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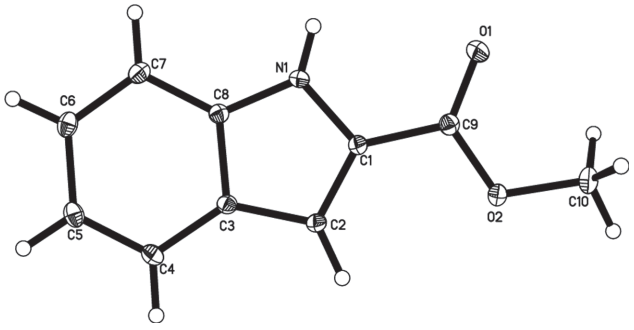
Crystal structure of methyl 1*H*-indole-2-carboxylate, $C_{10}H_9NO_2$



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Abstract

$C_{10}H_9NO_2$, monoclinic, $P2_1/c$ (no. 14), $a = 5.6463(6)$ Å, $b = 21.470(3)$ Å, $c = 7.3961(9)$ Å, $\beta = 112.015(4)°$, $V = 831.24(17)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.051$, $wR_{ref}(F^2) = 0.133$, $T = 150$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.67 × 0.35 × 0.24 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.0 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	68°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	24965, 3336, 0.048
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2626
$N(param)_{refined}$:	123
Programs:	SHELX [1], Bruker programs [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.27027(17)	0.55624(4)	0.09698(13)	0.01908(19)
O2	0.69759(16)	0.54864(4)	0.25586(13)	0.01698(18)
N1	0.21554(18)	0.43512(4)	0.22395(14)	0.01329(18)
C1	0.4485(2)	0.46450(5)	0.27445(16)	0.01219(19)
C2	0.6409(2)	0.42628(5)	0.39115(16)	0.0132(2)
H2A	0.8181	0.4357	0.4461	0.016*
C3	0.5216(2)	0.36967(5)	0.41345(16)	0.01238(19)
C4	0.6108(2)	0.31344(5)	0.51472(17)	0.0155(2)
H4A	0.7879	0.3074	0.5875	0.019*
C5	0.4369(2)	0.26728(5)	0.50654(18)	0.0165(2)
H5A	0.4954	0.2296	0.5763	0.020*
C6	0.1736(2)	0.27510(5)	0.39641(17)	0.0163(2)
H6A	0.0586	0.2424	0.3929	0.020*
C7	0.0793(2)	0.32932(5)	0.29350(17)	0.0151(2)
H7A	−0.0976	0.3343	0.2182	0.018*
C8	0.2556(2)	0.37669(5)	0.30490(16)	0.01200(19)
C9	0.4584(2)	0.52724(5)	0.19995(16)	0.0135(2)
C10	0.7243(2)	0.61044(5)	0.18754(18)	0.0182(2)
H10A	0.9018	0.6170	0.2008	0.027*
H10B	0.6776	0.6415	0.2654	0.027*
H10C	0.6115	0.6146	0.0502	0.027*
H1N1	0.075(4)	0.4499(9)	0.139(3)	0.035(5)*

Source of material

A suspension containing 1*H*-indole-2-carboxylic acid (2.00 g, 12.42 mmol) and few drops of concentrated sulfuric acid in absolute methanol (30 mL) was refluxed for 4 h. The reaction mixture was cooled to ambient temperature and the

obtained solid was filtered off to afford 2.00 g (92%) of methyl 1*H*-indole-2-carboxylate as yellow crystals, m.p. 434–435 K [3]. Slow evaporation of methanolic solution of this product yielded its pale yellow single crystals.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The methyl groups were idealized and refined using rigid groups allowed to rotate about the O—C bond (AFIX 137 option of the SHELXL-2013 program [1]). The U_{iso} values of the hydrogen atoms of the methyl groups were set to $1.5U_{eq}$(C) and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}$(C).

Comment

Indole-bearing compounds have received a significant research interest due to their ubiquitous natural occurrence, e.g. in marine natural products, fungal metabolites and vinca alkaloids [4]. Indoles constitute the scaffold of a number of pharmaceuticals and they are considered an important class of therapeutic agents in medicinal chemistry with diverse biological activities such as antimicrobial [5], anti-inflammatory [6], antitumor [7], analgesic [8], management of sleep disorders [9] and antiviral [10]. The title compound is an indole-bearing molecule which can be used for the preparation of different biologically active compounds endowed with a broad spectrum of biological activities.

The asymmetric unit of the title structure contains one molecule where the indole moiety is essentially planar. Centrosymmetric dimers are formed via N1—H1N1 · · · O1 hydrogen bonds [D · · · A distance 2.8830(14) Å and D—H · · · A angle 154.4(18)]. When viewed perpendicular to the bc plane, the overall packing can be described as alternating layers of parallel molecules arranged in a herringbone fashion.

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