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Electrochemical Synthesis of 3-(Het)aryl-5-methylthioisothiazoles from β-Oxodithioesters *via* β-amino-α,β-unsaturated dithioesters

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In this study, an efficient and sustainable electrochemical strategy for the synthesis of 3-(het)aryl-5-methylthioisothiazoles from readily accessible β-oxodithioesters is described. The protocol proceeds *via* a highly regioselective condensation of β-oxodithioesters with ammonium acetate to generate β-amino-α,β-unsaturated dithioesters, which undergo intramolecular oxidative cyclisation mediated by electrochemically generated iodine. A catalytic amount of tetra-*n*-butylammonium iodide serves as the iodine source, while constant-current electrolysis was carried out using reticulated vitreous carbon (RVC) as the anode and platinum as the cathode. This electrochemical approach provides an operationally simple and environmentally benign alternative to conventional oxidative cyclisation methods by eliminating the requirement for stoichiometric chemical oxidants and significantly reducing waste generation. This method unveils an attractive protocol for the synthesis of isothiazoles and overcomes limitations of traditional methods such as need of stoichiometric amounts of oxidants and generation of waste.

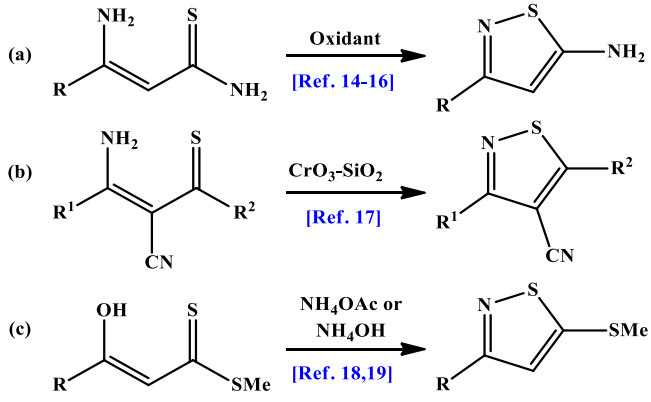
Keywords: Isothiazoles, β-Oxodithioesters, β-Enaminodithioesters, Electroorganic reaction, Green chemistry.

INTRODUCTION

Isothiazoles are an important class of sulfur-containing heterocycles with diverse biological activities for example, antibacterial [1], antifungal [2], anticancer [3], anti-inflammatory [4] and antiviral [5] properties. Their therapeutic potential has stimulated sustained interest in the development of efficient synthetic methods. Several approaches have been reported for isothiazole synthesis [6], such as the demethoxylative cycloaddition of alkynyl oxime ethers with sodium sulfide [7], Rh-catalysed cyclisation of 1,2,3-thiadiazoles with nitriles [8], one-pot reactions of enaminoesters, difluorobromoesters and sulphur [9] and KOH-mediated oxidative cyclisation of dithioesters with benzyl cyanides [10]. Practical and sustainable methods remain desirable for accessing these valuable heterocyclic frameworks.

Electro-organic synthesis has re-emerged as an attractive approach in synthetic chemistry since electricity replaces stoichiometric chemical oxidants, offering a cleaner and more sustainable alternative [11]. Electrochemically generated halogens, particularly chlorine, bromine and iodine, have found wide application in oxidative transformations of organic substrates [12,13]. A survey of the literature revealed only one electrochemical method for isothiazole synthesis using sulphonyl-substituted 2-alkenenitriles. Conventional preparations largely rely on oxidative cyclisation of β-amino-α,β-unsaturated thioamides with iodine, hydrogen peroxide or selenium dioxide (**Scheme-Ia**) [14-16], oxidative annulation of β-thioenaminones using silica-supported chromium trioxide (**Scheme-Ib**) [17] or conversion of β-oxodithioesters with ammonium acetate or ammonium hydroxide (**Scheme-Ic**) [18,19]. These protocols commonly require stoichiometric oxidants, elevated temperatures, prolonged reaction times or suffer from side-product formation.

Our research group has developed several electrochemical methods for the synthesis of sulphur- and phosphorus-containing compounds, including 1,3,4-thiadiazoles [20], 1,2,4-thiadiazoles [21], phosphonates [22], reductive coupling products [23], thiosulfonates [24], *etc*.. Building on these studies, we now report the first electrochemical synthesis of isothia-



Scheme-I: Previous works

zoles from β-oxodithioesters *via* β-amino-α,β-unsaturated dithioesters.

EXPERIMENTAL

All reagents and solvents were obtained from commercial chemical suppliers in India and were used as such. Reactions were monitored by thin layer chromatography technique (commercially available precoated plates, Merck $60F_{254}$, 0.25 mm thickness) and were visualised using UV rays. Melting points were determined by Superfit India melting apparatus and are uncorrected. IR spectra were recorded using Perkin-Elmer spectrometer. NMR spectra were recorded using Bruker NMR spectrometer (400 MHz). Mass spectra were recorded by using a Waters-SynaptG2 mass spectrometer.

General procedure for the synthesis of 3-(het)aryl-5-methylthioisothiazoles (6a-i): A mixture of β-oxodithioester (**4**, 1 mmol), ammonium acetate (2 mmol) and 4 Å molecular sieves (1 g) in benzene (5 mL) was refluxed under nitrogen atmosphere for 1 h. After the completion of reaction (monitored by TLC), molecular sieves were removed by filtration and benzene was removed by distillation under reduced pressure. The obtained β-enaminodithioester (**5**) was dissolved in THF (3 mL) followed by the dissolution of tetra *n*-butylammonium iodide (0.1 mmol). The resultant solution was electrolysed by passing a constant current of 10 mA through reticulated vitreous carbon (RVC) and platinum as anode and cathode respectively for 3 h. After the completion of reaction, water (25 mL) was added to the reaction mixture. The aqueous layer was extracted with ethyl acetate (25 mL) twice. The combined ethyl acetate layer was washed with brine (25 mL), dried over anhydrous sodium sulphate and concentrated under reduced pressure (**Scheme-II**). The obtained crude products were purified by column chromatography (stationary phase: 60-120 mesh silica; mobile phase: hexane).

5-(Methylthio)-3-phenylisothiazole (6a): Yellow liquid; IR (neat, ν_{max}, cm^{-1}): 2922, 1594, 1537, 518; ^1H NMR (400 MHz, $CDCl_3$, δ ppm): 7.91 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.40-7.47 (m, 3H, Ar-H), 7.36 (s, 1H, Ar-H), 2.64 (s, 3H, SMe); ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 167.9, 164.6, 134.6, 129.3, 128.8, 126.9, 118.9, 18.7; HRMS (ESI) *m/z*: calcd. for $C_{10}H_9NS_2$ $[M + H]^+$ 208.0255; found, 208.0252.

3-(4-Chlorophenyl)-5-(methylthio)isothiazole (6b): Pale yellow solid; m.p.: 98-100 ºC; IR (neat, ν_{max}, cm^{-1}): 2925, 1598, 1525, 524; ^1H NMR (400 MHz, $CDCl_3$, δ ppm): 7.83 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.41 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.32 (s, 1H, Ar-H), 2.65 (s, 3H, SMe); ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 166.6, 165.2, 135.3, 133.1, 129.0, 128.1, 118.7, 18.7; HRMS (ESI) *m/z*: calcd. for $C_{10}H_8ClNS_2$ $[M + H]^+$ 241.9865; found, 241.9868.

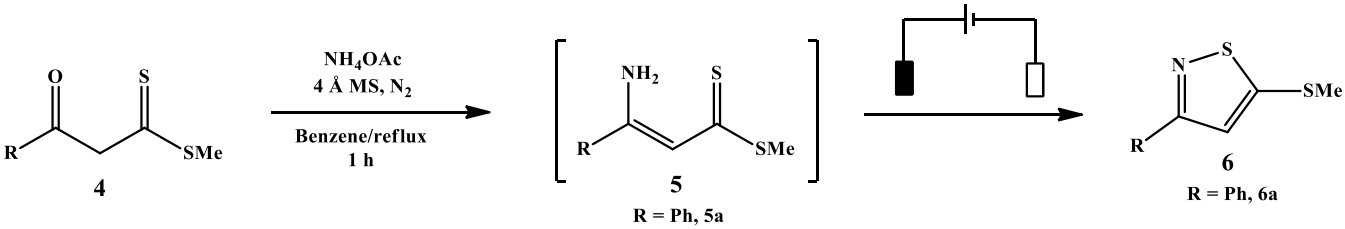
3-(4-Bromophenyl)-5-(methylthio)isothiazole (6c): Orange solid; m.p.: 78-80 ºC; IR (neat, ν_{max}, cm^{-1}): 2921, 1599, 1529, 528; ^1H NMR (400 MHz, $CDCl_3$, δ ppm): 7.76 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.56 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.31 (s, 1H, Ar-H), 2.64 (s, 3H, SMe); ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 166.6, 165.2, 133.5, 132.0, 128.4, 123.6, 118.6, 18.7; HRMS (ESI) *m/z*: calcd. for $C_{10}H_8BrNS_2$ $[M + H]^+$ 285.9360 and 287.9339; found, 285.9362 and 287.9336.

5-(Methylthio)-3-(4-nitrophenyl)isothiazole (6d): Red solid; m.p.: 104-106 ºC; IR (neat, ν_{max}, cm^{-1}): 2920, 1600, 1532, 536; ^1H NMR (400 MHz, $CDCl_3$) δ 8.30 (d, *J* = 8.0 Hz, 2H, Ar-H), 8.07 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.43 (s, 1H, Ar-H), 2.68 (s, 3H, SMe); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.8, 166.4, 165.2, 132.5, 127.6, 124.2, 118.9, 18.6; HRMS (ESI) *m/z*: calcd. for $C_{10}H_8N_2O_2S_2$ $[M + H]^+$ 253.0105; found, 253.0107.

5-(Methylthio)-3-(*p*-tolyl)isothiazole (6e): Pale yellow solid; m.p.: 52-54 ºC; IR (neat, ν_{max}, cm^{-1}): 2932, 1604, 1546, 541; ^1H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.34 (s, 1H, Ar-H), 7.24 (d, *J* = 8.0 Hz, 2H, Ar-H), 2.64 (s, 3H, SMe), 2.39 (Ar-Me); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.9, 164.3, 139.4, 132.0, 129.5, 126.7, 118.8, 21.4, 18.7; HRMS (ESI) *m/z*: calcd. for $C_{11}H_{11}NS_2$ $[M + H]^+$ 222.0411; found, 222.0413.

3-(4-Methoxyphenyl)-5-(methylthio)isothiazole (6f): Pale yellow solid; m.p.: 68-70 ºC; IR (neat, ν_{max}, cm^{-1}): 2935, 1601, 1548, 555; ^1H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.30 (s, 1H, Ar-H), 6.95 (d, *J* = 8.0 Hz, 2H, Ar-H), 3.85 (s, 3H, OMe), 2.64 (s, 3H, SMe); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 164.2, 160.5, 128.2, 127.7, 118.5, 114.1, 55.4, 18.7; HRMS (ESI) *m/z*: calcd. for $C_{11}H_{11}NOS_2$ $[M + H]^+$ 238.0360; found, 238.0362.

3-(3,4-Dimethoxyphenyl)-5-(methylthio)isothiazole (6g): Yellow solid; m.p.: 96-98 ºC; IR (neat, ν_{max}, cm^{-1}):



Scheme-II: Synthetic route of 3-substituted-5-methylthioisothiazoles

2931, 1598, 1551, 561; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (s, 1H, Ar-H), 7.39 (d, *J* = 8.0 Hz, 1H, Ar-H), 7.32 (s, 1H, Ar-H), 6.91 (d, *J* = 8.0 Hz, 1H, Ar-H), 3.97 (s, 3H, OMe), 3.93 (s, 3H, OMe), 2.65 (s, 3H, SMe); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 164.3, 150.1, 149.3, 127.9, 119.7, 118.6, 110.9, 109.6, 56.00, 55.97, 18.7; HRMS (ESI) *m/z*: calcd. for $C_{12}H_{13}NO_2S_2$ $[M + H]^+$ 268.0466; found, 268.0469.

5-(Methylthio)-3-(naphthalen-2-yl)isothiazole (6h): Yellow solid; m.p.: 106-108 ºC; IR (neat, ν_{max}, cm^{-1}): 2936, 1594, 1558, 572; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 8.35 (s, 1H, Ar-H), 8.08 (d, *J* = 8.0 Hz, 1H, Ar-H), 7.90-7.92 (m, 3H, Ar-H), 7.49-7.53 (m, 3H, Ar-H), 2.68 (s, 3H, SMe); ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 167.8, 164.7, 133.7, 133.3, 132.0, 128.61, 128.59, 127.8, 126.7, 126.5, 126.3, 124.4, 119.1, 18.7; HRMS (ESI) *m/z*: calcd. for $C_{14}H_{11}NS_2$ $[M + H]^+$ 258.0411; found, 258.0410.

5-(Methylthio)-3-(thiophen-2-yl)isothiazole (6i): Yellow liquid; IR (neat, ν_{max}, cm^{-1}): 2925, 1598, 1561, 556; ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 7.44 (d, *J* = 8.0 Hz, 1H, Ar-H), 7.36 (d, *J* = 8.0 Hz, 1H, Ar-H), 7.24 (s, 1H, Ar-H), 7.08 (t, *J* = 4.0 Hz, 1H, Ar-H), 2.64 (s, 3H, SMe); ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 164.8, 162.3, 138.7, 127.7, 127.2, 125.8, 118.2, 18.6; HRMS (ESI) *m/z*: calcd. for $C_8H_7NS_3$ $[M + H]^+$ 213.9819; found, 213.9815.

RESULTS AND DISCUSSION

The β-oxodithioesters (**4**) were regioselectively condensed with ammonium acetate in refluxing benzene under a N_2 atmosphere using 4 Å molecular sieves to afford β-amino-α,β-unsaturated dithioesters (**5**) (**Scheme-II**). This protocol represents an improved version of the reported method [25], in which molecular sieves replace the Dean-Stark apparatus for efficient removal of the water formed during the reaction.

Optimisation of the electrochemical cyclisation was carried out using (*Z*)-methyl 3-amino-3-phenylprop-2-enedithioate (**5a**, R = Ph) under constant-current electrolysis (10 mA). Electrolysis in benzene employing tetra-*n*-butylammonium iodide (10 mol%) as the iodide source, RVC as the anode and platinum as the cathode failed to obtain the desired 5-(methylthio)-3-phenylisothiazole (**6a**) (Table-1, entry 1). Replacing benzene with DMF afforded **6a** in 56% yield (entry 2), while THF increased the yield to 87% (entry 3). Ethanol proved less effective, giving **6a** in 45% yield (entry 4). Substituting tetra-*n*-butylammonium iodide with ammonium iodide or potassium iodide decreased the yields to 26% and 20%, respectively (entries 5 and 6). Replacing the RVC anode with platinum under otherwise identical conditions also reduced the yield to 29% (entry 7). These results established tetra-*n*-butylammonium iodide (10 mol%), THF, an RVC anode, and a platinum cathode as the optimal reaction conditions.

The scope of the method was evaluated under the optimised conditions. β-Oxodithioesters bearing electron withdrawing substituents such as chloro, bromo and nitro groups afforded the corresponding isothiazoles **6b-d** in 75-84% yields. Methyl- and methoxy-substituted substrates afforded isothiazoles **6e-g** in 85-90% yields. The corresponding 2-naphthyl and 2-thienyl analogues **6h** and **6i** were isolated in 81% and 78% yields, respectively. In general, electron-donating substituents afforded slightly higher yields than electron-withdrawing substituents. Several of the synthesised isothiazoles have been reported previously and can be prepared by earlier methods [18,19]. A comparison of the present protocol with the reported procedures (Table-2) shows shorter reaction times together with improved product yields.

TABLE-1
OPTIMISATION OF REACTION CONDITIONS FOR THE SYNTHESIS OF 5-(METHYLTHIO)-3-PHENYLISOTHIAZOLE (**6a**)

Entry	Anode/cathode	Electrolyte	Solvent	Yield (%)
1	RVC/Pt	*n*-Bu_4NI	Benzene	0
2	RVC/Pt	*n*-Bu_4NI	DMF	56
3	RVC/Pt	*n*-Bu_4NI	THF	87
4	RVC/Pt	*n*-Bu_4NI	EtOH	45
5	RVC/Pt	NH_4I	THF	26
6	RVC/Pt	KI	THF	20
7	Pt/Pt	*n*-Bu_4NI	THF	29

Reaction conditions: **5a** (1 mmol), electrolyte (10 mol%), I = 10 mA, CCE, 3 h.

TABLE-2
SUBSTRATE SCOPE AND ISOLATED YIELDS UNDER OPTIMIZED ELECTROCHEMICAL CONDITIONS

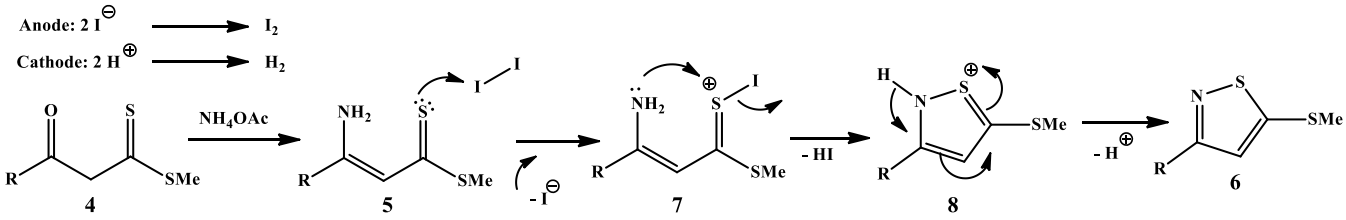
Entry	R	Compd.	Yield (%)	Yield (%)[a] [18]	Yield (%)[b] [19]
1	C_6H_5	**6a**	87	75	10
2	4-ClC_6H_4	**6b**	84	75	–
3	4-BrC_6H_4	**6c**	80	–	10
4	4-$NO_2C_6H_4$	**6d**	75	–	–
5	4-MeC_6H_4	**6e**	85	70	12
6	4-$MeOC_6H_4$	**6f**	90	70	5
7	3,4-$(MeO)_2C_6H_3$	**6g**	88	–	–
8	2-Naphthyl	**6h**	81	–	–
9	2-Thienyl	**6i**	78	80	12

Reaction conditions: **4** (1 mmol), NH_4OAc (2 mmol), MS (1 g), benzene (5 mL), N_2, reflux, 1 h; RVC/Pt, *n*-Bu_4NI (10 mol%), THF (3 mL) 3 h; [a] 7 h; [b] 9-12 h.

A plausible reaction mechanism is outlined in **Scheme-III**. Ammonia generated from ammonium acetate undergoes highly regioselective condensation with β-oxodithioesters (**4**) to form β-amino-α,β-unsaturated dithioesters (**5**). During electrolysis, iodide is oxidized at the anode to molecular iodine, which reacts with **5** to generate cationic intermediate **7**. Intramolecular cyclisation affords intermediate **8** and subsequent deprotonation furnishes the corresponding 3-(het)aryl-5-methylthioisothiazoles (**6a-i**). Simultaneously, proton reduction at the platinum cathode releases H_2 gas, completing the catalytic electrochemical cycle.

Conclusion

In summary, a highly regioselective protocol has been developed for the synthesis of β-amino-α,β-unsaturated dithioesters through the reaction of β-oxodithioesters with ammonium acetate in refluxing benzene in the presence of 4 Å molecular sieves. After completion of the reaction, the molecular sieves were removed by filtration and the solvent was evaporated under reduced pressure. The resulting β-amino-α,β-



Scheme-III: Possible mechanism of formation of isothiazoles

unsaturated dithioesters were subjected to constant current electrolysis using a catalytic amount of tetra-*n*-butyl-ammonium iodide, which functioned as both the supporting electrolyte and the iodine source. Electrolysis was performed with reticulated vitreous carbon (RVC) as the anode and platinum as the cathode, leading to the formation of 3-(het)-aryl-5-methylthioisothiazoles. The *in situ* generation of iodine eliminates the need for stoichiometric oxidants such as I_2, H_2O_2, SeO_2 and chromium-based oxidants, thereby reducing chemical waste and improving atom economy. The requirement of benzene as solvent in the condensation step remains a limitation of the present protocol.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language and no data, results or scientific conclusions were generated by AI. All analyses, interpretations and conclusions are the authors' own. The authors reviewed and edited the content and take full responsibility for the published work.

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