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SYNTHESIS OF 3-ALKYL-2-(ARYLIMINO) THIAZOLIDIN-4-ONE'S DERIVATIVES USING IONIC LIQUID

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Abstract : This study has been undertaken to investigate An efficient, regioselective synthesis of 2-arylimino-3-alkyl-thiazolidin-4-ones by applying [bmIm]OH ionic liquid as a catalyst for cyclization of substituted thiourea with ethyl bromoacetate is reported. The cyclized product 2-arylimino-3-alkyl-thiazolidin-4-ones were reacted with aromatic aldehydes under Knoevenagel condensation using [bmIm]OH as catalyst. Reusability of catalyst with good yields under green reaction conditions is the most remarkable feature of this synthetic method

Index Terms **- Regeioselective, 2-arylimino-3-alkyl-thiazolidin-4-ones, ionic liquid [bmIm] OH, Knoevenagel condensation, aromatic aldehydes.**

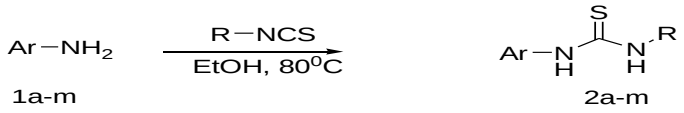
I. INTRODUCTION

There has been significant attention in the chemistry of thiazolidin-4-one ring structures, which is a core structure in various synthetic medicines displaying a wide-ranging spectrum of biological activities.[1] Thiazolidin-4-one ring also occurs in nature; Actithiazic acid [(–)-2-(5-carboxypentyl) thiazolidin-4-one)] isolated from streptomyces strains exhibits highly specific in vitro activity against Mycobacterium tuberculosis. [2] Thiazolidin-4-one derivatives are known to exhibit diverse bioactivities such as anti-convulsant,[3] anti-diarrheal,[4] anti-platelet activating factor,[5] anti-histaminic,[6] anti-microbial, [7] anti-diabetic, [8] cyclooxygenase inhibitory, [9] Ca2+ channel blocker, [10] PAF antagonist, [11] cardio protective, [12] anti-ischemic, [13] anticancer, [14] antiHIV, [15] non-peptide thrombin receptor antagonist[16] and tumor necrosis factor-α antagonist activities. [17] However, the 2-imino derivatives of thiazolidinones are explored to a lesser extent. Several methods are reported in literature for the preparation of Thiazolidinone derivatives. [18] This involves three-component one-pot synthesis, [19] ionic liquid mediated eco-friendly preparation, [20] DCC or HBTU catalyzed synthesis and solid phase synthetic protocol. [21] The use of task-specific ionic liquid-phase (ethylene glycol) as synthetic equivalent of ionic liquid-phase matrices for the preparation of a small library of 4-thiazolidinones in presence of (DCC/ DMAP as catalyst) was reported. [19a] Dandia et al. have reported a microwave-assisted three-component, regioselective one-pot cyclocondensation method for the synthesis of a series of novel spiro[indole-thiazolidinones] using an environmentally benign procedure at atmospheric pressure in an open vessel.[22]. Holmes et al. reported solution and polymer-supported synthesis of 4-thiazolidinones derived from amino acids [23] Recently Maclean et al. reported an encoded 4-thiazolidinone library on solid phase [24]. Different protocols have been developed allowing the synthesis of imino thiazolidin-4-one skeletons. [25] Ottana et al. also reported synthesis of 4-thiazolidinones using the starting material N-propyl-N'-phenylthiourea, [25b].Cesur et al. and Vicini et al. have reported another method of synthesis of 4-thiazolidinones by the use of thiocyanate, alkylisothiocynate and ammonium thiocyanate with hydrazide/ acetamide, followed by the treatment with ethyl bromoacetate and sodium acetate [25a ,25f]. These methods involves use of strong base like Et3N, DIPEA, KOH, NaOH, inorganic bases such as Sodium acetate and use of polar protic solvent and heating conditions these methods forms mixture of regiisomers. Basic ionic liquid [bmIm] OH can be used as mild base catalyst and solvent for these reactions. [BmIm] OH has advantage of low toxicity, recovery and reuse of ionic liquid and mild reaction condition.

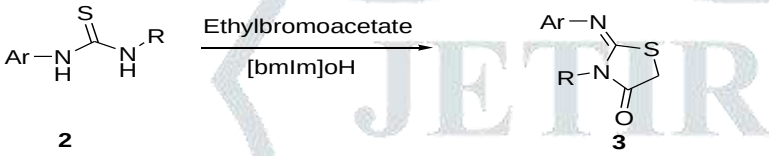
In recent years, considerable amount of growing research in the field of green organic chemistry is the application of ionic liquids as environmentally benign reaction media,[26] catalysts,[27] and reagents.[28] A basic functionalize, task specific ionic liquid, [bmIm]OH have been extensively applied as a catalyst in different organic reactions. [29]

II. RESULTS AND DISCUSSION

In our preliminary experiments, we synthesized 1-aryl-3-alkyl thioureas derivatives starting from reaction of suitable anilines with alkyl thiocyanate in ethanol. (Scheme-1)



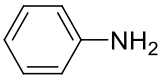
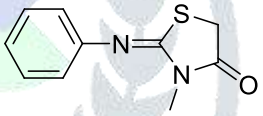
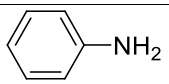
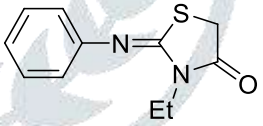
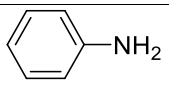
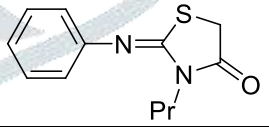
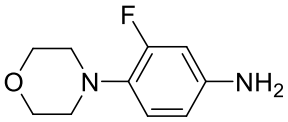
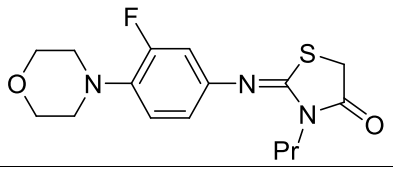
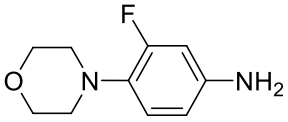
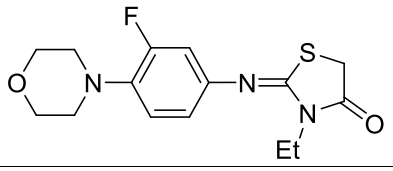
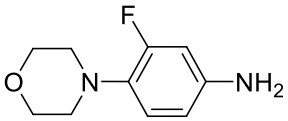
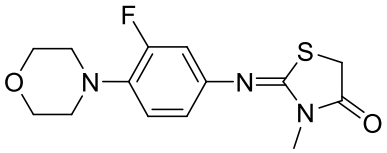
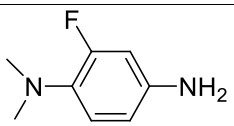
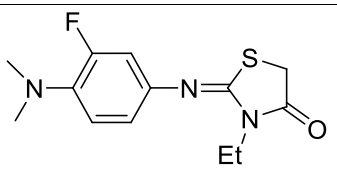
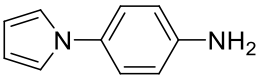
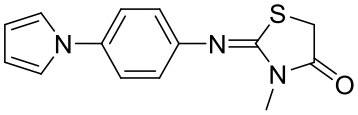
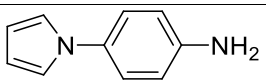
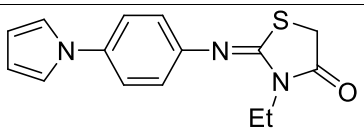
Scheme-1

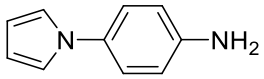
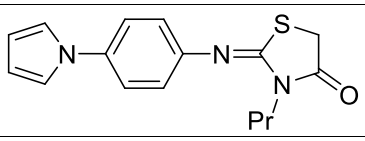
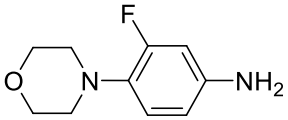
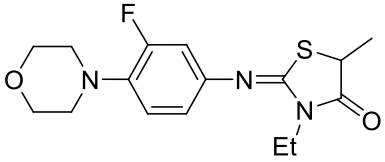
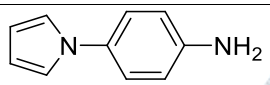
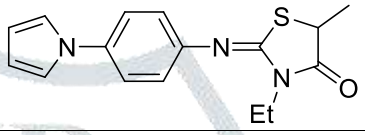
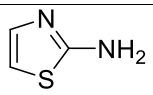
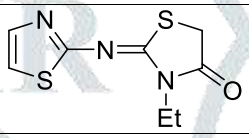


Scheme-2

Typically, aryl thioureas are characterized by IR absorptions at 3350-3320, 3250-3200 for the free and associated NH and at 1230-1250cm-1 for thiocarbonyl groups respectively and confirmed by its mass. Table1

Table1 Synthesis of 2-arylimino-3-alkyl-thiazolidin-4-ones using [bmIm]OH

Sr. No.(1)	Ar	R	**2** %Yield	**3**	**3** %yield*
a		Methyl	95		90
b		Ethyl	97		91
c		Propyl	91		93
d		Propyl	89		95
e		Ethyl	92		94
f		Methyl	88		90
g		Ethyl	84		89
h		Methyl	91		94
i		Ethyl	95		93

j		Propyl	94		94
k		Ethyl	92		95b
l		Ethyl	95		94b
m		Ethyl	92		98

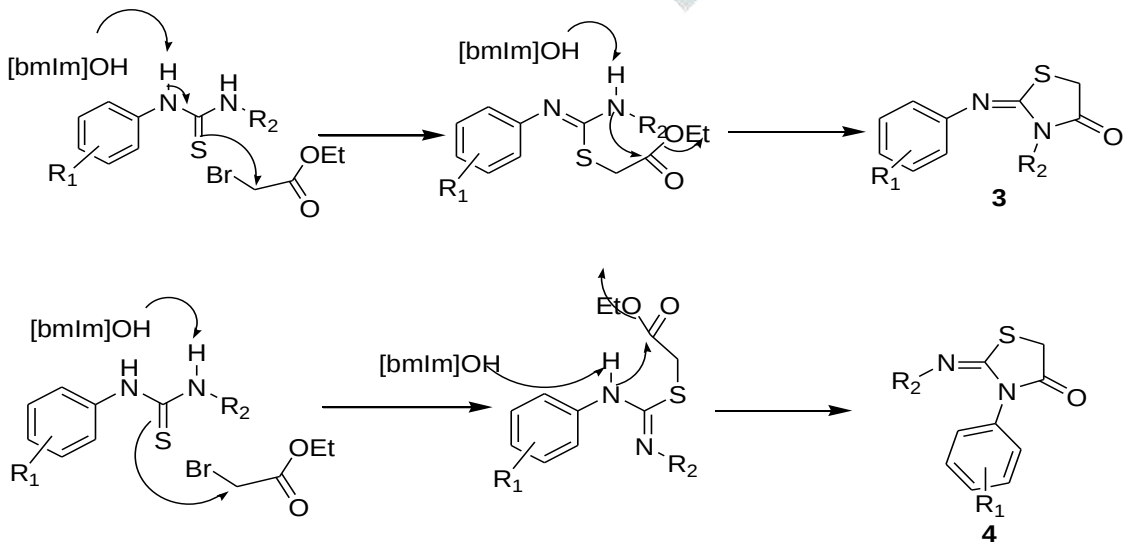
Then, we began to harness the basic Ionic liquid [bmIm] OH as a catalyst and solvent which evidenced its catalytic efficacy in the reaction affording 2-arylimino-3-alkyl-thiazolidin-4-ones in excellent yields. By applying the basic Ionic liquid [bmIm]OH, involving use of ethyl bromoacetate, Ionic liquid acts as base, the 2-arylimino-3-alkyl-thiazolidin-4-ones with various substituents on aromatic ring (3a-m) were Synthesized (**Scheme2**) from the corresponding 1-aryl-3-alkyl thioureas (1a-m) in good yields as shown inTable1.

We used ionic liquid in excess quantity because it served as catalyst as well as solvent in this reaction.

Cyclized product 3 was characterized by Mass spectroscopy shows (M+1) 205.6. Comp 3 was also characterized by 1H NMR spectrum which represents C-5 protons at 3.95 ppm of the iminothiazolidinone nucleus. IR spectral data showing C=O in the 1705 cm -1 and strong band of the C=N group in 1637 cm -1

Under these conditions, in majority of the cases only one Regio isomers were obtained. We have also carried these reactions using Triethylamine as a base it will take the longer reaction time and mixture of product obtained. It may be speculated that the difference in basicity of [bmIm]OH used in this reaction compared with triethylamine may play crucial role in accelerating the reaction and improving the yield of compound **3.**

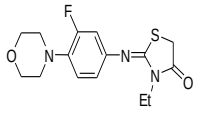
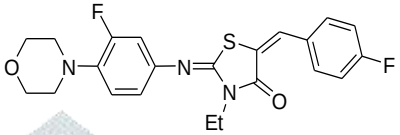
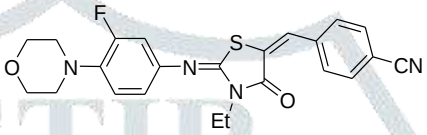
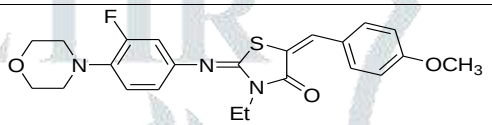
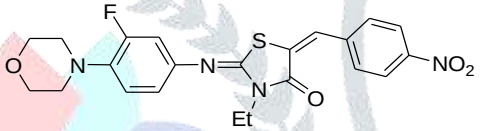
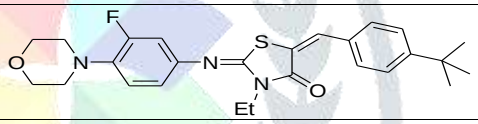
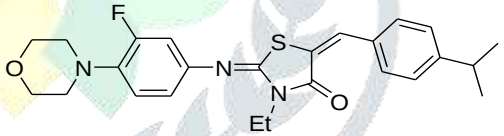
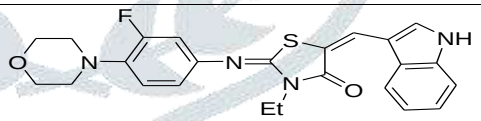
Base-catalyzed cyclization of 1-aryl-3-alkyl thioureas with ethyl bromoacetate may lead to different condensation products by S or N intramolecular cyclization because use of strong base like Et3N, DIPEA, KOH or polar aprotic solvent, use of strong base leads to a mixture of regioisomers **3** and **4** obtained by initial S-attack followed by N1 or N3cyclization (**Scheme-3**)



In order to verify similar type of reaction occurred using thiazolilamino-N'-methylthiourea with ethyl bromoacetate under same conditions we got one regioisomer (3m) with 98% yield. We also tried the reaction of thiourea of (2k) and (2l) with ethylbromopropaonate and we got one regioisomer with 93%, 95% yield respectively. The ionic liquid used in the reaction was recovered from aqueous layer and washed with diethyl ether to remove any organic impurities and dried under vacuum to get the pure ionic liquid and is reuse for the above reactions. We have tested reusability of ionic liquid for compound (3e), upon use of three times, showed no loss of its activity and does not vary yield of final product.

Then, we tried the Knoevenagel condensation of compound 3j with aromatic aldehyde the basic IL [bmIm] OH as a catalyst. 5-benzylidene-3-(3-fluoro-4-yl-morpholin-4-yl-phenylimino) thiazolidin-4-one derivatives were obtained in excellent yields. (Scheme-4) the results were shown in (Table2)

Table2 Knoevenagel condensation of compound **3j** with aromatic aldehyde using [bmIm]OH

Sr. No.	**3j**	**Benzaldehyde**	Knoevenagel product	3 %yield*
a		4-Flouro		94
b		4-Cyano		91
c		4-Methoxy		88
d		4-Nitro		90
e		4-t-butyl		93
f		4-isopropyl		95
g		Indole-3-carboxaldehyde		92

III.CONCLUSION

In conclusion, regioselective synthesis of 2-arylimino-3-alkyl-thiazolidin-4-ones using task specific ionic liquid [bmIm]OH catalyzed cyclization of 1-aryl-3-alkylthioureas with ethyl bromoacetate The advantages of this method is mild and green reaction conditions , higher yields and use of ionic liquid which is prepared from easily available starting materials . Also formation of one regioisomer occurred during the synthesis. Knoevenagel condensation of this less reactive iminothiazolidinone was done using [bmIm]OH as catalyst and solvent.

IV.EXPERIMENTAL

Methods

The task specific ionic liquid [bmIm]OH was prepared according to the literature [29a]. Progress of reaction was monitored by silica gel-G coated TLC plates in Ethyl acetate: Hexane system (5:5). The spot was visualized by exposing dry plate in iodine vapours Melting points were determined by the melting point determination apparatus (TEMPO) in open capillary tubes and are uncorrected. Elemental analyses were done using Carlo Erba 1106 CHN Analyzer. Infrared spectra were recorded on Schimadzu 8201 PC, FTIR spectrophotometer (max in cm-1) spectrophotomer in KBr phase. Proton NMR spectra were recorded on Bruker Advance II 400 &200 NMR Ultra Shield Spectrometer using DMSO-d6/CDCl3 as a solvent and tetramethyl silane as internal standard. Chemical shift value is expressed in delta parts per million (ppm).

General Procedure for preparation N-Phenyl N'-methylthiourea (2a-m)

A solution of aniline(0.11mmol),was added to a solution of methylisothiocynate (0.12mmol) in absolute ethanol Reaction mixture was refluxed for 2hour.The reaction mixture was cooled to room temperature and solvent was removed under reduced pressure, the precipitate was filtered and recrystallized from cold ethanol to give off-white solid (1.2gm,87%) MS=m/z 165(M-1) IR (KBr) 3240(NH), 1170(C=S) cm-1; 1H NMR (CDCl3)3.11(s, 3H), 6.91(bs, 1H), 7.16(d, 2H), 7.34(d, 2H).

General Procedure for preparation 3-methyl-2-(phenylimino) thiazolidin-4-ones (3a-m)

To a solution of N-Phenyl N'-methylthiourea (2) (1 mmol) and ethyl bromoacetate in [bmIm] OH (1.2 mmol), and the resulting reaction mixture was stirred RT for 3h. After completion of the reaction (TLC check), cold water was added and extracted with Ethyl acetate (3 ×10 ml). The combined organic layers were washed with brine and dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was recrystallized using absolute ethanol to get regioisomer (3) 96%, Aqueous layer was re-extracted with ether (3 ×10 ml) to remove organic impurities. Aqueous layer was dried under vacuum at 90oC to get pure ionic liquid.

General Procedure for preparation 5-benzylidene-3-(3-fluoro-4-yl-morpholin-4-yl-phenylimino) thiazolidin-4-one derivatives (5a-g)

A mixture iminothiazolidin-4-one (3j) (1 mmol) and aldehyde (1.1 mmol) in [bmIm] OH (1.2 mmol) was stirred at rt. After 3h cold water was added and residue was filtered dried and recrystallized from hot ethanol to get pure 5-benzylidene-3-(3-fluoro-4-yl-morpholin-4-yl-phenylimino)-thiazolidin-4-one (5). Aqueous layer was re-extracted with ether (3 ×10 ml) to remove organic impurities and dried under vacuum at 90 ºC to get pure ionic liquid.

IV.SPECTROSCOPIC DATA

3-methyl-2-(phenylimino)thiazolidin-4-ones(3a)1HNMR:(CDCl3): δ 3.28 (s, 3H), 3.95 (s, 2H), 7.10 (d, 2H), 7.20 (d, 3H).;MS m/z 206.5 [M+H]+;IR:(KBr)1705 (C=O),1637(C=N) cm-1

3-ethyl-2-(phenylimino) thiazolidin-4-ones (3b) 1H NMR (CDCl3): δ 1.12(t J = 6.9 Hz,3H) 3.38(q, J = 6.9 Hz 2H), 3.97 (s, 2H), 7.10 (d, 2H), 7.20 (d, 3H).; MS m/z 221.2[M+H]+; IR:(KBr) 1705 (C=O),1637 (C=N) cm-1

3-propyl-2-(phenylimino) thiazolidin-4-ones (3c) 1H NMR (CDCl3): δ 1.01 (t, 3H), 1.78 (m, 2H), 3.82 (s, 2H), 3.87 (t, 2H), 7.15 (d, 2H)' 7.30 (m, 3H).; MS m/z 235.1[M+H]+; IR: (KBr): 1708 (C=O), 1635(C=N) cm-1

3-propyl-2-(3-fluoro-4-morpholin-4-yl-phenylimino)-thiazolidin-4-one (3d)1H NMR (CDCl3): δ 1.11(t,3H), 1.81(m, 2H),3.10-3.15(m,4H),3.78(m, 4H), 3.88(t, 2H) 4.01 (s, 2H), 6.70-6.81 (m, 2H), 6.95-7.10(m, 1H).; MS m/z 338.5 M+H]+ ;IR :(KBr) 1708 (C=O),1632(C=N) cm-1

3-ethyl-2-(3-fluoro-4-morpholin-4-yl-phenylimino)-thiazolidin-4-one(3e)1HNMR (CDCl3): 1.16 (t, J = 6.9 Hz, 3H), 2.97 (t, J = 3.4 Hz, 4H), 3.72-3.79 (m, 6H), 4.02 (s, 2H), 6.70- 6.82 (m, 2H), 6.97-7.06 (m, 1H);;MS m/z 324.5[M+H]+ IR:(KBr) 1709 (C=O),1630(C=N) cm-1

3-methyl-2-(3-fluoro-4-morpholin-4-yl-phenylimino)-thiazolidin-4-one (3f)1H NMR (CDCl3): δ 3.41(s, 3H), 2.98(m, 4H), 3.80 (m, 4H), 4.02 (s, 2H),6.79-6.90 (m, 2H), 7.10-7.15(m, 1H).; MS m/z 310.5 [M+H]+,IR: (KBr) 1715 (C=O),1630(C=N) cm-1

3-ethyl-2-(3-fluoro-4-dimethylamino-4-yl-phenylimino)-thiazolidin-4-one(3g)1H NMR (CDCl3): δ 1.10(t,3H),3.06(s, 6H), 3.68(t, 2H), 3.85 (s, 1H), 6.75-6.90(m, 2H), 7.20-7.28(m, 1H), MS m/z 282.5[M+H]+ ; IR:(KBr)1710 (C=O),1620 (C=N) cm-1

3-methyl -2-(4-(1-H-pyrol-1-ylphenylimino) thiazolidin-4-ones (3h) 1H NMR (CDCl3): δ 3.35 (s, 3H), 3.85 (s, 2H), 6.32 (m, 2H), 7.00-7.08 (m, 4H), 7.31 (d, 2H); MS m/z 272.6; [M+H]+ IR (KBr): 1708 (C=O), 1620 (C=N) cm-1

3-ethyl -2-(4-(1-H-pyrol-1-ylphenylimino) thiazolidin-4-ones (3i)1H NMR (CDCl3): δ 1.27(t J = 7.04 Hz,3H), 3.73(s, 2H), 3.91 (q J = 7.04 Hz, 2H), 6.34 (m, 2H), 7.00-7.08(m, 4H), 7.35(d,2H) MS m/z 286.6[M+H]+ ;IR: (KBr) 1715 (C=O),1625(C=N) cm-1

3-propyl -2-(4-(1-H-pyrol-1-ylphenylimino) thiazolidin-4-ones (3j)1H NMR (CDCl3): δ 1.27(t,3H), 1.80(m, 2H),3.85(s, 2H), 3.98 (q, 2H), 6.34 (m, 2H), 7.00-7.08(m, 4H), 7.35(d,2H) MS m/z 300.5[M+H]+ ;IR:(KBr) 1710 (C=O),1630(C=N) cm-1

3-ethyl-2-(3-fluoro-4-morpholin-4-yl-phenylimino)-5-methyl thiazolidin-4-one (3k) 1H NMR (CDCl3): δ 1.21(t,3H),1.52(d,3H),3.06(m, 4H), 2.98(m, 4H), 3.80 (m, 4H), 3.34 (t 2H),3.75(m,4H),3.99(m,1H) 6.68-6.76(m, 2H), 7.00-7.04(m, 1H), MS m/z 339.2[M+H]+ ;IR :(KBr) 1709 (C=O),1630(C=N). cm-1

3-ethyl-2-(4-(1-H-pyrol-1-ylphenylimino)-5-methyl thiazolidin-4-ones (3l) 1H NMR (CDCl3) δ: 1.27(t J = 7.1 Hz,3H), 1.62(d J = 7.9 Hz,3H),3.79(J = 7.1 Hz q, 2H), 4.02 (q, J = 7.9 Hz 1H), 6.42 (m, 2H), 7.12-7.20(m, 4H), 7.47(d,2H) MS m/z 300.2[M+H]+ ; IR: (KBr) 1715 (C=O),1625(C=N).cm-1

(5E)-3-Ethyl-2-(3-fluoro-4-morpholinophenylimino)-5-(4-ethoxybenzylidene)thiazolidin-4-one (5c): Yellow solid; mp: 138-140 0C; 1H NMR (CDCl3, 400MHz): δ 1.34 (t, J = 7.0 Hz, 3H), 3.10-3.14 (m, 4H), 3.83 (s, 3H), 3.90-4.00 (m, 4H), 4.03 (q, J =7.0 Hz, 2H), 6.75-6.82 (m, 2H), 6.94(d, J = 8.8 Hz, 2H), 6.98-7.10 (m, 1H), 7.41(d, J = 8.8 Hz, 2H), 7.71(s, 1H); MS (m/z) 442.5 [M+ +1]; IR (KBr): 2968, 2852, 2821, 1705, 1637, 1504, 1377, 1338, 1268, 1116, 1043, 923.

(5E)-5-((1H-indol-3-yl)methylene)-3-ethyl-2-(3-fluoro-4-morpholinophenylimino)thiazolidin-4-one (5g): Yellow solid; mp: 137-139 0C; 1H NMR (CDCl3, 400MHz): δ 1.36(t, J = 7.0 Hz, 3H), 3.11(t, J =4.4 Hz, 4H), 3.90(t, J =4.4 Hz, 4H), 4.05(q, J =7.0 Hz, 2H), 6.78-6.83(m, 2H), 6.96(t, J = 9.0 Hz, 1H), 7.28-7.32 (m, 2H), 7.42(d, J =7.6 Hz, 1H), 7.44(m, d, J =2.8 Hz, 1H), 7.85(d, J =7.6 Hz, 1H), 8.11(s, 1H), 8.62(bs, 1H, exchangeable with D2O); MS (m/z): 451.5 [M+ +1]; IR (KBr): 3403, 3174, 2955, 2891, 2837, 2700, 2360, 2251, 1701, 1633, 1602, 1114, 923.

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IV. CONFLICT OF INTEREST

Authors declared that they have no conflict of interest

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