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An efficient microwave-assisted synthesis of thieno[2,3-*b*]quinolines under solvent-free conditions

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A novel and efficient method for the synthesis of substituted thieno[2,3-*b*]quinolines has been developed. A simple one-pot reaction of 3-formyl-2-mercaptoquinolines **2a–l** with 1-chloroacetone, 2-chloroacetamide, ethyl chloroacetate and 2-chloro-1-phenylethanone in presence of catalytic amount of potassium carbonate under microwave irradiation and solvent-free conditions gave thieno[2,3-*b*]quinolin-2-ylethanone derivatives **3a–e**, thieno[2,3-*b*]quinoline-2-carboxamide derivatives **4a–e**, ethyl thieno[2,3-*b*]quinoline-2-carboxylate **5a–e** and phenyl(thieno[2,3-*b*]quinolin-2-yl)methanone derivatives **6a–e** compounds respectively. The structures of all the newly synthesised compounds were elucidated on the basis of elemental analysis, IR, ^{1}H NMR and mass spectral data.

Keywords: Microwave irradiation; Solvent-free synthesis; 3-Formyl-2-mercapto quinolines; Thieno[2,3-*b*]quinolines

1. Introduction

Thieno[2,3-*b*]quinoline derivatives are well documented for antibacterial [1–3], antifungal [4], antianaphylactic [5] and anti-inflammatory activity [6]. The antitumour drugs that intercalate DNA are of growing interest in the field of anticancer derivatives. Some of the derivatives undergoing clinical trials endowed with only one side chain are constituted by acridine-4-carboxamide DACA [7], mitonafide [8], azonafide [9] and imidazoacridones [10]. Various fused systems such as thiophene [11], furan and pyridine analogues of ellipticine [12] and benzothiazoloquinolines [13] have been studied for their intercalative properties. The synthesis of pyrimidothienoquinolines and their binding studies have been recently reported in literature [14–16].

Microwave-induced Organic Reaction Enhancement (MORE) Chemistry [17] has received considerable attention in the recent years due to several advantages such as short reaction times,

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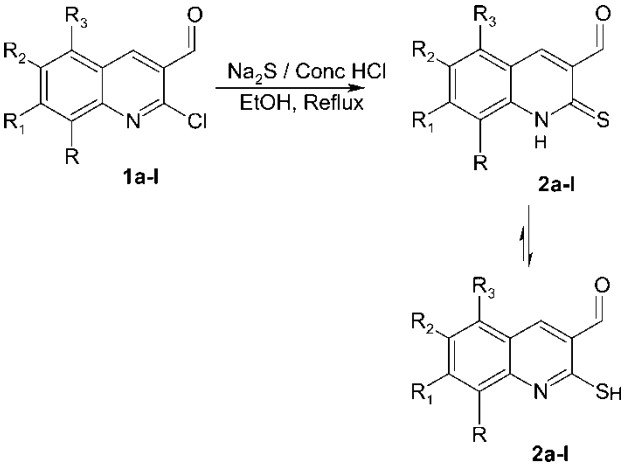


SCHEME 1 General synthetic procedure of 3-formyl-2-mercaptoquinolines derivatives **2a–l**.

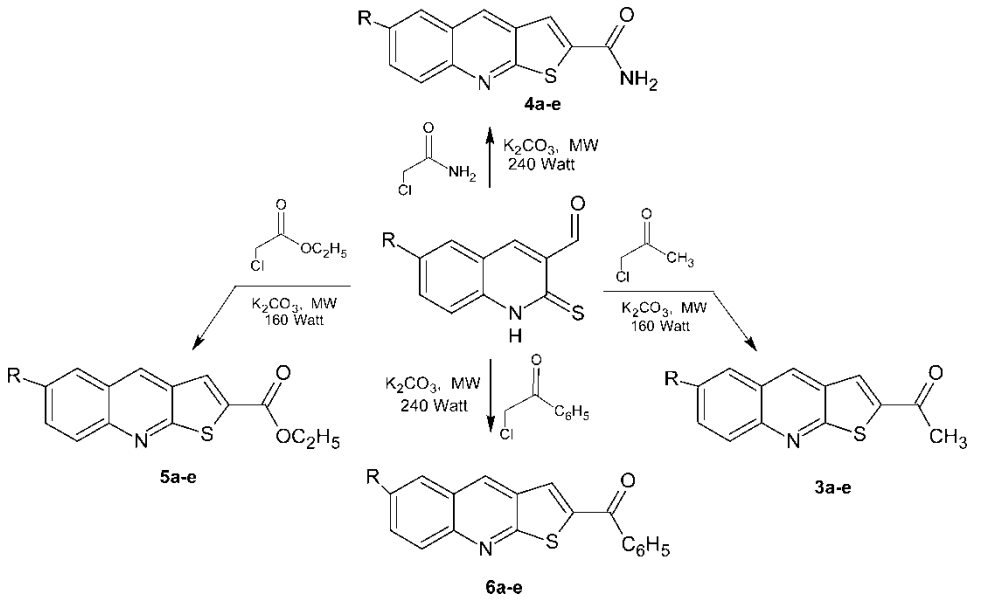
ease of work-up, excellent yields and cost-effectiveness. Moreover, it is an environmentally friendly technique and is understood to be a step towards green chemistry [18]. Since the appearance of pioneering reports [19, 20] on the application of microwaves for chemical synthesis in polar solvents, the approach has blossomed into a useful technique for several reactions of synthetic importance [21, 22].

Recently, we have reported [23] the efficient synthesis of a family of 3-formyl-2-mercaptoquinolines from 3-formyl-2-chloroquinolines (scheme 1). In continuation of our work on condensed heterocycles [24, 25] and in view of growing interest in the field of anticancer agent, herein we wish to report an efficient synthesis of novel title compounds **3a–e**, **4a–e**, **5a–e** and **6a–e** by application of microwave energy.

2. Results and discussion

A wide range of substituted thieno[2,3-*b*]quinolines such as **3a–e**, **4a–e**, **5a–e** and **6a–e** were prepared by treating 3-formyl-2-mercaptoquinolines **2a–l** with 1-chloroacetone, 2-chloroacetamide, ethyl chloroacetate and 2-chloro-1-phenylethanone in the presence of potassium carbonate under microwave irradiation as shown in (scheme 2). 3-Formyl-2-mercaptoquinolines **2a–l** were prepared from 3-formyl-2-chloroquinolines **1a–l**. The structural elucidation of the newly synthesised compounds was established on the basis of their IR, ^{1}H NMR and mass spectral data.

As an example, the IR spectrum of **2a** showed the absorption band at 1640 cm^{-1} corresponding to −CHO group, 3410–3490 cm^{-1} corresponding to the tautomeric form of the NH group, which was found to be absent in the IR spectrum of **3a**. The ^{1}H NMR spectrum of **2a** exhibits broad singlet at δ 11.20 ppm corresponding to tautomeric form of the NH group and singlet at δ 10.31 ppm corresponding to −CHO group, both of which were found to be absent in **3a**. A singlet at δ 2.51 ppm corresponding to the $-COCH_3$ group of **3a** indicated the attachment of the reactive partner to the quinoline substrate, presumably at the sulfur. Further evidence for condensation of the aldehydes after sulfur substitution was the absence of a singlet near at δ 5.5 ppm corresponding to an unreacted –S–CH_2– group. Finally, the



SCHEME 2 General synthetic procedure of thieno[2,3-*b*]quinoline derivatives **3a**–**e**, **4a**–**e**, **5a**–**e** and **6a**–**e**. (R: **a**, H; **b**, CH_3; **c**, OCH_3; **d**, Cl; **e**, Br).

structure assigned was confirmed by its mass spectrum through the appearance of a molecular ion peak at $m/z = 277$.

To the best of our knowledge, the preparation of thieno[2,3-*b*]quinoline derivatives has not been reported under solvent free condition. These ring systems have the potential to provide the basis for further derivatisation.

3. Experimental

IR spectra were taken on a Perkin Elmer 157 Infrared spectrophotometer. The ^{1}H NMR spectra (300 MHz) were recorded on a Bruker supercon FT NMR instrument using TMS as internal standard and mass spectra on a Jeol JMS-D 300 Mass spectrometer operating at 70 eV. Melting points were determined in open capillary and are uncorrected. Purity of the compounds was checked by TLC on silica gel and purification was by column chromatography. The microwave-assisted procedures were carried out in a LG microwave oven specially designed for organic synthesis operating was a maximum power of 1000 W.

3.1 *Preparation of methyl ketones 3. Example; thieno[2,3-b]quinolin-2-yl methyl ketone (3a)*

A mixture of **2a** (1800 mg, 10 mmol), 1-chloroacetone (920 mg, 10 mmol) and potassium carbonate (690 mg, 10 mmol) were ground for uniform mixing. The mixture was then irradiated by microwave radiation in a domestic microwave oven for 10 min at an interval of 1 min at 160 Watt as required to complete the reaction (TLC). The reaction mixture was then poured into water, stirred, filtered and dried. The crude product was purified by column chromatography on silica gel with ethyl acetate-benzene (9:1) as eluent to gave 1900 mg (83%) of **3a**. In a similar way, **3b**–**e** were prepared in 82–85% yield.

3.2 *Thieno[2,3-b]quinolin-2-yl methyl ketone (3a)*

Solid, mp. 280 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.51 (3H, s, $COCH_3$), 7.61–9.72 (6H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1653. [M+], 227. Calcd. (%) for $C_{13}H_9NOS$: C; 68.70, H; 3.99, N; 6.16, S; 14.11. Found: C; 68.13, H; 3.87, N; 6.26, S; 14.19.

3.3 *6-Methylthieno[2,3-b]quinolin-2-yl methyl ketone (3b)*

Solid, mp. 285 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.61 (3H, s, $COCH_3$), 2.46 (3H, s, Ar-CH_3), 7.62–9.83 (5H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1650. [M+], 241. Calcd. (%) for $C_{14}H_{11}NOS$: C; 69.68, H; 4.59, N; 5.80, S; 13.29. Found: C; 69.49, H; 4.29, N; 5.60, S; 13.27.

3.4 *6-Methoxythieno[2,3-b]quinolin-2-yl methyl ketone (3c)*

Solid, mp. 292 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.63 (3H, s, $COCH_3$), 4.01 (3H, s, Ar-OCH_3), 7.31–9.13 (5H, m, Ar-H); IR ν (KBr) (cm^{-1}): 1650. [M+], 257. Calcd. (%) for $C_{14}H_{11}NO_2S$: C; 65.35, H; 4.31, N; 5.44, S; 12.46. Found: C; 65.79, H; 4.13, N; 5.12, S; 12.49.

3.5 *6-Chlorothieno[2,3-b]quinolin-2-yl methyl ketone (3d)*

Solid, mp. 289 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.50 (3H, s, $COCH_3$), 7.72–9.85 (5H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1652. [M+], 261. Calcd. (%) for $C_{13}H_8ClNOS$: C; 59.66, H; 3.08, N; 5.35, S; 12.25. Found: C; 59.38, H; 3.01, N; 5.15, S; 12.29.

3.6 *6-Bromothieno[2,3-b]quinolin-2-yl methyl ketone (3e)*

Solid, mp. 285 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.41 (3H, s, $COCH_3$), 7.63–9.72 (5H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1650. [M+], 306. Calcd. (%) for $C_{13}H_8BrNOS$: C; 51.00, H; 2.63, N; 4.57, S; 10.47. Found: C; 50.07, H; 2.32, N; 4.34, S; 10.49.

3.7 *Preparation of carboxamides 4. Example; thieno[2,3-b]quinoline-2-carboxamide 4a*

A mixture of **2a** (1800 mg, 10 mmol), 2-chloroacetamide (935 mg, 10 mmol) and potassium carbonate (690 mg, 10 mmol) were ground for uniform mixing. It was irradiated by microwave radiation in a domestic microwave oven for 8 min at an interval of 1 min at 240 Watt. The reaction mixture was poured into ice cold water, the resulting brown solid was collected by filtration, dried and recrystallized from ethyl acetate-chloroform (8:2) to provide **4a** (1710 mg, 75%). The same procedure was adopted for the synthesis of **4b–e** compounds and obtained yield was found to be 78–80%.

3.8 *Thieno[2,3-b]quinoline-2-carboxamide (4a)*

Solid, mp. 215 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 7.23–7.54 (2H, s, NH_2), 7.51–9.33 (6H, m, Ar-H); IR (KBr) ν (cm^{-1}): 3450–3485, 1640. [M+], 228. Calcd. (%) for $C_{12}H_8N_2OS$: C; 63.14, H; 3.53, N; 12.27, S; 14.05. Found: C; 63.24, H; 3.63, N; 12.25, S; 14.02.

3.9 *6-Methylthieno[2,3-b]quinoline-2-carboxamide (4b)*

Solid, mp. 226 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.32 (3H, s, Ar-CH_3), 7.31–7.53 (2H, s, NH_2) 7.63–9.52 (5H, m, Ar-H); IR (KBr) ν (cm^{-1}): 3450–3485, 1642. [M+], 242. Calcd. (%) for $C_{13}H_{10}N_2OS$: C; 64.44, H; 4.16, N; 11.56, S; 13.23. Found C; 64.24, H; 4.12, N; 11.65, S; 13.27.

3.10 *6-Methoxythieno[2,3-b]quinoline-2-carboxamide (4c)*

Solid, mp. 210 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.02 (3H, s, Ar-OCH_3), 7.21–7.42 (2H, s, NH_2), 7.52–9.52 (5H, m, Ar-H); IR (KBr) ν (cm^{-1}): 3450–3485, 1641. [M+], 258. Calcd. (%) for $C_{13}H_{10}N_2O_2S$: C; 60.45, H; 3.90, N; 10.85, S; 12.41. Found: C; 60.49, H; 3.88, N; 10.79, S; 12.38.

3.11 *6-Chlorothieno[2,3-b]quinoline-2-carboxamide (4d)*

Solid, mp. 228 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 7.22–7.53 (2H, s, NH_2), 7.51–9.32 (5H, m, Ar-H); IR (KBr) ν (cm^{-1}): 3450–3485, 1643. [M+], 262. Calcd. (%) for $C_{12}H_7ClN_2OS$: C; 54.86, H; 2.69, N; 10.66, S; 12.21. Found: C; 54.83; H; 2.63, N; 10.69, S; 12.39.

3.12 *6-Bromothieno[2,3-b]quinoline-2-carboxamide (4e)*

Solid, mp. 230 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 7.30–7.42 (2H, s, NH_2), 7.61–9.34 (5H, m, Ar-H); IR (KBr) ν (cm^{-1}): 3450–3485, 1642. [M+], 307. Calcd. (%) for $C_{12}H_7BrN_2OS$: C; 46.92, H; 2.30, N; 9.12, S; 10.44. Found: C; 46.91, H; 2.29, N; 9.10, S; 10.47.

3.13 *Preparation of ethyl esters 5. Example; ethyl thieno[2,3-b]quinoline-2-carboxylate (5a)*

A mixture **2a** (1800 mg, 10 mmol), ethyl chloroacetate (1220 mg, 10 mmol) and potassium carbonate (690 mg, 10 mmol) were ground for uniform mixing. The mixture was irradiated by microwave radiation in a domestic microwave oven for 12 min at an interval of 1 min at 160 Watt as required to complete the reaction. The yellow solid was filtered off, washed with water and dried. The obtained product was purified by column chromatography on silica gel with ethyl acetate-chloroform (7:3) as eluent to gave 2133 mg (83%) of **5a**. In a similar way, the same procedure was followed for the synthesis of **5b**–**e** in 70–80% yield.

3.14 *Ethyl thieno[2,3-b]quinoline-2-carboxylate (5a)*

Solid, mp. 175 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 1.31 (3H, t, CH_3), 4.32 (2H, q, OCH_2) 7.61–9.20 (6H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1740. [M+], 257. Calcd. (%) for $C_{14}H_{11}NO_2S$: C; 65.35, H; 4.31, N; 5.44, S; 12.44. Found: C; 65.38, H; 4.33, N; 5.47, S; 12.49.

3.15 *Ethyl 6-methylthieno[2,3-b]quinoline-2-carboxylate (5b)*

Solid, mp. 182 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 1.41 (3H, t, CH_3), 2.33 (3H, s, Ar-CH_3), 4.22 (2H, q, OCH_2) 7.50–9.21 (5H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1745 [M+], 271.

Calcd. (%) for $C_{15}H_{13}NO_2S$: C; 66.40, H; 4.83, N; 5.16, S; 11.82. Found: C; 66.38, H; 4.79, N; 5.14, S; 11.79.

3.16 *Ethyl 6-methoxythieno[2,3-b]quinoline-2-carboxylate (5c)*

Solid, mp. 185 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 1.31 (3H, t, CH_3), 4.01 (3H, s, Ar-OCH_3), 4.21 (2H, q, OCH_2) 7.62–9.45 (5H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1748. [M+], 287. Calcd. (%) for $C_{15}H_{13}NO_3S$: C; 62.70, H; 4.56, N; 4.87, S; 11.16. Found: C; 62.46, H; 4.52, N; 4.89, S; 11.18.

3.17 *Ethyl 6-chlorothieno[2,3-b]quinoline-2-carboxylate (5d)*

Solid, mp. 198 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 1.51 (3H, t, CH_3), 4.32 (2H, q, OCH_2) 7.71–9.50 (5H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1744. [M+], 291. Calcd. (%) for $C_{14}H_{10}ClNO_2S$: C; 57.63, H; 3.45, N; 4.80, S; 10.99. Found: C; 57.58, H; 3.41, N; 4.78, S; 10.95.

3.18 *Ethyl 6-bromothieno[2,3-b]quinoline-2-carboxylate (5e)*

Solid, mp. 182 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): ^{1}H NMR (300 MHz) (DMSO-d_6) (δ) ppm: 1.21 (3H, t, CH_3), 4.23 (2H, q, OCH_2) 7.71–9.43 (5H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1745. [M+], 336. Calcd. (%) for $C_{14}H_{10}BrNO_2S$: C; 50.01, H; 3.00, N; 4.17, S; 9.54. Found: C; 50.03, H; 3.01, N; 4.16, S; 9.52.

3.19 *Preparation of phenyl ketones 6. Example; thieno[2,3-b]quinolin-2-yl phenyl ketone (6a)*

A mixture of **2a** (1800 mg, 10 mmol), 2-chloro-1-phenylethanone (1540 mg, 10 mmol) and potassium carbonate (690 mg, 10 mmol) were ground for uniform mixing. The mixture was then irradiated by microwave radiation in a domestic microwave oven for 8 min at an interval of 1 min at 240 Watt. The reaction mixture was then poured into water, stirred, filtered and dried. The crude product was recrystallized from DMF to provide 1900 mg (69%) of pure **6a**. In a similar way, **6b–e** were prepared in 70–74% yield.

3.20 *Thieno[2,3-b]quinolin-2-yl phenyl ketone (6a)*

Solid, mp. 205 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 7.30–9.61 (11H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1640. [M+], 289. Calcd. (%) for $C_{18}H_{11}NOS$: C; 74.22, H; 3.83, N; 4.84, S; 11.08. Found: C; 74.19, H; 3.80, N; 4.81, S; 11.07.

3.21 *6-Methylthieno[2,3-b]quinolin-2-yl phenyl ketone (6b)*

Solid, mp. 217 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.32 (3H, s, Ar-CH_3), 7.31–9.61 (10H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1642. [M+], 303. Calcd. (%) for $C_{19}H_{13}NOS$: C; 75.22, H; 4.32, N; 4.62, S; 10.57. Found: C; 75.39, H; 4.29, N; 4.69, S; 10.59.

3.22 *6-Methoxythieno[2,3-b]quinolin-2-yl phenyl ketone (6c)*

Solid, mp. 222 °C. ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.24 (3H, s, Ar-OCH_3), 7.56–9.72 (10H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1641. [M+], 319. Calcd. (%) for $C_{19}H_{13}NO_2S$: C; 71.45, H; 4.10, N; 4.39, S; 10,04. Found: C; 71.39, H; 4.9, N; 4.37, S; 10,03.

3.23 *6-Chlorothieno[2,3-b]quinolin-2-yl phenyl ketone (6d)*

Solid, mp. 237 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm) 7.31–9.73 (10H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1642. [M+], 323. Calcd. (%) for $C_{18}H_{10}ClNOS$: C; 66.77, H; 3.11, N; 4.33, S; 9.90. Found: C; 66.73, H; 3.10, N; 4.30, S; 9.87.

3.24 *6-Bromothieno[2,3-b]quinolin-2-yl phenyl ketone (6e)*

Solid, mp. 228 °C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 7.31–9.65 (10H, m, Ar-H); IR (KBr) ν (cm^{-1}): 1644. [M+] 368. Calcd. (%) for $C_{19}H_{10}BrNOS$: C; 58.71, H; 2.74, N; 3.80, S; 8.71. Found: C; 58.83, H; 2.64, N; 3.78, S; 8.69.

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