

The reaction of carbon disulphide with α -haloketones and primary amines in the presence of potassium iodide as catalyst

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Abstract. A simple, mild and convenient method has been developed for the synthesis of 3,4,5-trialkyl-1,3-thiazole-2(3*H*)-thione derivatives through one pot three-component reaction between a primary amine, carbon disulphide, and α -haloketone in the presence of potassium iodide at room temperature conditions. The products were obtained with excellent yield and appropriate reaction times. This reaction represents a rapid and unprecedented route to the described molecules that have biological specifications.

Keywords. carbon disulphide; α -haloketone; primary amines; potassium iodide; thiazole; nucleophilic addition.

1. Introduction

Multicomponent reactions (MCR) have appeared as an efficient and powerful tool in modern synthetic organic chemistry due to their valued features such as atom economy, straightforward reaction design, and the opportunity to construct target compounds by the introduction of several diversity elements in a single chemical event. Since all the organic reagents employed are consumed and incorporated into the target compound, purification of products resulting from MCR is also simple.¹ MCR, leading to interesting heterocyclic scaffolds, are especially useful for the construction of diverse chemical libraries of 'druglike' molecules. The isocyanide-based MCRs are very important in this area.^{2–4} Among the known multicomponent reactions to date, the most valuable reactions are those based on isocyanides. Isocyanide-based multicomponent reactions (abbreviated to IMCRs by Ugi and Dömling) by virtue of their synthetic potential, their inherent atom efficiency, convergent nature, ease of implementation, and the generation of molecular diversity, have attracted considerable attention because of the advantages that they offer to the field of combinatorial chemistry.^{5–9}

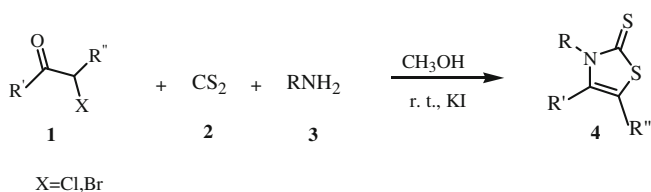
Organic compounds containing five-membered aromatic heterocyclic rings constitute a wide range of compounds in the nature and often play an important role in various biochemical processes. As a result, aromatic heterocycles such as thiophenes, benzothiophene

derivatives, and their reduced forms are significant structural fragments in many pharmaceutical and chemical compounds. Thiazoline and thiazole compounds have been found to represent nematocidal, insecticidal, antibacterial, antifungal, antiviral, and antioxidant activity.^{10–15} Tetrahydrothiophene is an important building block of a large quantity of compounds that are very interesting from the viewpoint of biological activity. Its derivatives have showed antisecretory and antiulcer activities.¹⁶ Because of the potential use of thiophenes and annulated thiophenes as pharmaceuticals, conjugated polymers, organic conductors, semiconductors and light emitting devices, their synthesis are of special interest.^{17–22} Alkylation of an intermediate dimethylaloketene dithioacetal is a general strategy employed for the synthesis of alkylthiophenes or thienothiophenes.^{23–26} As part of our ongoing program to develop efficient and robust methods for the preparation of heterocyclic compounds,^{27–39} we have prepared 3-alkyl-4-phenyl-1,3-thiazole-2(3*H*)-thionewithout catalyst in our last report⁴⁰ and now we wish to report the preparation of a 3, 4, 5- trialkyl-1, 3- thiazole- 2 (3*H*)- thione derivatives through a one-pot multicomponent condensation reaction (MCR) in the presence of potassium iodide (scheme 1).

2. Experimental

Starting materials and solvents were obtained from Merck (Germany) and Fluka (Switzerland) and were

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Scheme 1. Three-component synthesis of 3,4,5-trialkyl-1,3-thiazole-2(3H)-thione derivatives **4** in the presence of potassium iodide.

used without further purification. TLC and NMR spectroscopy were used to follow the reactions. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were measured on a Jasco 6300 FTIR spectrometer. ^1H and ^{13}C NMR spectra were measured (CDCl_3 solution) with a BRUKER DRX-250 Avance spectrometer at 250.0 and 62.5 MHz, respectively. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer. Preparative layer chromatography (PLC) plates were prepared from Merck silica gel (F_{254}) powder.

2.1 General procedure for the preparation of compounds **4**

The mixture of α -haloketone **1** (1 mmol) and potassium iodide 0.1 g (0.6 mmol) in CH_3OH (7 ml) was stirred for 30 min and then the solution of primary amine (1 mmol) and carbon disulphide (1 mmol) was added, and the mixture was stirred for 24 h. The solvent was removed under reduced pressure and the viscous residue was purified by preparative layer chromatography (PLC) [silica gel (F_{254}) powder; petroleum ether-ethyl acetate (10:2)]. The solvent was removed under reduced pressure and the products were obtained. The characterization data of the compounds are given below.

2.1a 3-(2-Furylmethyl)-4,5-diphenyl-1,3-thiazole-2(3H)-thione (4a): White powder, (yield: 85%). IR (neat): $\nu = 3150, 3000, 1625, 1600, 1475, 1300, 1100\text{ cm}^{-1}$. ^1H NMR (250 MHz, CDCl_3) δ (ppm): 5.34 (s, 2H, CH_2), 6.12 (s, 1H, CH of furan), 6.24 (s, 1H, CH of furan), 7.00–7.47 (m, 11H, CH_{arom} and CH of furan). ^{13}C NMR (62.5 MHz, CDCl_3) δ (ppm): 44.66 (CH_2), 109.59, 110.47 (CH of furan), 125.00 (C of alkene), 128.02, 128.23, 128.64, 129.10, 130.00, 130.87 (10CH), 130.05, 130.19 (C), 138.63 (C of alkene), 142.04 (CH of furan), 147.94 (C of furan), 186.80 (C=S). MS, m/z (%): 349 (80), 316 (48), 210 (52), 171 (80), 151 (86), 105 (84), 81 (100), 69 (40), 53 (48). Analysis of $\text{C}_{20}\text{H}_{15}\text{NOS}_2$ (349.47). (% calculation/found): C: 68.74/68.80, H: 4.33/4.39, N: 4.01/3.95.

2.1b 3-Benzyl-4-(4-methoxyphenyl)-1,3-thiazole-2(3H)-thione (4b): White powder, (yield: 87%). IR (neat): $\nu = 3150, 3000, 1625, 1600, 1475, 1300, 1100\text{ cm}^{-1}$. ^1H NMR (250 MHz, CDCl_3) δ (ppm): 3.82 (s, 3H, OCH_3), 5.39 (s, 2H, CH_2), 6.45 (s, 1H, CH of alkene), 6.82–7.21 (m, 9H, CH_{arom}). ^{13}C NMR (62.5 MHz, CDCl_3) δ (ppm): 50.82 (CH_2), 55.40 (OCH_3), 108.65 (CH of alkene), 114.01, 126.98, 127.52, 128.49, 130.89 (9CH), 122.79, 135.42, 160.67 (3C), 144.75 (C of alkene), 188.86 (C=S). MS, m/z (%): 313 (56), 297 (80), 280 (36), 229 (44), 206 (72), 149 (44), 135 (64), 91 (92), 69 (100), 57 (56). Analysis of $\text{C}_{17}\text{H}_{15}\text{NOS}_2$ (313.44). (% calculation/found): C: 65.14/65.08, H: 4.82/4.88, N: 4.47/4.41.

2.1c 4-(4-Methoxyphenyl)-3-(4-methylbenzyl)-1,3-thiazole-2(3H)-thione (4c): White powder, (yield: 90%). IR (neat): $\nu = 3100, 3000, 1650, 1600, 1475, 1200\text{ cm}^{-1}$. ^1H NMR (250 MHz, CDCl_3) δ (ppm): 2.29 (s, 3H, CH_3), 3.82 (s, 3H, OCH_3), 4.82 (s, 2H, CH_2), 5.94 (s, 1H, CH of alkene), 6.84–7.26 (m, 8H, CH arom). ^{13}C NMR (62.5 MHz, CDCl_3) δ (ppm): 21.10 (CH_3), 46.95 (CH_2), 55.36 (OCH_3), 98.28 (CH of alkene), 113.94, 127.07, 129.19, 130.53 (8CH), 123.77, 133.59, 137.09, 160.29 (4C), 137.61 (C of alkene), 172.85 (C=S). Analysis of $\text{C}_{18}\text{H}_{17}\text{NOS}_2$ (327.46). (% calculation/found): C: 66.02/66.06, H: 5.23/5.17, N: 4.28/4.22.

2.1d 3-(2-Furylmethyl)-4-(4-methoxyphenyl)-1,3-thiazole-2(3H)-thione (4d): White powder, (yield: 94%). IR (neat): $\nu = 3150, 3000, 1625, 1600, 1475, 1300, 1100\text{ cm}^{-1}$. ^1H NMR (250 MHz, CDCl_3) δ (ppm): 3.85 (s, 3H, OCH_3), 4.80 (s, 2H, CH_2), 5.95 (s, 1H, CH of alkene), 6.03, 6.24 (s, 2CH, CH of furan), 6.92–7.29 (m, 5H, CH_{arom} and CH of furan). ^{13}C NMR (62.5 MHz, CDCl_3) δ (ppm): 40.48 (CH_2), 55.38 (OCH_3), 98.32, (CH of alkene), 108.60, 110.44, 142.21 (3CH of furan), 114.04, 130.61 (4CH), 123.50, 160.37 (2C), 137.22 (C of alkene), 149.36 (C of furan), 172.55 (C=S). Analysis of $\text{C}_{15}\text{H}_{13}\text{NO}_2\text{S}_2$ (303.40). (% calculation/found): C: 59.38/59.32, H: 4.32/4.26, N: 4.62/4.56.

2.1e 3-(2-Methoxybenzyl)-4-(4-methoxyphenyl)-1,3-thiazole-2(3H)-thione (4e): White powder, (yield: 87%). IR (neat): $\nu = 3100, 3000, 1600, 1475, 1200\text{ cm}^{-1}$. ^1H NMR (250 MHz, CDCl_3) δ (ppm): 3.67, 3.79 (2s, 6H, 2OCH_3), 4.87 (s, 2H, CH_2), 5.99 (s, 1H, CH of alkene), 6.74–7.22 (m, 8H, CH arom). ^{13}C NMR (62.5 MHz, CDCl_3) δ (ppm): 42.72 (CH_2), 55.12, 55.31 (2OCH_3), 98.07 (CH of alkene), 109.94, 113.85, 120.55, 126.78, 128.30, 130.08 (8CH), 123.81,

124.64, 156.31, 160.14 (4C), 137.99 (C of alkene), 172.79 (C=S). Analysis of $C_{18}H_{17}NO_2S_2$ (343.46). (% calculation/found): C: 62.94/62.88, H: 4.99/4.93, N: 4.08/4.02.

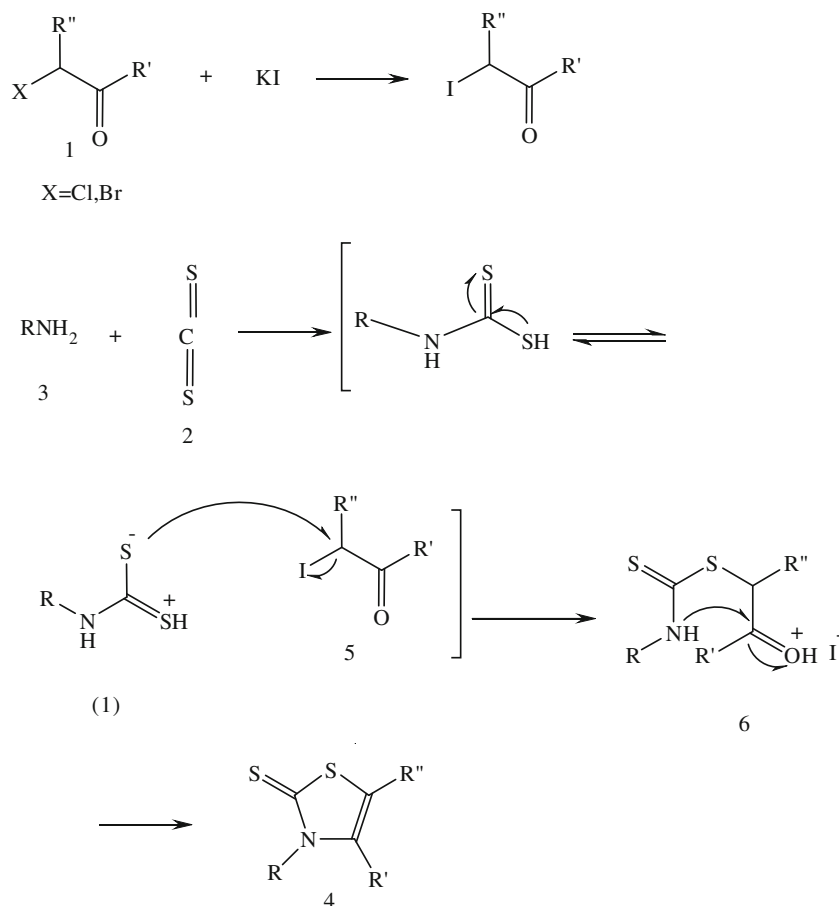
2.1f 3-(2-Methoxybenzyl)-4-methyl-1,3-thiazole-2(3H)-thione (**4f**): White powder, (yield: 88%). IR (neat): $\nu = 3100, 3000, 1600, 1475, 1200\text{ cm}^{-1}$. ^1H NMR (250 MHz, CDCl_3) δ (ppm): 2.01(s, 3H, CH_3), 3.86 (s, 3H, OCH_3), 4.95 (s, 2H, CH_2), 5.78 (s, 1H, CH of alkene), 6.85–6.90 (m, 4H, CH_{arom}). ^{13}C NMR (62.5 MHz, CDCl_3) δ (ppm): 15.12(CH_3), 41.21 (CH_2), 55.24(OCH_3), 97.48 (CH of alkene), 110.11, 120.79, 126.61, 128.55 (4CH), 126.50, 158.61 (2C), 134.46 (C of alkene), 185.52 (C=S). Analysis of $C_{12}H_{13}NOS_2$ (251.37). (% calculation/found): C: 57.34/57.40, H: 5.21/5.15, N: 5.57/5.51.

2.1g 3-(2-Methoxybenzyl)-4,5-dimethyl-1,3-thiazole-2(3H)-thione (**4g**): White powder, (yield: 90%). IR (neat): $\nu = 3100, 3000, 1600, 1475, 1200\text{ cm}^{-1}$. ^1H NMR (250 MHz, CDCl_3) δ (ppm): 1.90, 2.09 (2s, 6H, 2CH_3), 3.85 (s, 3H, OCH_3), 4.91 (s, 2H, CH_2),

6.79–7.21 (m, 4H, CH_{arom}). ^{13}C NMR (62.5 MHz, CDCl_3) δ (ppm): 11.22 (CH_3), 12.12 (CH_3), 41.44 (CH_2), 55.29 (OCH_3), 108.28 (C of alkene), 110.06, 120.78, 126.61, 128.40 (4CH), 124.50, 158.61 (C), 142.46 (C of alkene), 171.92 (C=S). Analysis of $C_{13}H_{15}NOS_2$ (265.39). (% calculation/found): C: 58.83/58.77, H: 5.70/5.76, N: 5.28/5.22.

2.1h 3-(2-Furylmethyl)-4,5-dimethyl-1,3-thiazole-2(3H)-thione (**4h**): White powder, (yield: 92%). IR (neat): $\nu = 3100, 3000, 1600, 1475, 1200\text{ cm}^{-1}$. ^1H NMR (250 MHz, CDCl_3) δ (ppm): 2.12, 2.28 (2s, 6H, 2CH_3), 5.40 (s, 2H, CH_2), 6.32, 6.47, 7.33 (3CH of furan). ^{13}C NMR (62.5 MHz, CDCl_3) δ (ppm): 11.63 (CH_3), 12.78 (CH_3), 43.75 (CH_2), 110.05, 110.74, 142.30 (3CH of furan), 117.32, 134.52 (2C of alkene), 148.17 (C of furan), 186.92 (C=S). Analysis of $C_{10}H_{11}NOS_2$ (225.33). (% calculation/found): C: 53.30/53.36, H: 4.92/4.98, N: 6.22/6.28.

2.1i 4,5-Dimethyl-3-(4-methylbenzyl)-1,3-thiazole-2(3H)-thione (**4i**): White powder, (yield: 90%). IR (neat): $\nu = 3100, 3000, 1600, 1475, 1200\text{ cm}^{-1}$.



Scheme 2. Proposed mechanism for the formation of 3,4,5-trialkyl-1,3-thiazole-2(3H)-thione derivatives **4** in the presence of potassium iodide.

Table 1. Synthesis of 3,4,5-trialkyl-1,3-thiazole-2(3*H*)-thione derivatives **4a–i** in the presence of potassium iodide.

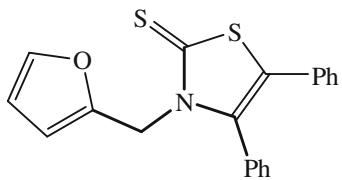
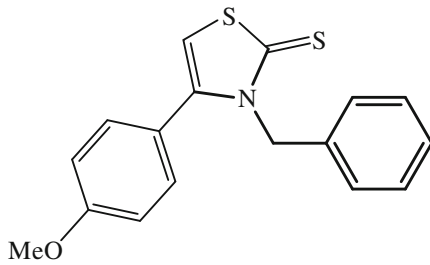
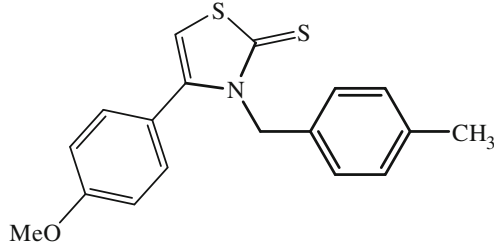
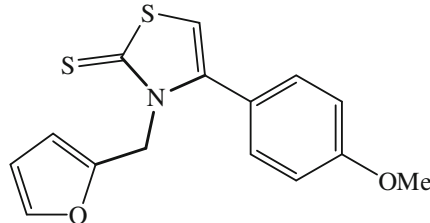
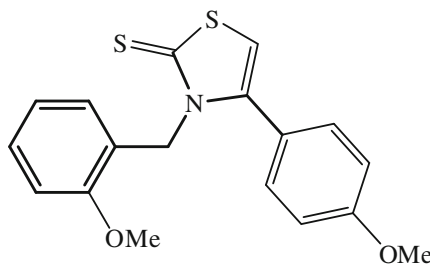
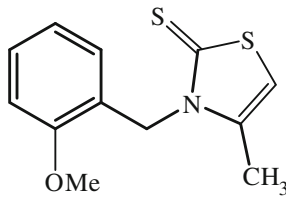
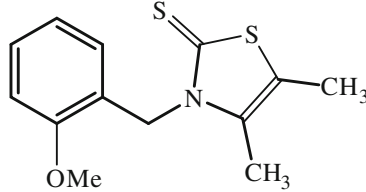
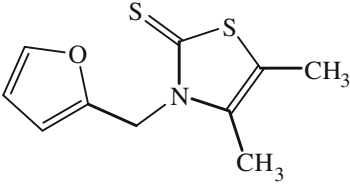
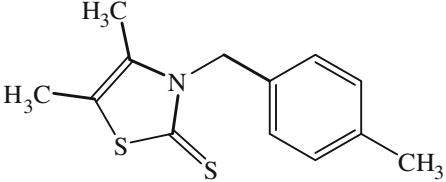
Entry	R	R'	R''	Product	Yield (%)
4a	2-Furylmethyl	Phenyl	Phenyl		92
4b	Benzyl	4-Methoxy phenyl	H		93
4c	4-Methyl benzyl	4-Methoxy phenyl	H		94
4d	2-Furylmethyl	4-Methoxy phenyl	H		95
4e	2-Methoxy benzyl	4-Methoxy phenyl	H		90
4f	2-Methoxy benzyl	Methyl	H		90
4g	2-Methoxy benzyl	Methyl	Methyl		92

Table 1. (continued).

Entry	R	R'	R''	Product	Yield (%)
4h	2-Furylmethyl	Methyl	Methyl		91
4i	4-Methyl benzyl	Methyl	Methyl		89

^1H NMR (250 MHz, CDCl_3) δ (ppm): 2.04, 2.12, 2.31 (s, 9H, 3 CH_3), 5.47 (s, 2H, CH_2), 7.11 (s, 4H, CH_{arom}). ^{13}C NMR (62.5 MHz, CDCl_3) δ (ppm): 11.58, 12.87, 21.00 (3 CH_3), 50.53 (CH_2), 95.28 (C of alkene), 126.66, 129.45 (4CH), 132.04, 137.00 (2C), 138.46 (C of alkene), 171.92 (C=S). Analysis of $\text{C}_{13}\text{H}_{15}\text{NS}_2$ (249.39). (% calculation/found): C: 62.61/62.55, H: 6.06/6.00, N: 5.62/5.68.

3. Results and discussions

We examined the reaction of primary amines, carbon disulphide with α -haloketone in the presence of potassium iodide in dry CH_3OH at room temperature (25 °C) and we obtained the corresponding 3,4,5-trialkyl-1,3-thiazole-2(3H)-thione derivatives **4** in excellent yields. The reaction proceeds smoothly and cleanly under mild and neutral conditions, also no side products were observed. The compounds **4** were stable when stored at room temperature for several months.

The structures of products were deduced from their ^1H NMR, ^{13}C NMR, IR, and mass spectra. The mass spectra of these compounds displayed molecular ion peaks at the appropriate m/z values. The ^1H NMR spectrum of **4a** consisted of a singlet for CH_2 at

$\delta = 5.34$ ppm, two singlet for 2CH of furan at $\delta = 6.12$ and 6.24 ppm, and a multiplet at $\delta = 7.00$ –7.47 ppm for eleven aromatic protons and furan proton. The ^1H decoupled ^{13}C NMR spectrum of **4a** showed 16 distinct resonances, partial assignment of these resonances is given in section 2. The ^1H and ^{13}C NMR spectra of compounds **4b**–**i** were similar to those of **4a**, except for the aromatic moiety, which exhibited characteristic signals with appropriate chemical shifts.

A possible mechanism for the present reaction is shown in scheme 2, which envisages a tandem sequence. On the basis of the established chemistry of trivalent nitrogen nucleophiles, the successful nucleophilic attack by amines on a carbon atom is facilitated when the latter is conjugated with a carbonyl group, or when it is a part of an otherwise activated unsaturated bond. First, the nucleophilic addition of the primary amine **3** to carbon disulphide **2** generates the nucleophilic carbamodithioic acid **5**.²⁶ The next step involves nucleophilic attack of carbamodithioic acid **5** at the carbon of haloketone **1**, leading to intermediate **6**, and then ring closure by intramolecular attack of nitrogen at the carbonyl carbon to afford the 3,4,5-trialkyl-1,3-thiazole-2(3H)-thione derivatives **4**.

Potassium iodide was found to catalyse the synthesis of 3,4,5-trialkyl-1,3-thiazole-2(3H)-thione

Table 2. Synthesis of 3,4,5-trialkyl-1,3-thiazole-2(3H)-thione **4a** under various amounts of catalyst.

Entry	Catalyst (g)	Temp. (°C)	Time (h)	Yield (%)
1	KI (0.05)	r.t.	7	50
2	KI (0.07)	r.t.	7	60
3	KI (0.10)	r.t.	7	85
4	KI (0.15)	r.t.	5	85
5	KI (0.20)	r.t.	5	85

Table 3. Synthesis of 3,4,5-trialkyl-1,3-thiazole-2(3H)-thione **4a** in various solvents.

Entry	Solvent	Temp. (°C)	Time (h)	Yield (%)
1	H_2O	r.t.	7	–
2	$\text{C}_2\text{H}_5\text{OH}$	r.t.	7	50
3	CH_3OH	r.t.	5	85
4	CH_2Cl_2	r.t.	7	20
5	CHCl_3	r.t.	7	20

derivatives **4** from α -haloketone **1**, the primary amines **2**, and carbondisulphide **3**, in methanol (table 1). The use of just 0.1 g of potassium iodide (per mmol of reactants) is sufficient to push the reaction forward. Higher and lower amounts of potassium iodide (KI) did not considerably improve the yields (table 2). We also used other solvents in the presence of potassium iodide, but in all cases, increasing reaction times and decreasing yields of **4** were observed (table 3).

4. Conclusion

In this research work, a simple, mild and efficient method has been reported for preparation of 3,4,5-trialkyl-1,3-thiazole-2(3H)-thione derivatives. The products obtained in excellent yields under mild reaction and with simple work-up conditions. The products have been confirmed by physical and spectroscopic data such as; IR, ¹HNMR, ¹³CNMR, MS spectroscopy, and CHN elemental analyses.

Supplementary information

The electronic supporting information can be seen in www.ias.ac.in/chemsci.

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