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Short Note

Ethyl 3-{2-[(3-Methyl-1*H*-indol-2-yl)carbonyl]hydrazinylidene}-butanoate

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Abstract: The title compound, ethyl 3-{2-[(3-methyl-1H-indol-2-yl)carbonyl]hydrazinylidene} butanoate (**3**), was prepared *via* reaction of 3-methyl-1*H*-indole-2-carbohydrazide (**1**) and ethyl 3-oxobutanoate (**2**) under reflux. The structure of the synthesized compound was assigned on the basis of elemental analysis, IR, ^{1}H-NMR, mass spectral and X-ray data.

Keywords: 3-methyl-1*H*-indole-2-carbohydrazide; X-ray

Indoles are among the most important nitrogen-containing heterocyclic molecules, found extensively in biological systems which play a vital role in biochemical processes. The indole ring system is found in many natural products, pharmaceutical agents and polymer materials [1–7]. The interesting chemical properties of indole have inspired chemists to design and synthesize a variety of indole derivatives [8]. In view of this finding, in the present paper we describe the synthesis of a new indole derivative with expected biological activity.

Results and Discussion

3-Methyl-1*H*-indole-2-carbohydrazide (**1**) [9] was allowed to react with ethyl 3-oxobutanoate (**2**) in ethanolic solution under reflux. The expected product of this reaction was assumed to be one of the seven structures **3–9** shown in Scheme 1. IR, ^{1}H-NMR, mass spectral data and single-crystal X-ray diffraction studies (Figure 1 and Table 1) of the isolated product were in full agreement with the structure **3**, but not with the other structures **4–9**.

Scheme 1. Synthesis of the title compound (**3**).

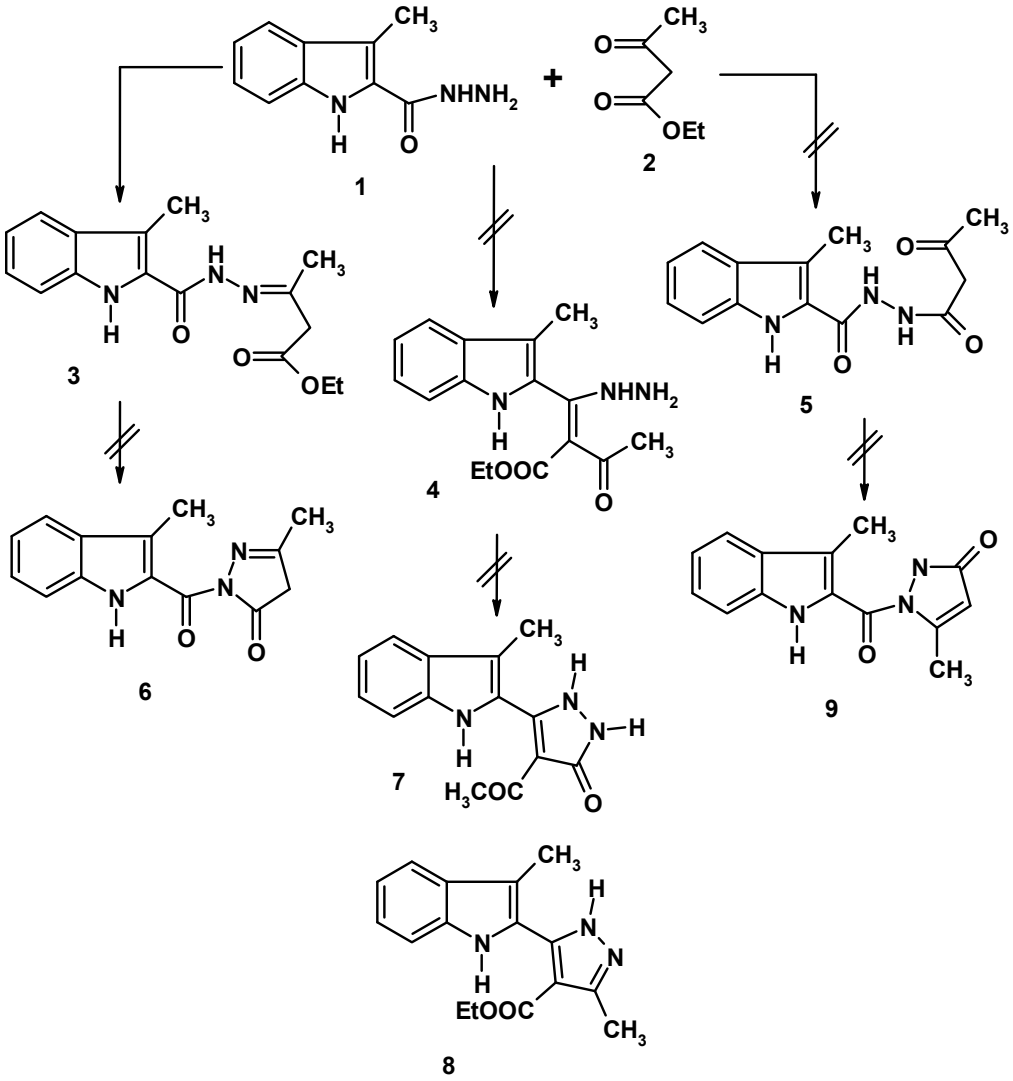

Figure 1. The X-ray crystal structure of compound **3**.

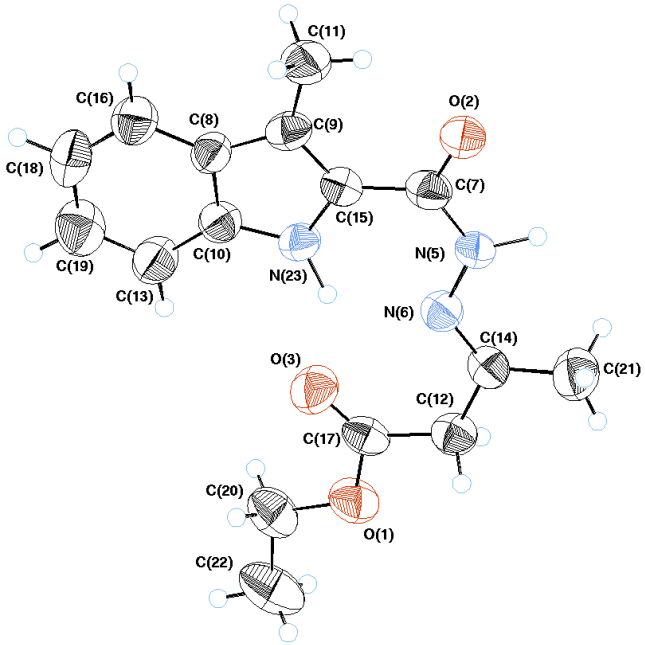

Table 1. Selected bond lengths and bond angles in the ORTEP representation of compound **3** in the crystal. The crystallographic numbering does not reflect systematic numbering.

Bond length, Å	**Bond length, Å**	**Bond length, Å**
C7—C15, 1.472	N6—C14, 1.286	C12—C17, 1.517
N5—C7, 1.359	C14—C21, 1.487	O1—C17, 1.337
N5—N6. 1.380	C12—C14, 1.493	O1—C20, 1.450
Angle (ω)	**Angle (ω)**	**Angle (ω)**
C17—O1—C20, 115.7	O2—C7—N5, 117.4	N23—C10—C8, 108.4
C10—N23—C15, 108.5	O2—C7—C15, 120.3	N23—C10—C13, 129.4
N6—N5—C7, 121.0	N5—C7—C15, 122.3	C8—C10—C13, 122.2

Crystallographic Analysis

The crystals were mounted on a glass fiber. All measurements were performed on an ENRAF NONIUS FR 590. The data were collected at a temperature of 25 °C using the ω scanning technique to a maximum of a 20 of 22.986°. The structure was solved by direct method using SIR 92 and refined by full-matrix least squares. Non-hydrogen atoms were refined anisotropically. Hydrogen atoms were located geometrically and were refined isotropically [10].

Crystal Data for Compound 3

$C_{16}H_{19}N_3O_3$, M = 301.346, monoclinic, a = 19.3108 (8), b = 7.7168 (4), c = 21.0944 (10) A^o, v = 3131.2 (3), $\alpha = \gamma = 90.00°$, $\beta = 95.065\ (3)°$, space group: $P2_1/c$, Z=8, $D_x = 1.279$ Mg m^{-3} reflection 937 measured, $\theta_{max} = 27.50°$. Figure 1 illustrates the structure as determined [10].

Synthesis of Ethyl 3-{2-[(3-Methyl-1*H*-indol-2-yl)carbonyl]hydrazinylidene}butanoate (3)

A mixture of the hydrazide **1** (1.89 g, 10 mmol) and ethyl 3-oxobutanoate **2** (10 mmol) in absolute ethanol (20 mL) was heated at reflux temperature for 2 h. The reaction mixture was then cooled and the formed precipitate was filtered off, washed with ethanol to afford the title compound **3**. Yield: 84%; yellow microcrystals (from ethanol); mp: 238–240 °C. IR (KBr): v 1706, 1668 (2 C=O), 3409, 3234 (2 NH) cm^{-1}. ^1H NMR (DMSO-d_6): δ 1.24 (t, *J* = 7.0 Hz, 3H, CH_3), 2.03 (s, 3H, CH_3), 2.53 (s, 3H, CH_3), 3.43 (s, 2H, CH_2), 4.23 (q, *J* = 7.0 Hz, 2H, CH_2), 6.87–7.98 (m, 4H, ArH), 10.38 (s, 1H, D_2O exchangeable, NH), 11.21 (s, 1H, D_2O exchangeable, NH). MS *m/z* (%): 301 (M^+, 42), 189 (100), 155 (318), 117 (46), 77 (21).

Anal. Calcd for $C_{16}H_{19}N_3O_3$ (301.34): C, 63.77; H, 6.36; N, 13.94. Found C, 63.39; H, 6.46; N, 13.65.

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