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Ethyl 2-acetyl-3-[2-(1*H*-indol-3-yl)ethylamino]propenoate

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Key indicators

Single-crystal X-ray study
T = 183 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.111
Data-to-parameter ratio = 13.1

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

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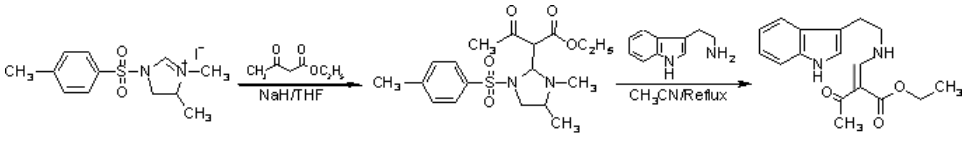
The title compound, $C_{17}H_{20}N_2O_3$, was synthesized by the substituted one-carbon unit transfer reaction of tryptamine with an imidazolidine derivative. There are intramolecular and intermolecular N—H···O hydrogen bonds in the crystal structure.

Comment

Secondary enamines have attracted a great deal of attention in recent years because of their range of applications (Duthaler, 2003; Stanovnik & Svete, 2004; Elassar & El-Khair, 2003). Moreover, functionalized secondary enamine derivatives may enable chemical and biological studies on these derivatives, which will be used in pharmaceutical research. We were therefore interested in the synthesis of secondary enamine derivatives during our investigation of tetrahydrofolate coenzyme models (Li *et al.*, 2004). Tetrahydrofolate is involved in the biological transfer of a carbon unit at different oxidation levels (Blakley, 1969), which can transfer —C═C— or ═CH— groups *via* mechanisms analogous to those operative in biochemical processes (Bieraugel *et al.*, 1983; Pandit & Bieraugel, 1979).



(I)

The title compound, (I), was obtained by the substituted one-carbon unit transfer reaction of tryptamine with the imidazolidine derivative which was produced by the addition reaction of 1-tosyl-3,4-dimethylimidazolinium iodide with a carbanion of acetylacetate. This might be regarded as a

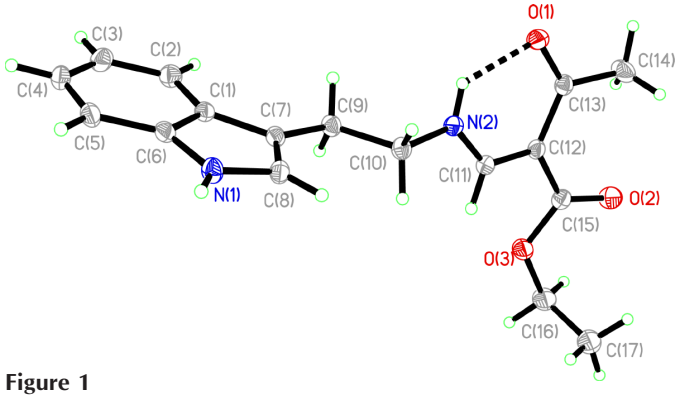


Figure 1
The molecular structure of (I), showing 50% probability ellipsoids. The dashed line indicates a hydrogen bond.

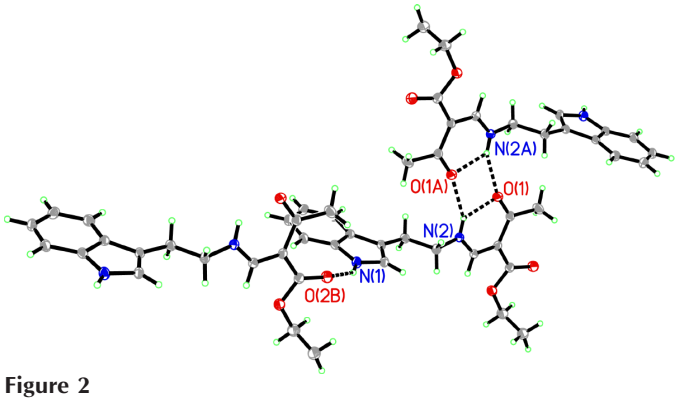


Figure 2
The three-centered hydrogen bonds between two O atoms of two carbonyl groups and atom N2. [Symmetry codes: (A) $-x, 1-y, 1-z$; (B) $x, y-1, z+1$.]

substituted one-carbon unit transfer reaction which mimics the function of tetrahydrofolate coenzymes.

The molecular conformation of (I) is illustrated in Fig. 1, and some features of the geometry are listed in Table 1. A number of intra- and intermolecular hydrogen bonds (Table 2) stabilize the crystal structure. Three-centered hydrogen bonds are formed between two O atoms of two carbonyl groups and atom N2 (Fig. 2).

Experimental

Sodium hydride (1.8 mmol) was added to a solution of acetyl acetate (1.5 mmol) in dry tetrahydrofuran (10 ml), which was cooled in an ice–water bath. The reaction mixture was stirred for 30 min, then 1-tosyl-3,4-dimethylimidazolinium iodide (1 mmol) was added and the mixture was allowed to warm to room temperature and stirred continuously for 3 h, then quenched with water. The solution was extracted with dichloromethane, dried over anhydrous sodium sulfate and concentrated. The residue was purified by column chromatography to afford a yellow oil, (II). The imidazolidine derivative, (II) (1 mmol), and tryptamine (1 mmol) in anhydrous acetonitrile (10 ml) were refluxed, the solution concentrated and the residue purified by column chromatography to afford compound (I) (yield 85%). Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{17}H_{20}N_2O_3$
$M_r = 300.35$
Triclinic, $P\overline{1}$
$a = 8.4342\ (18)$ Å
$b = 9.391\ (2)$ Å
$c = 10.360\ (2)$ Å
$\alpha = 79.941\ (3)^\circ$
$\beta = 71.597\ (3)^\circ$
$\gamma = 89.332\ (3)^\circ$
$V = 765.8\ (3)$ Å^3
$Z = 2$
$D_x = 1.303$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1672 reflections
$\theta = 2.5$–26.9°
$\mu = 0.09$ mm^{-1}
$T = 183\ (2)$ K
Block, colorless
0.40 × 0.30 × 0.30 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.494$, $T_{max} = 0.973$
3165 measured reflections
2636 independent reflections
2154 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 25.0^\circ$
$h = -10 \rightarrow 8$
$k = -10 \rightarrow 11$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.111$
$S = 1.01$
2636 reflections
201 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C13	1.2358 (19)	N1—C6	1.368 (2)
O2—C15	1.2105 (19)	N1—C8	1.369 (2)
O3—C15	1.347 (2)	N2—C11	1.2998 (19)
O3—C16	1.448 (2)	N2—C10	1.4556 (19)
C1—C7—C9—C10	171.15 (15)	C7—C9—C10—N2	−168.51 (13)
C11—N2—C10—C9	−110.86 (18)	C10—N2—C11—C12	−172.13 (15)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.88	2.02	2.6518 (17)	128
N2—H2···O1^{i}	0.88	2.32	3.0393 (18)	139
N1—H1···O2^{ii}	0.88	2.07	2.8572 (17)	149

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $x, y-1, 1+z$.

H atoms attached to C and N atoms were placed in geometrically idealized positions, and constrained to ride on their parent atoms, with C—H = 0.93–0.96 Å and N—H = 0.88 Å, and with U_{iso}(H) = $1.2U_{eq}$(parent atom).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL/PC*.

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