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Microwave assisted one pot four-components solvent free base catalyzed rapid synthesis of (*Z*)-N-alkyl-5-arylidene-2-thioxothiazolidin-4-one derivatives

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Abstract: One pot multi-component of alkyl amine, chloroacetyl chloride, carbon disulfide and substituted aromatic aldehydes rapid process synthesis of known rhodanine derivatives (5a-r) under microwave irradiation method solvent free, using simple KOH as base. All the products were well characterized by the comparison of their spectral data such as IR, Mass and ^{1}H-NMR.

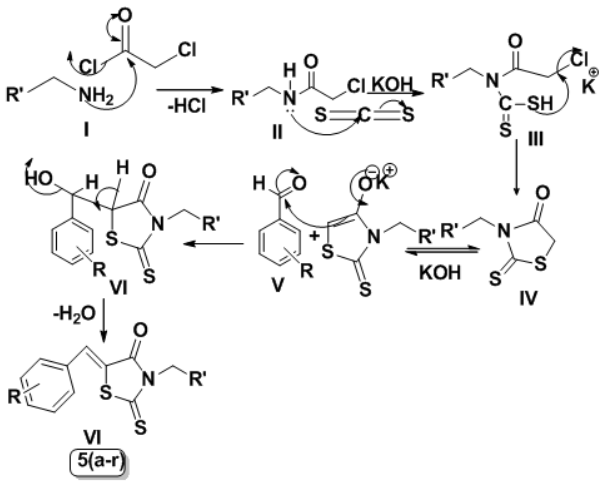
Keywords: Solvent free, Multi-component reactions, Microwave, Arylidene-2-thioxothiazolidin-4-one.

Introduction

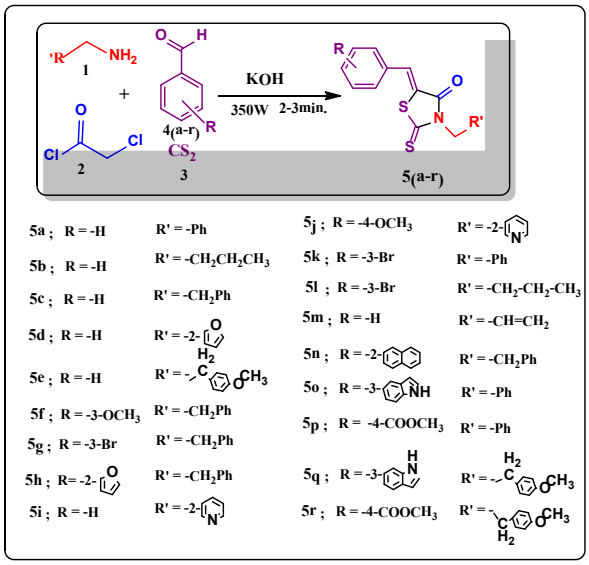
Many researcher's devote to multicomponent green approach reactions (MCGRs) are one of the most important methods for the synthesis of natural products, in medicinal and combinatorial chemistry [1-2], these reactions avoid time consuming and costly processes for the purification of various precursors and isolation of intermediates [3-4]. Multi-component reactions are often accompanied by significant increases in molecular complexity and remarkable selectivity [5-6]. The significance of microwave irradiation with use of catalysts or reagents, under solvent-free conditions in organic reactions to occur expeditiously at ambient pressure, thus providing exclusive chemical processes with special attributes such as enhanced reaction rates and higher yields and reduced the time compared to thermal reactions [7-16]. Sulfur-containing Rhodanine scaffold is bioactive natural products [17-21]. Which is used for the treatment of diabetic or therapy of AIDS [22-26], dyes for dye sensitized solar cells: Spectroscopy, energy levels and photovoltaic performance[27-29]. The previously synthesizedrhodanine derivatives such as (*Z*)-3-benzyl-5-benzylidene-2-thioxothiazolidin-4-

one [30-34]. However, some of these methods have disadvantages such as the employment of a hazardous and economically solvent or reagent and catalyst, time consuming process. The Biologically important arylidene rhodanine have been not reported solvent free, potassium hydroxide as base catalyzed under microwave irradiation technique, In continuation of our research interest to search of environmentally benign protocol [35-40]. Herein, we report a facile, mild and effective methodology under microwave irradiation method for the synthesis of (Z)-3-benzyl-5-benzylidene-2-thioxothiazolidin-4-one (Scheme **1**.).

Scheme 1. Synthesis of arylidene rhodanine



Scheme 2. The proposed mechanism for the synthesis of Rhodanine derivatives



Scheme and various substituent to rhodanine moiety**(5a-r)**

Results and discussion:

In this report, initially to optimize the conditions for this four-component reaction, alkylamine (1 mmol), substituted aromatic aldehyde (1 mmol), carbon disulfide (1.5 mmol), and chloroacetyl chloride (1 mmol) were used as model substrates. The effects of various reaction parameters, including the temperature, the additive, and the order of addition of the reactants were examined on the model reaction, and the results are summarized in **Table 1**. generally, the base played an important role for the synthesis of arylidene rhodanine derivatives, screening of various bases ($NaHCO_3$, NH_4CO_3, NH_4OAc, NaOEt, Et_3N, Pyridine, K_2CO_3, KOH), solvent free the model reaction revealed that potassium hydroxide was the most effective for microwave irradiation in terms of yield and time (**Table 1**. entry **8**),

Table 1. Optimization of base with respect to time and yield[b]

Entry	Base	Microwave irradiation method	
		Time (min)	Yield (%)
1.	$NaHCO_3$	4	38
2.	NH_4CO_3	4	20
3.	NH_4OAc	4	56
4.	NaOEt	4	15
5.	Et_3N	5	38
6.	Pyridine	5	15
7.	K_2CO_3	5	84
8.	**KOH**	**2**	**96**

[b]Condition: alkyl amine (1 mmol), carbon disulphide (3 mmol), chloroacetyl chloride (1 mmol) andKOH (3 mmol) and the aromatic aldehyde (1 mmol).

the reaction yields were not increased by the use of greater quantities of KOH, prolonged reaction times, it was found that the one pot, multi-component had a considerable effect on the yields. When the one-pot reaction of benzyl-amine (1.0 mmol), carbon disulfide (3 mmol), and chloroacetyl chloride (1 mmol) in microwave irradiation, followed by the co-addition of KOH (3.0 mmol) and benzaldehyde (1.0 mmol) in one portion, surprisingly, the reaction proceeded rapidly and gave better yield (96%) within 2-3 min (**Table 1**. entry no. 8) Thus, the optimum conditions for the four component for the synthesis of 5-arylidene rhodanine required the use of KOH (3 equiv), normal microwave irradiation technique. Using the optimized reaction conditions, the scope of these easily available starting of this four-component, reactions were investigate(**Table 1**). Herein, we observed base KOH catalyst under microwave irradiation gave better yield in very shorter reaction time (**Table 1.** and entry no. 5, **8.**).All examples were tested good to excellent yield (Table **2.**). Aromatic aldehydes with different functional

Table 2. Synthesis and physical data of rhodanine derivatives:

Entry	Product	Microwave Irradiation Method[a]		Melting point (^{0}C)
		Yield[b](%)	Time(min)	
1	**5a**	**96**	**2**	159-160
2	**5b**	78	3	116-117
3	**5c**	85	3	126-128
4	**5d**	**89**	3	136-137
5	**5e**	94	**2**	149-150
6	**5f**	84	3	124-126
7	**5g**	87	3	101-103
8	**5h**	79	3	149-151
9	**5i**	86	3	153-155
10	**5j**	86	3	152-154
11	**5k**	85	3	113-114
12	**5l**	78	3	112-115
13	**5m**	79	3	142-143
14	**5n**	83	3	175-177
15	**5o**	84	3	152-155
16	**5p**	86	3	163-165
17	**5q**	88	3	239-241
18	**5r**	89	3	202-205

[a]Reaction condition alkyl amine (1 mmol), carbon disulphide (3 mmol), chloroacetyl chloride (1 mmol) andKOH (3 mmol) and the aromatic aldehyde (1 mmol) wereadded in two different reaction condition,[b]Isolated yield.

groups such as chloro, bromo, ester, or methoxy were applied in the reaction, the little electronic effects was influence on the aldehydes on the yield of product while benzyl amine with electron-donating groups reacted rapidly, and gave good yield (**Table 2**. entry **5**.17,18) and un-substituted aryl amine gave better yield (Table **2**. Entry **1**) than substituted electron withdrawing group to the benzyl

amine, in addition, 2-furaldehyde and indole 3-carbaldehyde also gave moderate yields of products (Table **2**. Entry **8,15**), analogously, the reaction was also general with respect to the aryl aldehydes substrates, while various aliphatic primary amines such as 2-phenylethylamine, n-propylamine, and n-butylamine reacted to give the corresponding yields (Table **2**. Entry **2, 12, 13**). It is worth noting that 2-furyl and 2-pyridyl amine also gave the desired products in moderate yields (Table **2**. Entry **4, 9, 10**). On the basis of the results obtained from (Table **2**.) and according to proposed mechanism in Scheme **2**. In all the cases, the rhodanine derivatives precipitated under solvent free were obtained in excellent yields after recrystallization from ethanol, no side products were observed after reaction workup; thus we believed that the present methodology opens new possibilities for the synthesis of rhodanine derivatives, the completion of reaction was confirmed by the disappearance of the starting on TLC and the structure of compounds (**5**) were substantiated by IR, ^{1}HNMR and CHN analysis, thermodynamically more stable *Z*-isomer were obtained[41].The IR spectrum of representative compound(*Z*)-3-benzyl-5-benzylidene-2 thioxothiazolidin-4-one, shows a strong absorption band at the region 1690-1705 cm^{-1}which is due to a carbonyl group thiazolidinone moiety.^{1}HNMR spectra of compound **5** show one singlet for the methylene proton*(2H)*in the range δ 4.96-4.99 ppm and another one signal for the methylene proton*(1H)* in the range δ 7.78-7.82 ppm at lower field values than those expected for the *E*- isomer. This strongly indicated that compound have *Z*-configuration.

Experimental:

Chemistry

The products were purified by simple filtration and recrystallization from ethanol, the reaction progress monitor by thin layer chromatography The melting points of the compounds were determined in open head capillary and are uncorrected. The FT-IR spectra were provided on Bruker Avance 100 MHz instruments, ^{1}H-NMR were recorded on a 300 MHz, NMR instrument Bruker (Avance) $CDCl_3$ as solvent. Elemental analyses were performed on a Perkin Elmer 2400 Series II Elemental CHNS analyzer. Microwave reactions have been carried out in a Biotage Microwave synthesizer. All the product are well characterized by the comparison of their spectral data (IR, ^{1}H-NMR, physical data, melting point etc. with those reported in literature)[30-34]

*General method for the preparation of **5***

Microwave irradiation: A cold solution of chloroacetyl chloride (1 mmol) was dropwise added to equimolar amount of mixture of alkyl amine (1 mmol) and carbon disulphide (3 mmol), stirred to obtained homogenous mixture, were subjected to microwave irradiation (350W, 100^{0}C) for 50-60 sec. then KOH (3 mmol) and the aromatic aldehyde (1 mmol) were added to the mixture,were further microwave irradiated (350 W) for further 1-2 min. The progress reaction was monitored by TLC, after completion of the reaction, mixture was allowed to stand for 5-10 min., Water (5 mL) was added and the product precipitated immediately. The solid products were washed with water and filtered and the crude rhodanine derivatives were purified by recrystallized from hot ethanol to afford the pure product, yield (78-96%).

Spectral Characterization data:

***(Z)-3-benzyl-5-benzylidene-2-thioxothiazolidin-4-one* (5a):** Yield: 96%; m.p. 159-160^{0}C;Anal. Calcd for $C_{17}H_{13}NOS_2$: C, 65.56; H, 4.21; N, 4.50 %, Found: C, 65.71; H, 3.99; N, 4.80; IR (KBr,cm^1):1696 (CONH),1542

(Ar), 1612(C=C), 1180 (C=S), 1011 (C-S);^{1}H NMR (100 MHz, $CDCl_3$, δ / ppm):4.93 (2H, s), 7.21 (2H, d), 7.32 (2H, t), 7.19 (1H, t), 7.80 (1H, S), 7.58 (2H, d), 7.35 (2H, t), 7.29 (1H, d); MS (m/z, %): 311.04 M^+

(Z)-5-benzylidene-3-butyl-2-thioxothiazolidin-4-one(5b):Yield: 78%; m.p. 116-117^{0}C;Anal. Calcd for $C_{14}H_{15}NOS_2$: C, 60.62; H, 5.45; N, 5.05 %, Found: C, 60.69; H, 5.27; N, 5.23; IR (KBr,cm^1):1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S);^{1}H NMR (100 MHz, $CDCl_3$,δ /ppm):7.30-7.60(5H, m), 7.78 (1H, s), 4.02 (2H, t), 1.50 (2H, m), 1.30 (2H, sext), 0.92(3H, t) ; MS (EI): m/z (%) : 277.05, M^+

(Z)-5-benzylidene-3-phenethyl-2-thioxothiazolidin-4-one(5c):Yield: 85%; m.p: 126-128^{0}C;Anal. Calcd for $C_{18}H_{15}NOS_2$; C, 66.43; H, 4.65; N, 4.30%,Found: C, 66.63; H, 4.51; N, 4.43; IR (KBr,cm^1) :1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S).;^{1}H NMR (100 MHz, $CDCl_3$, δ / ppm):7.30-7.60 (5H, m), 7.82 (1H, s), 3.15 (2H, t), 2.80 (2H, t),7.28-7.40(5H, m); MS (EI): m/z (%) :325.05, M^+

(Z)-5-benzylidene-3-(furan-2-ylmethyl)-2-thioxothiazolidin-4-one(5d):Yield: 89%; m.p: 136-137^{0}C;Anal. Calcd for $C_{15}H_{11}NO_2S_2$; C, 59.78; H, 3.68; N, 4.65%,Found: C, 62.69; H, 3.78; N, 20.85; IR (KBr,cm^1) :1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S), 1033 (C-O);^{1}H NMR (100 MHz, $CDCl_3$, δ / ppm):7.32-7.60 (5H, m), 7.81 (1H, s), 4.04 (2H, s), 6.25 (1H, d),6.45 (1H, t), 7.62 (1H, d); MS (EI): m/z (%) : 301.38, M^+

(Z)-5-benzylidene-3-(4-methoxyphenethyl)-2-thioxothiazolidin-4-one(5e):Yield: 94%; m.p: 149-150^{0}C;Anal. Calcdfor:$C_{19}H_{17}NO_2S_2$; C, 64.20; H, 4.82; N, 3.94%,Found: C, 64.31; H, 4.79; N, 4.04; IR (KBr,cm^1) :1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S), 1033 (C-O);^{1}H NMR (100 MHz, $CDCl_3$, δ /ppm):7.32-7.60 (5H, m), 7.81 (1H,s),3.16 (2H, t), 2.81 (2H, t),7.15 (2H, d), 6.92 (2H, d),3.18(3H, s); MS (EI): m/z (%), 355.05 M^+

***(Z)-5-(3-methoxybenzylidene)-3-phenethyl-2-thioxothiazolidin-4-one(5f)*:**Yield: 84%; m.p: 124-126^{0}C;Anal. Calcd for $C_{19}H_{17}NO_2S_2$; C, 64.20; H, 4.82; N, 3.94%,Found: C, 64.25; H, 4.72; N, 3.98; IR (KBr,cm^1) :1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S), 1033 (C-O);^{1}H NMR (100 MHz, $CDCl_3$, δ / ppm):7.25-7.45 (5H, m), 2.81 (2H,t), 3.16 (2H, t), 7.81 (1H, s),7.60 (1H, s), 7.54 (1H,d),7.28(1H, t), 7.49(1H, d); MS (EI): m/z (%) : 355.05 M^+

***(Z)-5-(3-bromobenzylidene)-3-phenethyl-2-thioxothiazolidin-4-one (5g*):**Yield: 86%; m.p: 101-103^{0}C;Anal. Calcd for $C_{18}H_{14}BrNOS_2$; C, 53.47; H, 3.49; N, 3.46%, Found: C, 53.59; H, 3.39; N, 3.79; IR (KBr,cm^1) :1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S), 710 (C-X);^{1}H NMR (100 MHz, $CDCl_3$, δ / ppm):7.25-7.45 (5H, m), 3.81 (2H, t), 3.16 (2H, t), 7.81 (1H, s),7.15 (2H, d), 7.60 (1H, t), 3.82 (3H, s); MS (EI): m/z (%) : 402.34 M^+

***(Z)-5-(furan-2-ylmethylene)-3-phenethyl-2-thioxothiazolidin-4-one(5h)*:**Yield: 79%; m.p: 149-151^{0}C;Anal. Calcd for $C_{16}H_{13}NO_2S_2$; C, 60.93; H, 4.15; N, 4.44%, Found: C, 70.00; H, 4.01; N, 4.59; IR (KBr,cm^1) :1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S), 1033 (C-O); ^{1}H NMR (100 MHz, $CDCl_3$, δ / ppm):7.25-7.42(5H, m), 2.81 (2H, t), 3.16 (2H, t), 7.34 (1H, s),7.64 (1H, d), 6.84 (1H, t), 8.12 (1H, d); MS (EI): m/z (%) : 315.03 M^+

(Z)-5-benzylidene-3-(pyridin-2-ylmethyl)-2-thioxothiazolidin-4-one(5i):Yield: 86%; m.p: 153-155^{0}C;Anal. Calcd for $C_{16}H_{12}N_2OS_2$; C, 61.51; H, 3.87; N, 8.97%,Found: C, 61.61; H, 3.67; N, 9.15; IR (KBr,cm^1) :1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S); ^{1}H NMR (100 MHz, $CDCl_3$, δ /ppm):7.30-

7.62(5H, m), 7.78 (1H, s), 4.56 (2H, s), 7.34 (1H, d),7.72 (1H, t), 7.32 (1H, t), 8.44 (1H, d); MS (EI): m/z (%) : 312.03 M^+

***(Z)-5-(4-methoxybenzylidene)-3-(pyridin-2-ylmethyl)-2-thioxothiazolidin-4-one(5j)*:**Yield: 86%; m.p: 152-154^{0}C;Anal. Calcd for $C_{17}H_{14}N_2O2S_2$; C, 59.63; H, 4.21; N, 8.18%, Found: C, 59.67; H, 4.03; N, 8.29; IR (KBr,cm^1) :1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S), 1037 (C-O); ^{1}H NMR (100 MHz, $CDCl_3$, δ /ppm): 3.84(3H, s), 6.91 (2H, d), 7.58 (2H, d), 7.79 (1H, s), 4.58 (2H, s), 7.31 (1H, d),7.72 (1H, t), 7.32 (1H, t), 8.43 (1H, d); MS (EI): m/z (%) : 342.04 M^+

(Z)-3-benzyl-5-(3-bromobenzylidene)-2-thioxothiazolidin-4-one(5k):Yield:85%;m.p:113-114-116^{0}C;Anal. Calcd for $C_{17}H_{12}BrNOS_2$; C, 52.31; H, 3.10; N, 3.59%, Found: C, 52.51; H, 2.99; N, 3.78; IR (KBr,cm^1) :1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S), 725 (C-X); ^{1}H NMR (100 MHz, $CDCl_3$, δ /ppm):7.20-7.32(5H, m), 4.96 (2H, s), 7.81 (1H, s), 7.60 (1H, s),7.55 (1H, d), 7.30 (1H, t), 7.50 (1H, d); MS (EI): m/z (%) : 388.95 M^+

(Z)-5-(3-bromobenzylidene)-3-butyl-2-thioxothiazolidin-4 one(5l):Yield:78%;m.p:112115^{0}C;Anal. Calcd for $C_{14}H_{14}BrNOS_2$;C, 47.19; H, 3.96; N, 3.93%,Found: C, 47.29; H, 3.91; N, 4.01; IR (KBr,cm^1) : 1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S), 721 (C-X); ^{1}H NMR (100 MHz, $CDCl_3$, δ /ppm):7.60(1H, s), 7.46 (1H, d), 7.30 (1H, t), 7.55 (1H, d),7.81 (1H, s), 4.02 (2H, t), 7.50 (2H, m), 1.30 (2H, q), 0.89 (3H, t); MS (EI): m/z (%) : 354.97 M^+

***(Z)-3-allyl-5-benzylidene-2-thioxothiazolidin-4-one(5m)*:**Yield:79%;m.p:142-143^{0}C;Anal. Calcd for $C_{13}H_{11}NOS_2$; C, 59.74; H, 4.24; N, 5.36%, Found: C, 59.89; H, 4.20; N, 5.46; IR (KBr,cm^1) :1696 (CONH), 1540(Ar), 1612(C=C), 1180 (C=S), 1011(C-S); ^{1}H NMR (100 MHz, $CDCl_3$, δ /ppm):7.30-7.62 (5H, m), 7.81 (1H, s), 4.68 (2H, d), 5.88 (1H, d),5.18-5.72 (2H, d); MS (EI): m/z (%) : 261.028 M^+

(Z)-5-(naphthalen-2-ylmethylene)-3-phenethyl-2-thioxothiazolidin-4-one(5n):Yield:83%;m.p: 175-177^{0}C;Anal. Calcd for $C_{22}H_{17}NOS_2$; C, 70.37; H, 4.54; N, 3.73%, Found: C, 70.50; H, 4.34; N, 3.83; IR (KBr,cm^1) :1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S); ^{1}H NMR (100 MHz, $CDCl_3$, δ /ppm) :7.25-7.40 (5H, m), 2.15 (2H, t), 2.80 (2H, t), 7.81 (1H, s),7.49 (1H, d), 7.76 (1H, d), 7.82 (1H, s), 8.01 (2H, d), 7.60 (1H, t); MS (EI): m/z (%) : 375.07 M^+

(Z)-5-((1H-indol-3-yl)methylene)-3-benzyl-2-thioxothiazolidin-4-one(5o):Yield:84%; m.p: 152-155^{0}C;
Anal.Calcd for $C_{19}H_{14}N_2OS_2$;C, 65.12; H, 4.03; N, 7.99%,Found: C, 65.22; H, 4.23; N, 8.02; IR (KBr,cm^1) :3120 (N-H), 1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S); ^{1}H NMR (100 MHz, $CDCl_3$, δ /ppm):7.22-7.35(5H,m), 4.96 (1H, s), 7.80 (1H, s), 7.75 (1H, s),10.01 (1H, s), 7.64 (1H, d), 7.18 (1H, t), 6.95 (1H, t), 7.12 (1H, d); MS (EI): m/z (%) : 350.05 M^+

(Z)-methyl4-((3-benzyl-4-oxo-2-thioxothiazolidin-5-ylidene)methyl)benzoate (5p):Yield:86%; m.p: 163-165^{0}C;Anal. Calcd for $C_{19}H_{15}NO_3S_2$; C, 61.77; H, 4.09; N, 3.79%, Found: C, 61.87; H, 4.00; N, 3.89; IR (KBr,cm^1) : 1734 ($COOCH_3$),1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1041 (C-O), 1011(C-S); ^{1}H NMR (100 MHz, $CDCl_3$, δ /ppm):7.20-7.35 (5H, m), 4.95 (1H, s), 7.81 (1H, s), 747 (2H, d),7.68 (2H, d), 3.90 (3H, s); MS (EI): m/z (%) : 369.04 M^+

(Z)-5-((1H-indol-3-yl)methylene)-3-(4-methoxyphenethyl)-2-thioxothiazolidin-4-one (5q):Yield:88%; m.p: 239-241^{0}C;Anal.

Calcd for $C_{21}H_{18}N_2O_2S_2$; C, 63.93; H, 4.60; N, 7.10%,Found: C, 64.00; H, 4.50; N, 7.23; IR (KBr,cm^1) : 3120 (N-H), 1696 (CONH), 1540 (Ar), 1612(C=C), 1180 (C=S), 1011(C-S); ^{1}H NMR (100 MHz, $CDCl_3$, δ /ppm):7.10-7.99(5H, m), 7.78(1H, s), 7.82 (1H, s), 3.18 (2H, t), 2.85 (2H, t),7.19 (2H, d), 6.94 (2H, d), 3.85 (3H, s); MS (EI): m/z (%) : 394.08 M^+

(Z)-methyl4-((3-(4-methoxyphenethyl)-4-oxo-2-thioxothiazolidin-5ylidene)methyl)benzoate (5r):Yield: 89%; m.p: 202-205^{0}C;Anal. Calcd for $C_{21}H_{19}NO_4S_2$; C, 61.00; H, 4.63; N%, Found: C, 61.12; H, 4.53; N, 3.47; IR (KBr,cm^1):1739 ($COOCH_3$), 1711 (CONH), 1609 (Ar), 1610(C=C), 1180 (C=S), 1041 (C-O), 1011(C-S);^{1}H NMR (100 MHz, $CDCl_3$, δ /ppm):3.90(3H, s), 7.67(2H, d), 7.51 (2H, d), 7.81 (1H, s), 3.18 (2H, t), 2.82 (2H, t), 7.20(2H, d),6.95(2H, d),3.92(3H, s),MS (EI): m/z (%) : 413.07 M^+

Conclusion:

In conclusion, we have successfully developed an easy access to reported series of (*Z*)-3-benzyl-5-benzylidene-2-thioxothiazolidin-4-one derivatives (**5a-r**), by microwave irradiation technique, good to excellent yield in very less time of reaction, easy workup, and cheap starting materials, avoiding hazardous organic solvents are significant features of these methods.An effort toward the synthesis of other important drug molecules with a Rhodanine moiety by conventional and non-conventional method is ongoing in our laboratory.

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