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Ethyl 2-(2,3-dioxoindolin-1-yl)acetate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.104
Data-to-parameter ratio = 12.9

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

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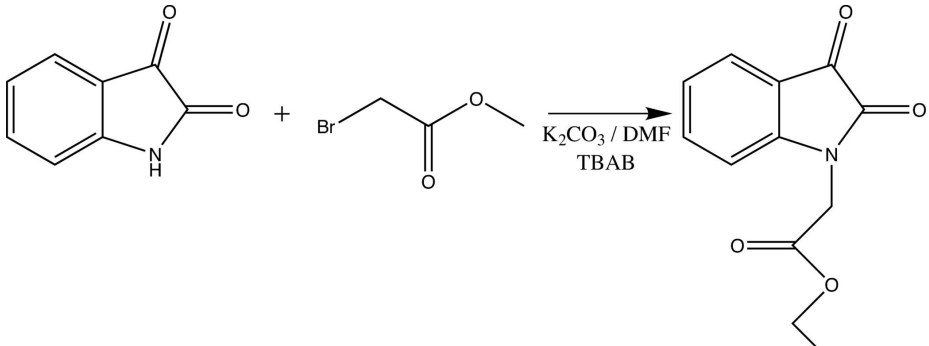
The molecular structure of the title compound, $C_{12}H_{11}NO_4$, at 100 (2) K is characterized by a planar indole ring system, containing a long C—C bond in the 1,2-diketone unit.

Comment

Isatin (1*H*-indole-2,3-dione) is a natural product found in certain plants of the Isatis genus. It is also found in the human body, as a metabolic derivative of adrenalin. Several derivatives of isatin exhibit a wide range of pharmacological activity (Pandeya *et al.*, 2005). Furthermore, it has been shown that isatin and its derivatives are active with respect to the central nervous system (Geronikaki *et al.*, 2004) and have antimicrobial (Raj *et al.*, 2003), anticancerous (Eshba & Salama, 1985) and anti-HIV (Pandeya *et al.*, 1999) properties. Isatin has also proved to be an interesting precursor for the synthesis of new heterocyclic systems endowed with potential biological activity (Bouhfid *et al.*, 2005).



(I)

The synthesis of the title compound, (I), is shown in the scheme above. The 1*H*-indoline ring system is planar, with a maximum deviation of 0.042 (2) Å for atom C1, and makes an angle of 79.04 (8)° with the ester unit. Isatin derivatives are characterized by a rather long bond between the carbon atoms of the diketone group. A search of the Cambridge Structural Database (Version 5.28; Allen, 2002) revealed that the average bond distance for 23 entries is 1.56 (3) Å, which is longer than a normal Csp^2—Csp^2 bond due to steric repulsion between the ketone functions. The value of C1—C3 in (I) is 1.558 (2) Å.

Experimental

To a solution of isatin (10 mmol) in dimethylformamide (50 ml), ethyl bromoacetate (12 mmol), potassium carbonate (12 mmol) and

tetrabutylammonium bromide (1 mmol) were added. The reaction mixture was stirred at room temperature for 24 h. After removal of the salts by filtration, the solvent was removed under reduced pressure and the resulting residue was treated with dichloromethane (20 ml). The pure compound was isolated after a second filtration and recrystallized from methanol (yield 1.77 g, 76%; m.p. 401–403 K).

Crystal data

$C_{12}H_{11}NO_4$
M_r = 233.22
Monoclinic, $P2_1/c$
a = 13.6433 (6) Å
b = 4.9505 (3) Å
c = 16.2899 (8) Å
β = 104.246 (2)°
V = 1066.40 (10) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.93 mm^{-1}
T = 100 (2) K
0.5 × 0.2 × 0.2 mm

Data collection

Bruker SMART 6000 diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.594, T_{max} = 0.831
10352 measured reflections
2000 independent reflections
1725 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.104
S = 1.06
2000 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

H atoms were positioned geometrically and refined as riding, with C—H = 0.95–0.99 Å and U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl and 1.2 for all other H atoms.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

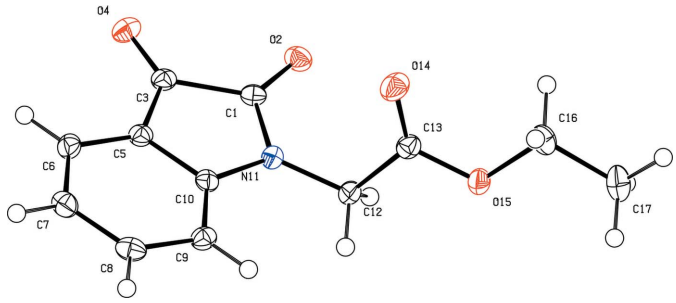


Figure 1
The molecular structure of the title compound, (I), showing the atom-labeling scheme and with displacement ellipsoids drawn at the 50% probability level.

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