the title compound, (I) (Fig. 1), the bond lengths and angles are in normal ranges (Allen *et al.*, 1987; Allen, 2002).

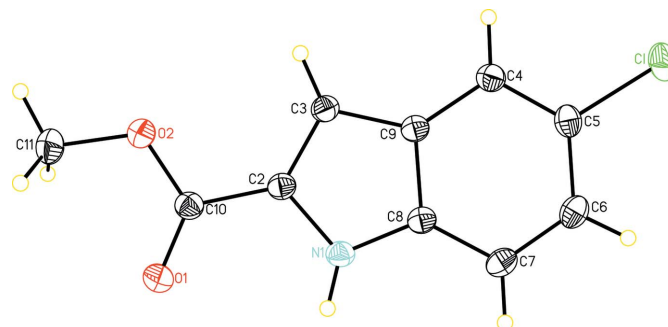


Figure 1
 A drawing of the title molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 20% probability level.

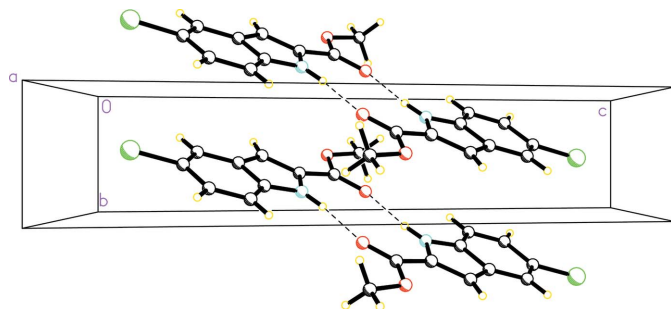


Figure 2

A packing diagram of (I), viewed down the *a* axis. Dashed lines indicate hydrogen bonds.

An examination of the deviations from the least-squares planes through individual rings shows that rings *A* (N1/C2/C3/C8/C9) and *B* (C4–C9) are each planar. The indole ring system is planar, with a puckering amplitude $Q_T = 0.021$ (3) Å (Cremer & Pople, 1975). The dihedral angle between rings *A* and *B* is 0.52 (3)°. The mean plane of the methoxy carbonyl group subtends a dihedral angle of 3.43 (2)° with respect to the indole ring system.

As can be seen from the packing diagram (Fig. 2), intermolecular N–H···O hydrogen bonds (Table 1) link the molecules, forming centrosymmetric dimers. Dipole-dipole and van der Waals interactions are effective in the molecular packing.

Experimental

The title compound was prepared following the reported procedure (Narayana *et al.*, 2005). Methylpyruvate-4-chlorophenylhydrazine (2 g, 8.8 mmol) was taken up in polyphosphoric acid (PPA) (10 ml) and continuously stirred. The reaction mass was slowly heated to 333–343 K and maintained for 4 h. Progress of the reaction was monitored by TLC. The reaction mass was cooled and added to water (100 ml), forming a slurry. The separated solid was filtered and washed with water. The dried crude product was purified with charcoal in ethyl acetate at 323 K, filtered and slowly cooled to room temperature and kept overnight with stirring. After one more recrystallization from ethyl acetate, compound (I) was obtained as colorless crystals from an acetone/toluene (8:2) mixture (yield 65%, m.p. 496 K).

Crystal data

$C_{10}H_8ClNO_2$
 $M_r = 209.62$
 Monoclinic, $P2_1/n$
 $a = 12.786$ (2) Å
 $b = 4.0070$ (7) Å
 $c = 19.040$ (3) Å
 $\beta = 103.932$ (2)°
 $V = 946.7$ (3) Å³

$Z = 4$
 $D_x = 1.471$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.37$ mm⁻¹
 $T = 273$ (2) K
 Needle, colorless
 $0.71 \times 0.24 \times 0.17$ mm

Data collection

Bruker Apex2 CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan SADAABS (Sheldrick, 1996)
 $T_{\min} = 0.778$, $T_{\max} = 0.939$

8678 measured reflections
 2321 independent reflections
 1674 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$
 $\theta_{\text{max}} = 28.2^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.118$
 $S = 1.02$
 2321 reflections
 128 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 0.2468P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

| D—H···A | D—H | H···A | D···A | D—H···A |
|--------------------------|------|-------|-------------|---------|
| N1—H1A···O1 ⁱ | 0.86 | 2.03 | 2.8641 (19) | 164 |

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

H atoms were positioned geometrically, with N—H = 0.86 Å, C—H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C,N})$, where $x = 1.5$ for methyl H, and $x = 1.2$ for all other H atoms.

Data collection: APEX2 (Bruker 2006); cell refinement: APEX2; data reduction: APEX2; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker 2000); software used to prepare material for publication: SHELXTL.

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