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Data article

Ethyl 2-(4-fluorobenzylidene)-3-oxobutanoate: Synthesis, crystal structure and antimicrobial activities

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ABSTRACT

Ethyl 2-(4-fluorobenzylidene)-3-oxobutanoate was synthesized by Knoevenagel condensation reaction of 4-fluorobenzaldehyde and ethyl acetoacetate in the presence of catalytic amount of piperidine and trifluoroacetic acid in benzene under reflux conditions. The structure of the synthesized molecule was obtained by spectral studies and was confirmed by X-ray diffraction studies. The title compound crystallizes in the monoclinic crystal system under the space group P21/n. The structure adopts a *Z* conformation about the C=C bond. The synthesized new molecule was evaluated *in vitro* for its antifungal and antimicrobial susceptibilities.

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Specifications Table

Subject area	Chemical physics
Compound	Ethyl 2-(4-fluorobenzylidene)-3-oxobutanoate
Data category	Synthesis, ^{1}H NMR, mass spectra, crystallographic data
Data acquisition format	CIF for crystallography

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Data type	Analyzed
Procedure	The compound $C_{13}H_{13}FO_3$, ethyl 2-(4-fluoorobenzylidene)-3-oxobutanoate was synthesized and yellow rectangular block shaped crystals of the compound were obtained by slow evaporation technique. A single crystal of dimension $0.38 \times 0.44 \times 0.54$ mm of the title compound was selected and X-ray intensity data were collected with χ fixed at 54° and φ, from 0° to 360° at a scan width of 0.5°, exposure time of 3 s and a sample to detect distance of 50.0 mm at 293 K.
Data accessibility	CCDC 1491046 URL: https://www.ccdc.cam.ac.uk/conts/retrieving.html

1. Rationale

Knoevenagel condensation was a versatile tool for the synthesis of α, β-unsaturated carbonyl compounds [1], which involve the base catalysed reaction of aldehydes and reactive methylene compounds. Exploration of the simple molecules such as α, β-unsaturated carbonyl compounds in to bioactive compounds is a worthwhile contribution in the field of medicinal chemistry. α, β-unsaturated ketones were extensively used as a synthetic scaffolds in the construction of biologically potent molecules such as such as benzothiazepines [2,3], isoxazoles [4], pyrazoles [5], cyclopropyl esters [6], thiadiazoles [7], lignans [8] etc. In view of wide synthetic utilities associated with α, β-unsaturated carbonyl compounds, we herein report the synthesis, spectral analysis, single crystal X-ray diffraction studies, and antimicrobial activity studies of ethyl 2-(4-fluorobenzylidene)-3-oxobutanoate.

2. Procedure

2.1. Synthesis of the title compound

To the solution of ethyl acetoacetate, **2** (0.016 mol) in dry benzene (15 ml), piperidine (0.0016 mol) and trifluoro acetic acid (0.0016 mol) were added; the mixture was stirred for 15 min. After this, 4-fluorobenzaldehyde, **1** (0.016 mol) was added and then the mixture was refluxed on a water bath for 5 h. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was poured into ice cold water. The solid separated was filtered, washed with ice cold water and crystallized from ethyl acetate to obtain the target molecule Ethyl 2-(4-fluorobenzylidene)-3-oxobutanoate, **3** as yellow rectangular slab like crystals in 76% yield, m.p. 131 °C. The schematic representation of the reaction is given in Fig 1.

2.2. Spectral data

^{1}H NMR and ^{13}C NMR spectra were recorded on Agilent-NMR 400 MHz and 100 MHz spectrometer respectively. The solvent $CDCl_3$ with TMS as an internal standard was used to record the spectra. The

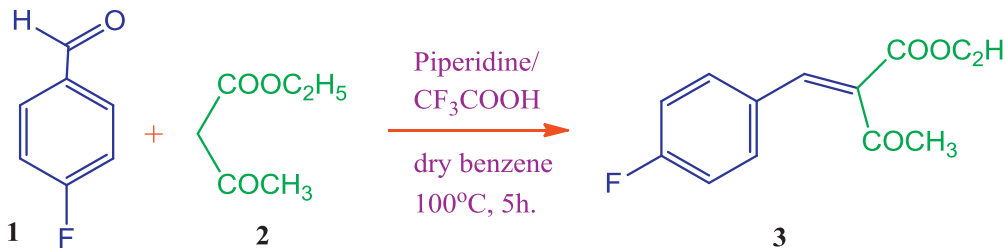

Fig. 1. Reaction pathway for the synthesis of Ethyl 2-(4-fluobenzylidene)-3 oxobutanoate.

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