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КРАТКИЕ СООБЩЕНИЯ

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CRYSTAL STRUCTURE OF 1-(4-CHLOROPHENYL)-3-{4-[2-(5-ETHYL-PYRIDIN-2-YL)-ETHOXY]-PHENYL}-PROPENONE

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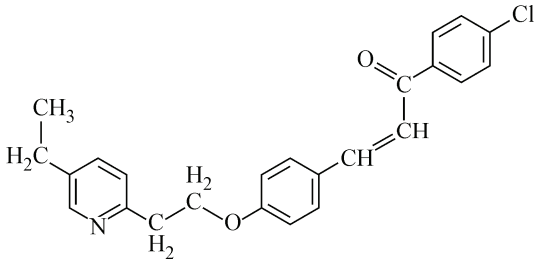
The title compound $C_{24}H_{22}ClNO_2$ belongs to the orthorhombic system, space group $Pca2_1$ with $a = 12.1771(10)$ Å, $b = 4.9305(4)$ Å, $c = 34.419(3)$ Å, $\alpha = \beta = \gamma = 90°$, $V = 2066.5(3)$ Å^3, $Z = 4$, $D_c = 1.260$ g/cm^3, $F(000) = 824$, $R = 0.0402$ and $wR = 0.1144$, $S = 1.034$, $T = 293$ K. The compound is a chalcone with 4-chlorophenyl and [(5-ethyl-pyridin-2-yl)-ethoxy]-phenyl substituents bonded at the opposite ends of a propenone group, the biologically active region. The propenone bridge makes dihedral angles of 10.61(23)° and 62.75(22)° respectively, with 4-chlorophenyl and the [(5-ethyl-pyridin-2-yl)-ethoxy]-phenyl group.

K e y w o r d s: chalcones, propenone, chlorophenyl, pyridine ring.

Introduction. Chalcones or 1,3-diaryl-2-propen-1-ones belong to the flavonoid family. Chemically, they consist of open-chain flavonoids in which two aromatic rings are joined by a three-carbon α-β-unsaturated carbonyl system. The rising prevalence of multi-drug resistant gram-positive and gram-negative bacteria continues to provide impetus for the search for and discovery of novel antimicrobial agents active against these pathogens [1]. A vast number of naturally occurring chalcones are polyhydroxylated in the aryl rings. The radical quenching properties of the phenolic groups present in many chalcones have raised interest in using the compounds or chalcone-rich plant extracts as drugs or food preservatives. Among many useful properties that chalcones have been reported to possess include anti-inflammatory, antimicrobial, antifungal, antioxidant, cytotoxic, HIV-1 protease inhibitory, tyrosinase inhibitory, antiprotozoal, antiulcer as well as other activities. More importantly, chalcones have shown several anticancer activities as inhibitors of cancer cell proliferation, carcinogenesis and metastasis [2—6]. Many chalcones have been assessed for their high antimalarial activity, which is probably a result of Michael addition of nucleophilic species to the double bond of the enone [2]. Some chalcones demonstrated the ability to block voltage-dependent potassium channels. Chalcones are also finding application as organic compounds reported to have NLO properties; chalcone derivatives are a recognized material because of their excellent blue light transmittance and good crystallization ability. The basic skeleton of chalcones is useful as the starting material for the synthesis of various biodynamic heterocyclic compounds such as cyclohexane derivatives and pyrazoline derivatives [7]. Owing to the importance of these flavonoid analogs, we report here the crystal structure of the title chalcone derivative.

Experimental. To a solution of 4-(2-(5-ethylpyridin-2-yl)ethoxy)benzaldehyde (0.01 mol) in methanol (50 ml), 4-chloro acetophenone (0.01 mol) was added in the presence of 2 % NaOH solution (5 ml). The reaction mixture was stirred for 10—12 h at room temperature. The solvent was distilled

off and the crude product was poured into ice water. The compound thus obtained was washed with water and recrystallised from ethanol. The compound was synthesized using the published method [1, 4, 5, 8, 9]. A chemical diagram of compound **1** is shown in Scheme 1. CIF file containing complete information on the studied structure was deposited with CCDC, deposition number 854383, and is freely available upon request from the following web site: www.ccdc.cam.ac.uk/data_request/cif.



X-ray crystallography. A brown crystal of title compound **1** with dimensions 0.35×0.30× ×0.25 mm was chosen for the data collection. The data were collected with graphite-monochromated MoK_α radiation (λ = 0.71073 Å). For compound **1** data collection: APEX2 [10]; cell refinement: APEX2/SAINT [10]; data reduction: SAINT/XPREP [10]; molecular graphics: ORTEP-3 [11] and Mercury [12] for Windows; publication software: PLATON [13]. The structure of compound **1** was solved by direct methods using SHELXS-97 [14] and refined using SHELXL-97 [14]. All non-hydrogen atoms were assigned anisotropic displacement parameters in the refinement. All hydrogen atoms were positioned geometrically and treated as riding on their parent atoms, with C—H = 0.93 Å (aromatic) and 0.97 Å (methylene), and refined using a riding model with U_{iso}(H) = 1.2U_{eq} or 1.5U_{eq} (parent atom). In the range 2.37—23.89, a total of 3210 reflections were collected, out of which 2693 were independent (R_{int} = 0.020). The maximum and minimum peaks and holes are 0.30 e/Å^3 and –0.18 e/Å^3 respectively. S = 1.034 and wR = 0.1144.

Results and discussion. The title compound $C_{24}H_{22}ClNO_2$ crystallized in the orthorhombic system, space group $Pca2_1$ with a = 12.1771(10) Å, b = 4.9305(4) Å, c = 34.419(3) Å, $\alpha = \beta = \gamma = 90°$, V = 2066.5(3) Å^3, Z = 4, D_c = 1.260 g/cm^3, F(000) = 824, R = 0.0402 and wR = 0.1144, S = 1.034, T = 293 K. The compound is a chalcone with 4-chlorophenyl and [(5-ethyl-pyridin-2-yl)-ethoxy]-phenyl substituents bonded at the opposite ends of a propenone group, the biologically active region. The propenone unit (C16—C18/O2) is planar [r.m.s. derivation 0.0052 Å] and torsion angle C16—C17—O2 being –1.67(61)°. The propenone bridge makes dihedral angles of 10.61(23)° and 62.75(22)° respectively with 4-chlorophenyl and the [(5-ethyl-pyridin-2-yl)-ethoxy]-phenyl group. The pyridine ring system and the benzene ring are oriented at a dihedral angle of 83.42(10)°. The ethoxy group is coplanar with the phenyl ring [dihedral angle = 7.68(27)°]. Bond lengths [15] and angles are within the normal range and are comparable to those of the closely related structures [2, 3, 6, 7, 16]. The widening of the exocyclic angles C17—C16—C13 [128.4(3)°] and C16—C13—C12 [123.1(3)°] deviates significantly from the normal value of 120° and might be due to the repulsion be-

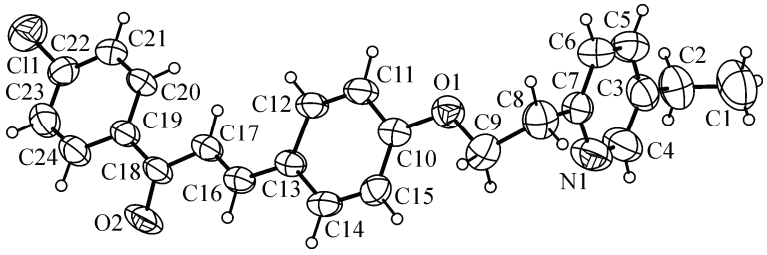


Fig. 1. The molecular structure of **1** with the atom numbering scheme. Displacement ellipsoids are drawn at the 50 % probability level

tween H12 and H17 at C17 (H12…H17 = 2.2403 Å). The ethyl group is equatorially substituted to the pyridine ring. While no classical hydrogen bonds are present, weak intermolecular C—H…π-ring interactions are observed, which contribute to the stability of crystal packing. In the crystal, the molecules are stacked in columns along the *c* axis and several intermolecular π—π interactions are present between the six-membered rings, with the shortest distance of 3.573 Å, as observed in [17].

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