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CRYSTAL STRUCTURE ANALYSIS OF ETHYL4-[4-HYDROXY-3-METHOXPHENYL]-6-METHYL-2-THIOXO-1,2,3,4-TETRAHYDROPYRIMIDINE-5-CARBOXYLATE

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Abstract: **The title compound crystallizes in the triclinic space group p -1, with a = 8.474(5) Å, b = 9.402(5) Å, c = 11.239(5) Å, α = 98.133(5)˚ , β = 108.117(5)˚, γ = 108.575(5)˚, V=777.4(7) Å^3 and the number of molecules in the unit cell Z=2. In the title compound, $C_{15}H_{18}N_2O_4S$, the dihydropyrimidine ring adopts a boat conformation. The dihedral angle between the benzene and tetrahydropyrimidine is 80.29(8)˚. In the crystal, molecules are linked via pairs of C—H…S hydrogen bonds, forming inversion dimmers with an $R_2^2(12)$ ring motif. The crystal structure is further stabilized by an intramolecular C—H…O hydrogen bonds forming a closed six membered ring.**

Keywords – **tetrahydropyrimidine, intermolecular, ring motif, hydrogen bonds.**

I. INTRODUCTION

Pyrimidines are the most important six membered heterocyclic compounds containing two nitrogen atoms at 1 and 3 position. The chemistry of pyrimdines has become increasingly important as a result of recent developments in medicinal chemistry. Pyrimidine derivatives have been investigated extensively due to their great biological significance and as main constituent of nucleic acids. Pyrimidines and their derivatives are considered to be important for drugs and agricultural chemicals. They are also found to exhibit remarkable pharmacological activities such as anti-cancer, anti-tumor, anti-inflammatory and antifungal etc and are used widely as agrochemicals, pharmaceuticals, dyes, organic additives in electroplating of steel and in the polymerization process[1,2]. Our interest in the preparation of pharmacologically active compounds led us to synthesize the title compound and we report its crystal structure herein.

II. MATERIALS AND METHODS

Single crystal X-ray diffraction analysis was carried out to confirm crystalline quality and also to identify the universal lattice parameters using Bruker Kappa APEXII CCD diffractometer[3]. The Mokα radiation of wavelength (λ = 0.71073 Å) and multi-scan technique for absorption were used for data collection. The lattice parameters were determined by the least-squares method on the basis of all reflections with $F^2 > 2\sigma (F^2)$

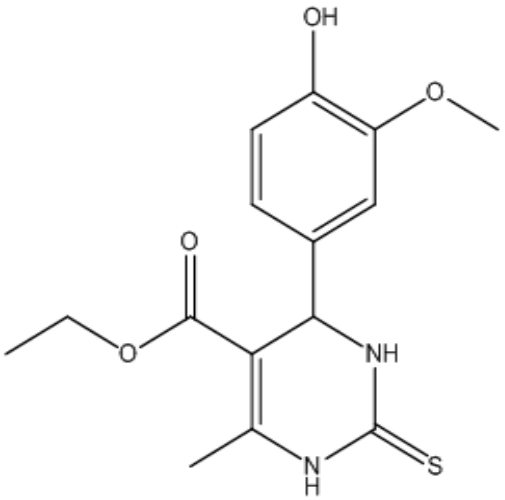
To an ethanolic solution of ethyl aceto acetate (2 ml, 0.01mol), 2-hydroxy-4-methoxybenzaldehyde (1.5 mL, 0.01 mol) and thiourea (2.3 g, 0.03 mol) were added followed by $CeCl_3\_7H_2O$ (931 mg). The reaction mixture was taken in a round-bottom flask and refluxed for 8 h. Then the reaction mixture was cooled and poured into crushed ice taken in a beaker with constant stirring. The solid separated out was filtered, washed with ice-cold water and then recrystallized from hot ethanol to afford the product [yield: 85%; m.p. 417- 419 K].

A suitable single crystal of dimension 0.20 x 0.15 x 0.10 mm was selected for X-ray structure determination. The structures were solved by direct methods using SHELXS-97 and refined by a full-matrix least-squares procedure using the program SHELXL-97[4]. The software used for molecular graphics are ORTEP-3 for windows[5] and PLATON[6]. The software used to prepare material for publication is WinGX publication routines[7]. All non-hydrogen atoms were assigned anisotropic displacement parameters in the refinement. H atoms were placed in geometrically idealized positions and refined as

riding on their parent, atoms with C-H distances fixed in the range 0.93-0.98 Å and N-H=0.86 Å with $U_{iso}(H)=1.5U_{eq}(CH_3)$ and $1.2U_{eq}(CH_2,CH,NH)$. The chemical structure of the title compound 1 is shown in **Scheme I**. Molecular structure of the title compound showing the atomic numbering scheme is shown in **Figure I**. The crystallography details for the structure determination of the compound are displayed in **Table I** and hydrogen bond geometry are listed in **Table II** respectively.

III. RESULT AND DISCUSSION

The chemical structure of the title compound as shown in **Scheme I.**



Scheme I

The dihydropyrimidine ring adopts boat conformation, with puckering parameters are q2 = 0.294 Å, q3 = 0.073 Å, Q = 0.303 Å, Θ = 76.1(4)° and Φ = 47.3(4)˚. The benzene ring is planar with r.m.s deviation of 0.0076 Å and makes a dihedral angle of 80.29(8)˚ with the hydropyrimidine ring. The ethyl acetate group attached to the pyrimidine ring shows an extended conformation [torsion angle = C9—C12—O3—C13 = 179.27(2)°].

Table I. Crystal data and structure refinement parameters

Formula weight	322.37
Crystal shape, color	Block, colorless
Temperature	293 K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	a = 8.474(5)Å b = 9.402(5)Å c = 11.239(5)Å α = 98.133° β = 108.117° γ = 108.575°
Volume	777.4(7) $Å^3$
Z	2
Density (calculated)	1.377 Mg/m^3
Absorption coefficient	0.228 mm^{-1}
F(000)	340
Crystal size	0.20 x 0.15 x 0.10 mm
Theta range for data collection	2° to 26.6°
Index ranges	- 10≤ h ≤10 -11≤ k ≤11 -14 ≤ 1 ≤14
Reflection collected	11409
Max. and min. transmission	0.978 and 0.946
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3233/0/203
Goodness-of-fit on F^2	1.087
R indices(all data)	R_1= 0.024
Final R indices[1>2sigma(1)]	0.037 wR_2 = 0.108
Largest diff. peak and holes	0.31 e.$Å^{-3}$ and -0.22 e.$Å^{-3}$

In the crystal the molecules are linked via a pair of C—H…S hydrogen bonds forming an inversion dimmers with $R_2^2(12)$ ring motif and form a chain parallel to the ca plane (**Figure II**).

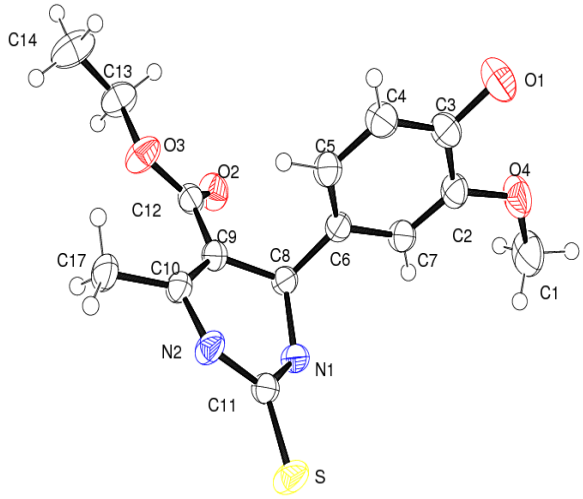


Figure I
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

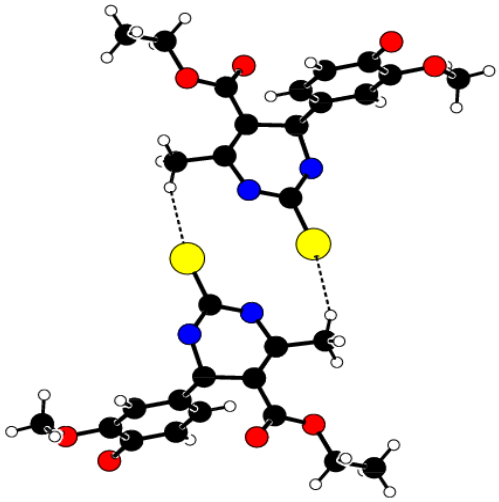


Figure II
Partial crystal packing diagram for the title compound, showing the $R_2^2(12)$ ring motifs. Hydrogen bonds are shown as dashed lines.

Table II - Hydrogen bond geometry (Å,°)

D—H...A	D—H	H...A	D...A	D—H...A
C17—H17A…S	0.96	2.87	3.781(4)	158

IV. CONCLUSION

We have synthesized the dihydropyrimidinones (DHPMS) derivative and characterized by single crystal X-ray diffraction analysis.

V. SUPPLEMENTARY DATA

CCDC 1448392 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data request/cif.

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