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A microwave-assisted, facile, regioselective Friedländer synthesis and antitubercular evaluation of 2,9-diaryl-2,3-dihydrothieno-[3,2-*b*]quinolines

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ABSTRACT

A series of 2,9-diaryl-2,3-dihydrothieno[3,2-*b*]quinolines have been synthesized regioselectively by Friedländer annulation of 5-aryldihydro-3(2*H*)-thiophenones and 2-aminobenzophenones in the presence of trifluoroacetic acid in good yields under microwave irradiation at 100 °C. The 2,9-diaryl-2,3-dihydrothieno[3,2-*b*]quinolines were screened for *in vitro* antimycobacterial activity against *Mycobacterium tuberculosis* H37Rv (MTB) and multi-drug resistant *M. tuberculosis* (MDR-TB). Among the 17 compounds screened, 7-chloro-2-(2,4-dichlorophenyl)-9-phenyl-2,3-dihydrothieno-[3,2-*b*]quinoline and 7-chloro-2-(3-nitrophenyl)-9-phenyl-2,3-dihydrothieno[3,2-*b*]quinoline display maximum activity with MIC of 0.90 and 0.95 μM against MTB and MDR-TB respectively.

1. Introduction

Quinolines constitute an important class of heterocycles as the quinoline structural unit is prevalent in naturally occurring quinoline alkaloids, therapeutics and synthetic analogues with important biological activities [1] such as antimalarial [2], antiasthmatic [3], antidiabetic [4], antibacterial [5], *in vitro* antifungal [6], antiviral [7], anti-inflammatory [8], immunosuppressive [9], HIV-1 integrase inhibitory [10], anti-breast cancer [11] and antiproliferative activities [12]. Sulfur-containing fused quinolines, in particular, thienoquinolines display significant biological and pharmacological activities [13–16]. The quinoline derivative, TMC 207 (Fig. 1), possessing a new mechanism of action, is in phase 2 clinical trials with very promising activity against MDR-TB [17]. Our recent studies on hybrid heterocycles with indole, thiophene/dihydrothiophene sub-structures (Fig. 2) display significant antimycobacterial activities [18]. This prompted the synthesis of thienoquinolines, with one phenyl ring (with a shift in its position from side chain of the quinoline derivative (TMC207) to the quinoline ring) and one variable aryl ring, incorporating all the encircled portions of the diarylquinoline and thienoindoles (Scheme 1) and screen them for antimycobacterial activities. Retrosynthesis points to the readily available starting materials 5-aryldihydro-3(2*H*)-thiophenone **1** [18] and 2-aminobenzophenone **2**, which upon Friedländer reaction could furnish the thienoquinolines **3** (Scheme 1).

This study forms a part of our research programme embarked on the construction of novel heterocycles [19] and/or their screening for antimycobacterial activities [20]. It is to be noted that tuberculosis (TB) has become an important world-wide public health problem with one-third of the world's population infected by the TB bacillus resulting in a death toll of 2 million, mostly from developing countries [21]. The pathogenic synergy of tuberculosis with HIV [22] enhances the overall incidence of TB in HIV-positive patients by 50 times relative to the rate for HIV-negative individuals [23]. The emergence of multi-drug resistant tuberculosis (MDR-TB) and extensively drug resistant tuberculosis (XDR-TB) further aggravates the problems associated with TB treatment and render the development of new drugs and strategies to efficiently treat this disease imperative.

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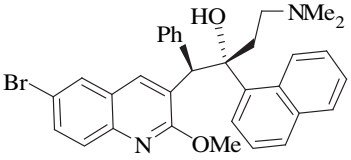


Fig. 1. Quinoline derivative, TMC 207.

2. Chemistry

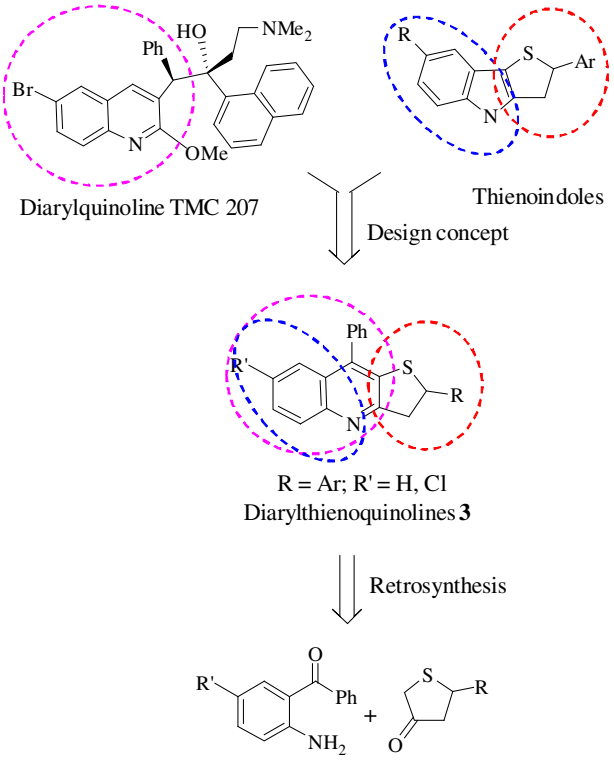
In the present work, the synthesis of a series of 2,9-diaryl-2,3-dihydrothieno[3,2-*b*]quinolines **3** was initially investigated under thermal conditions by heating a mixture of 5-aryldihydro-3(2*H*)-thiophenone **1**, [18] 2-aminobenzophenone **2** and trifluoroacetic acid (Scheme 2) in a 1:1:1.5 M ratio on an oil bath at 100 °C for 2–2.5 h in the absence of solvent. After completion of the reaction (TLC) and work up, the product **3** was isolated in a pure form by column chromatography.

This reaction was further investigated under solvent-free microwave irradiation with a view to rendering it efficient since reactions under microwave irradiation, in general, enhance the yield of the product besides accelerating the reactions, minimizing the decomposition of the reactants and/or products and affording enhanced selectivity relative to the thermal reactions [24]. Consequently, in the present study, the synthesis of 2,9-diaryl-2,3-dihydrothieno[3,2-*b*]quinolines (Table 1; Scheme 2) was performed by reacting a 1:1:1.5 M ratio of 5-aryldihydro-3(2*H*)-thiophenone **1**, 2-aminbenzophenone **2** and trifluoroacetic acid under solvent-free microwave irradiation at 100 °C. These reaction conditions were optimized by varying the amount of catalyst and temperature (Table 2). Typically, a mixture of 5-aryldihydro-3(2*H*)-thiophenone **1**, 2-aminoaryl ketone **2** and trifluoroacetic acid (1:1:1.5 mmol) in a sealed vial was irradiated in a focused microwave oven at 100 °C at 40 W power level with 1 bar pressure at high absorption level for 30 min. After completion of the reaction (TLC), the product **3** was isolated. This reaction proceeded more rapidly and afforded better yields (65–98%) of **3** than the thermal reaction (Table 1).

It is pertinent to note that the Friedländer reaction of *o*-aminobezophenone and 5-phenyldihydro-3(2*H*)-thiophenone **1a** in the presence of HCl at 120 °C was previously reported to afford 2,9-diphenyl-2,3-dihydrothieno[3,2-*b*]quinoline, **3a**, as the only compound in this series, in 75% yield [25]. Generally, Friedländer reactions are performed by heating either an aqueous or alcoholic solution of reactants under reflux in the presence of a catalyst or a mixture of the reactants at high temperatures (150–220 °C) without catalyst [26]. Different catalysts, *viz.* base [27], Brønsted acids [28], Lewis acid [29], inorganic salt [30], and ionic liquid [31] have been employed to effect this reaction. However, most of the previous methods employ harsh reaction conditions and/or expensive catalysts, encounter work up difficulties and afford low yields of the product. However, in the present work, the reaction in presence of trifluoroacetic acid results in a significantly better yield, *viz.* 80% from thermal and 98% from microwave reaction than 75% reported for **3a** in the presence of HCl under thermal conditions.

Although two regioisomeric thienoquinolines **3** and **3′** are possible in this reaction (Scheme 3) as the 5-aryldihydro-3(2*H*)-thiophenone **1** is unsymmetrical, the reaction furnishes only **3**, whose structure is in accord with the NMR spectroscopic data as described for a representative example, **3k**. The ^{1}H NMR spectrum of **3k** has two doublets of doublets and one triplet corresponding to the AMX spin system of the CH–CH_2 part of the thiene ring of **3k**. The diastereotopic 3-CH_2 hydrogens appear as doublets of doublets at 3.78 ppm (J = 16.7 and 8.3 Hz) and 3.90 ppm (16.7 and 8.3 Hz). These protons showed HMBCs with C-3a, C-8b, C-2 and C-1′ respectively at 162.7, 135.6, 50.2 and 136.2 ppm. The H-2 appears as a 1H triplet at 5.05 ppm (J = 8.3 Hz), which shows HMBCs (Fig. 3) with the C-3a at 162.7 ppm, C-3 at 45.7 ppm and both C-2′ and C-6′ at 129.0 ppm. Thus **3** could be readily distinguished from its regioisomer **3′**, since the latter should give two singlets for the methine hydrogens of the thiene ring. The structure of the thienoquinolines **3** was further confirmed by a single crystal X-ray crystallographic study of **3f** (Fig. 4) [32].

The regioselectivity of the reaction could be rationalized by the mechanism depicted in Scheme 3. Presumably, the transformation is triggered by the condensation of **1** and **2** affording the imine **4**, which tautomerises to furnish the enamine **5**, whose subsequent annulation affords **3**. The observed regioselectivity is ascribable to the stabilization of the enamine **5** by conjugative interaction of the enamine function with sulfur, which is not possible for the regioisomeric enamine **5′** (Scheme 3). Probably, enamine intermediate **5** alone (and not **5′**) is formed in this reaction that gives only **3**. That



Scheme 1. Design concept and retrosynthesis of diarylthienoquinolines **3**.

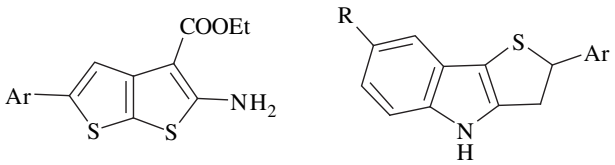
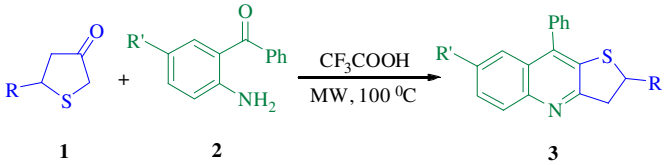


Fig. 2. Structure of hybrid heterocycles with antimycobacterial activities.



Scheme 2. Synthesis of 2,9-diaryl-2,3-dihydrothieno[3,2-*b*]quinolines **3**.

Table 1
Yields and antimycobacterial activities of **3**.

Compd.	R	R′	Reaction time (min)		Yield of **3** (%)		MIC (μM)	
			Thermal[a] (100 °C)	MW[b] (100 °C)	Thermal[c]	MW[c]	MTB	MDR-TB
3a	C_6H_5	H	120	30	80	98	36.82	–[d]
3b	*p*-MeC_6H_4	H	120	30	62	85	35.36	–[d]
3c	*p*-ClC_6H_4	H	120	30	74	87	16.72	–[d]
3d	*p*-FC_6H_4	H	150	30	68	82	8.76	–[d]
3e	*p*-$Pr^iC_6H_4$	H	150	30	49	60	32.76	–[d]
3f	*o*,*p*-$Cl_2C_6H_3$	H	120	30	61	75	3.82	15.30
3g	*o*-ClC_6H_4	H	150	30	58	70	8.39	-[d]
3h	*m*-$O_2NC_6H_4$	H	180	30	82	90	4.06	8.14
3i	1-naphthyl	H	120	30	85	93	32.09	–[d]
3j	C_6H_5	Cl	120	30	78	98	16.71	–[d]
3k	*p*-MeC_6H_4	Cl	150	30	67	80	8.07	–[d]
3l	*p*-ClC_6H_4	Cl	120	30	70	78	3.82	3.82
3m	*p*-FC_6H_4	Cl	120	30	65	78	3.98	3.98
3n	*o*,*p*-$Cl_2C_6H_3$	Cl	150	30	60	65	0.90	1.76
3o	*o*-ClC_6H_4	Cl	150	30	70	76	3.82	1.91
3p	*m*-$O_2NC_6H_4$	Cl	180	30	81	88	1.86	0.95
3q	1-naphthyl	Cl	150	30	85	93	29.48	–[d]
3r	H	Cl	120	30	73	90	–[d]	–[d]
Isoniazid							0.36	11.38
Rifampicin							0.12	3.80
Ciprofloxacin							4.71	37.73
Ethambutol							7.64	61.18

[a] Heated on an oil bath at 100 °C.
[b] Microwave irradiation programmed at 100 °C, 1 bar, 40 W with high absorption level.
[c] Isolated yield after column chromatography.
[d] Not tested.

the regioselectivity does not arise from steric effect due to interaction between the aryl rings of the quinoline and the sulfur heterocyclic rings of **3′** is evident from the fact that even in the case of the Friedländer reaction of unsubstituted dihyrothiophen-3(2*H*)-one, where no steric effect can be expected from the other regioisomer of type **3′**, the regioisomer of the type **3**, *viz*. **3r** alone is formed.

3. Biological results and discussion

All the thienoquinolines **3a**–**3q** were screened for their *in vitro* antimycobacterial activity against MTB and MDR-TB by agar dilution method. The MIC is defined as the minimum concentration of the compound required to completely inhibit the bacterial growth. The determination of MIC values was performed in duplicate at 7.40 pH. The MDR-TB clinical isolate was resistant to isoniazid, rifampicin, ethambutol and ciprofloxacin. The MICs of the synthesized compounds and standard drugs are listed in Table 1.

In the first phase of screening against MTB, all the compounds showed promising *in vitro* activity against MTB with MIC in the range of 0.90–36.82 μM. Seven compounds (**3f**, **3h**, **3l**, **3m**–**3p**) with MIC of 3.82, 4.06, 3.82, 3.98, 0.90, 3.82 and 1.86 μM were more potent than first line anti-TB drug, ethambutol (MIC: 7.64 μM). 7-chloro-2-(2,4-dichlorophenyl)-9-phenyl-2,3-dihydrothieno[3,2-*b*]quinoline (**3n**) is the most active compound with MIC of 0.90 μM against MTB. This compound **3n** is 5 and 8 times more active than ciprofloxacin and ethambutol respectively, while 3 and 8 times less active than isoniazid and rifampicin respectively.

Subsequently, seven compounds (**3f**, **3h**, **3l**–**3p**) of high activity against MTB were evaluated against MDR-TB. These compounds inhibited MDR-TB with MIC ranging from 0.95 to >15.30 μM (Table 1). All the seven compounds were found to be more active, 4–64.4 times than ethambutol (MIC: 61.18 μM). Six compounds (**3h**, **3l**, **3m**–**3p**) with MIC of 8.14, 3.82, 3.98, 1.76, 1.91 and 0.95 μM respectively displayed greater activity against MDR-TB than the anti-TB drug isoniazid (MIC: 11.38 μM), while three compounds **3n**–**3p** with MIC of 1.76, 1.91 and 0.95 μM are more potent against MDR-TB than rifampicin (MIC: 3.80 μM). 7-chloro-2-(3-nitrophenyl)-9-phenyl-2,3-dihydrothieno-[3,2-*b*]quinoline **3p** displayed maximum activity *in vitro* with MIC of 0.95 μM against MDR-TB, being 40, 12 and 4 times more potent than ciprofloxacin, isoniazid and rifampicin respectively.

A clear trend that emerges from the antimycobacterial activity data of the thienoquinolines against both MTB and MDR-TB is that

Table 2
Optimization of the microwave reaction by varying the amount of TFA and temperature.

S.No.	Temperature (°C)	Molar equiv. of TFA	Irradiation Time (min)[a]	Yield (%)[b]
1	100	0.2	30	38
2	100	0.5	30	45
3	100	1.0	30	80
4	100	1.5	30	98
5	60	1.5	150	60
6	80	1.5	120	62
7	90	1.5	60	78

[a] Maximum power of 40 W at very high absorption level was employed.
[b] Isolated yield after purification by column chromatography.

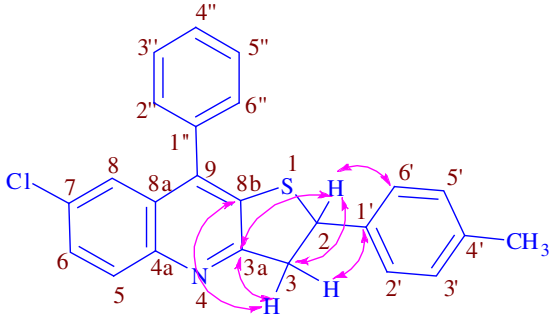


Fig. 3. Selected HMBCs in **3k**.

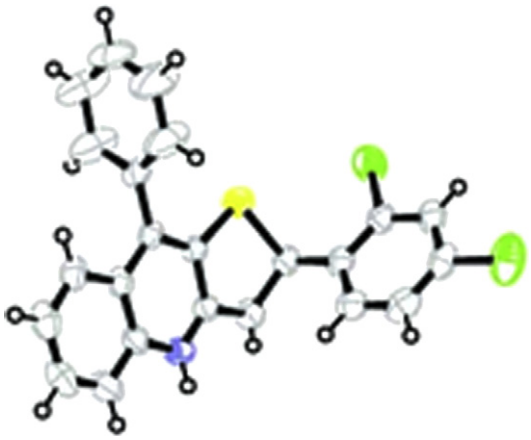


Fig. 4. ORTEP diagram of **3f**.

the thienoquinolines with chlorine at the 7-position displayed significantly greater activity than the corresponding unsubstituted compounds. It is found that the thienoquinolines with electron withdrawing groups like halogen or nitro group in the phenyl ring of the thiophene ring of **3** showed better activity than that with electron donating groups like alkyl. Disubstitution in the phenyl ring enhances the activity many-fold relative to monosubstitution.

4. Conclusion

In conclusion, a facile regioselective synthesis of 2,9-diaryl-2,3-dihydrothieno[3,2-*b*]quinolines by the Friedländer annulation of 2-aminobenzophenones with 5-aryldihydro-3(2*H*)-thiophenones in the presence of triflouroacetic acid is described under thermal as well as solvent-free microwave irradiation with higher yields being realised from the latter. These 2,9-diaryl-2,3-dihydrothieno[3,2-*b*]quinolines also displayed good *in vitro* antimycobacterial activity against both MTB and MDR-TB. The synthesis of a library of compounds belonging to the thieno[3,2-b]quinoline motif to facilitate in depth structure-activity investigations is currently underway in our research group.

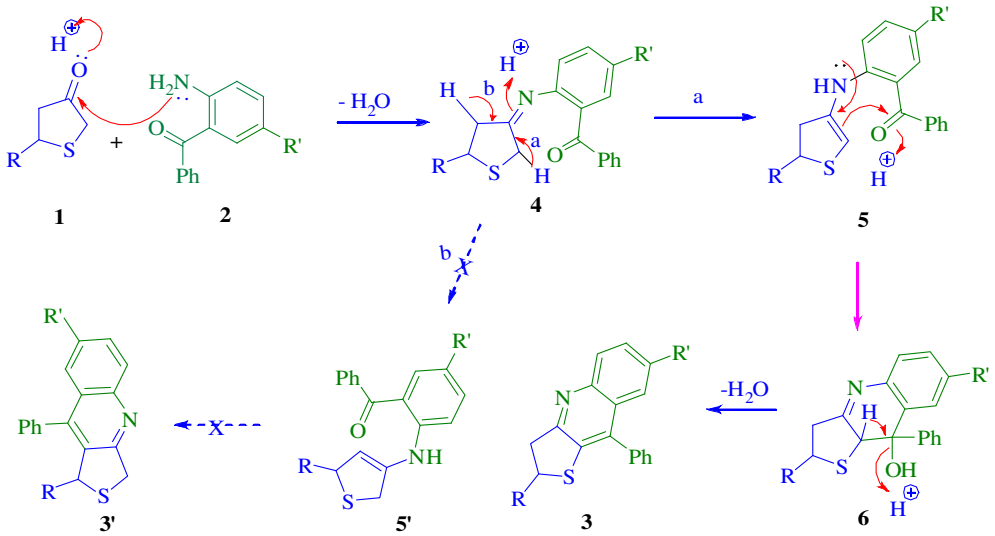
5. Experimental

The melting points were measured in open capillary tubes and are uncorrected. The ^{1}H, ^{13}C and the 2D NMR spectra were recorded on a Bruker (Avance) 300 MHz NMR instrument using TMS as internal standard and $CDCl_3$ as solvent. Chemical shifts are given in parts per million (δ-scale) and the coupling constants are given in Hertz. Silica gel-G plates (Merck) were used for TLC analysis with a mixture of petroleum ether (60–80 °C) and ethyl acetate as eluent. 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.

5.1. General procedure for synthesis of 2,9-diaryl-2,3-dihydrothieno[3,2-b]quinolines (3)

Conventional method: a mixture of 5-aryltetrahydro-3-thiophenone **1** (1 mmol), 2-aminobenzophenone **2** (1 mmol) and trifluoroacetic acid (1.5 mmol) was heated on an oil bath under solvent-free conditions with stirring at 100 °C for the time given in Table 1. After completion of the reaction as indicated by TLC, the reaction mixture was neutralized by the addition of an aqueous $NaHCO_3$ solution, the product was filtered and washed with water (2 × 10 ml). The crude product was purified by a short column chromatography on silica gel employing ethyl acetate-petroleum ether (1:4 v/v) as eluent to obtain pure thienoquinoline **3**.

Under microwave irradiation: a mixture of 5-aryltetrahydro-3-thiophenone (1 mmol), 2-aminobenzophenone (1 mmol) and triflouroacetic acid (1.5 mmol) was taken in a 10 ml quartz vial, sealed and placed in a Biotage microwave oven. The vial was subjected to microwave irradiation, programmed at 100 °C, 40 W, 1 bar pressure and very high absorption level for the time given in Table 1. After a period of 1–2 min, the temperature reached a plateau, 100 °C, and remained constant. After gas jet cooling to room temperature (3 min), the reaction mixture was neutralized with $NaHCO_3$, extracted with CH_2Cl_2 (2 × 5 mL), dried over $MgSO_4$ and concentrated *in vacuo* to give the crude product which was purified as done for the thermal reaction.



Scheme 3. Proposed mechanism for the regioselective formation of **3**.

*5.1.1. 2,9-Diphenyl-2,3-dihydrothieno[3,2-b]quinoline (**3a**)*

Pale yellow solid; Yield: 98%, m.p. 156–157 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.89 (m, 2H, 3-CH_2), 5.07 (t, 1H, J = 8.1 Hz, H-2), 7.25–7.34 (m, 3H, Ar-H), 7.43–7.51 (m, 8H, Ar-H), 7.62 (t, 2H, J = 8.6 Hz, Ar-H), 8.02 (d, 1H, J = 8.1 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 45.7, 50.3, 124.5, 126.4, 126.6, 127.1, 127.9, 128.5, 128.7, 128.9, 129.0, 134.0, 136.8, 138.6, 140.7, 146.4, 162.2. Anal. Calcd for $C_{23}H_{17}NS$: C, 81.38; H, 5.05; N, 4.13%. Found C, 81.44; H, 5.12; N, 4.19%.

*5.1.2. 2-(4-Methylphenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3b**)*

Pale yellow solid; Yield: 85%, m.p. 136–137 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 2.30 (s, 3H, CH_3), 3.80 (dd, 1H, J = 16.5, 8.5 Hz, H-3), 3.91 (dd, 1H, J = 16.5, 8.5 Hz, H-3), 5.04 (t, 1H, J = 8.5 Hz, H-2), 7.11 (d, 2H, J = 7.8 Hz, Ar-H), 7.31 (d, 2H, J = 7.8 Hz, Ar-H), 7.38–7.63 (m, 8H, Ar-H), 8.11 (d, 1H, J = 8.7 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 21.1, 45.8, 50.2, 124.6, 126.4, 126.6, 127.0, 127.9, 128.5, 128.8, 128.9, 129.1, 129.4, 134.2, 136.8, 137.6, 137.7, 138.5, 146.4, 162.3. Anal. Calcd for $C_{24}H_{19}NS$: C, 81.55; H, 5.42; N, 3.96%. Found C, 81.62; H, 5.47; N, 4.01%.

*5.1.3. 2-(4-Chlorophenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3c**)*

Pale yellow solid; Yield: 87%, m.p. 174–175 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.74 (dd, 1H, J = 16.8, 8.1 Hz, H-3), 3.93 (dd, 1H, J = 16.8, 8.1 Hz, H-3), 4.99 (t, 1H, J = 8.1 Hz, H-2), 7.25 (d, 2H, J = 8.4 Hz, Ar-H), 7.33 (d, 2H, J = 8.4 Hz, Ar-H), 7.39–7.62 (m, 8H, Ar-H), 8.01 (d, 1H, J = 8.4 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 45.7, 49.5, 124.6, 126.5, 126.6, 128.0, 128.5, 128.6, 128.9, 129.0, 133.5, 133.6, 136.7, 138.8, 139.4, 146.5, 161.7. Anal. Calcd for $C_{23}H_{16}ClNS$: C, 73.88; H, 4.31; N, 3.75%. Found C, 73.92; H, 4.35; N, 3.79%.

*5.1.4. 2-(4-Fluorophenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3d**)*

Pale yellow solid; Yield: 82%, m.p. 162–163 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.81 (dd, 1H, J = 16.7, 8.1 Hz, H-3), 3.98 (dd, 1H, J = 16.7, 8.1 Hz, H-3), 5.08 (t, 1H, J = 8.1 Hz, H-2), 7.03 (t, 2H, J = 7.5 Hz, Ar-H), 7.42–7.68 (m, 10H, Ar-H), 8.06 (d, 1H, J = 8.1 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 46.0, 49.6, 115.5, 115.8, 124.6, 126.5, 126.6, 128.0, 128.6, 128.7, 128.8, 128.9, 129.0, 133.7, 136.6, 136.8, 138.7, 146.5, 161.9. Anal. Calcd for $C_{23}H_{16}FNS$: C, 77.28; H, 4.51; N, 3.92%. Found C, 77.34; H, 4.57; N, 3.95%.

*5.1.5. 2-(4-Isopropylphenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3e**)*

Pale yellow solid; Yield: 60%, m.p. 125–126 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 1.26 (d, 6H, J = 6.9 Hz, 2CH_3), 2.91 (septet, 1H, J = 6.9 Hz, CH), 3.86 (dd, 1H, J = 16.6, 9.0 Hz, H-3), 3.96 (dd, 1H, J = 16.6, 8.4 Hz, H-3), 5.10 (t, 1H, J = 8.4 Hz, H-2), 7.21 (d, 2H, J = 8.4 Hz, Ar-H), 7.40 (d, 2H, J = 8.4 Hz, Ar-H), 7.46–7.62 (m, 6H, Ar-H), 7.65 (t, 2H, J = 8.4 Hz, Ar-H), 8.07 (d, 1H, J = 8.4 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 23.9, 33.7, 45.8, 50.2, 124.6, 126.4, 126.6, 126.8, 127.1, 127.9, 128.5, 128.7, 128.9, 129.1, 134.2, 136.8, 137.9, 138.5, 146.4, 148.7, 162.4. Anal. Calcd for $C_{26}H_{23}NS$: C, 81.85; H, 6.08; N, 3.67%. Found C, 81.91; H, 6.14; N, 3.71%.

*5.1.6. 2-(2,4-Dichlorophenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3f**)*

Pale yellow solid; Yield: 75%, m.p. 164–165 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.69 (dd, 1H, J = 16.8, 8.4 Hz, H-3), 4.03 (dd, 1H, J = 16.8, 8.4 Hz, H-3), 5.33 (dd, 1H, J = 8.4, 5.1 Hz, H-2), 7.06–7.13 (m, 1H, Ar-H), 7.34–7.48 (m, 7H, Ar-H), 7.60 (t, 2H, J = 7.5 Hz, Ar-H), 8.02 (d, 1H, J = 8.1 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 43.9, 45.1, 124.5, 126.5, 126.6, 127.5, 127.8, 128.1, 128.2, 128.5, 128.6, 128.8, 129.0, 129.4, 132.8, 133.9, 136.5, 137.4, 139.3, 146.5, 161.5. Anal. Calcd for $C_{23}H_{15}Cl_2NS$: C, 67.65; H, 3.70; N, 3.43%. Found C, 67.70; H, 3.74; N, 3.48%.

*5.1.7. 2-(2-Chlorophenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3g**)*

Pale yellow solid; Yield: 70%, m.p. 121–122 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.82 (dd, 1H, J = 16.8, 8.1 Hz, H-3), 4.10 (dd, 1H, J = 16.8, 8.1 Hz, H-3), 5.46 (dd, 1H, J = 8.1, 5.4 Hz, H-2), 7.11–7.18 (m, 2H, Ar-H), 7.29–7.63 (m, 8H, Ar-H), 7.68 (d, 1H, J = 8.1 Hz, Ar-H), 8.13 (d, 1H, J = 8.1 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 43.8, 45.4, 124.3, 126.2, 126.3, 127.0, 127.1, 127.7, 128.4, 128.6, 128.7, 128.8, 129.4, 133.0, 133.1, 136.5, 138.8, 138.9, 146.3, 161.7. Anal. Calcd for $C_{23}H_{16}ClNS$: C, 73.88; H, 4.31; N, 3.75%. Found C, 73.94; H, 4.36; N, 3.83%.

*5.1.8. 2-(3-Nitrophenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3h**)*

Pale yellow solid; Yield: 90%, m.p. 153–154 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.82 (dd, 1H, J = 16.7, 8.0 Hz, H-3), 4.06 (dd, 1H, J = 16.7, 8.0 Hz, H-3), 5.14 (t, 1H, J = 8.0 Hz, H-2), 7.42–7.68 (m, 8H, Ar-H), 7.78 (d, 1H, J = 7.8 Hz, Ar-H), 8.06 (d, 1H, J = 8.1 Hz, Ar-H), 8.14–8.17 (m, 1H, Ar-H), 8.34 (d, 1H, J = 1.8 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 45.7, 49.1, 122.3, 122.9, 124.7, 126.6, 126.7, 128.2, 128.7, 129.0, 129.9, 132.9, 133.2, 136.6, 139.2, 143.3, 146.7, 148.3, 161.0. Anal. Calcd for $C_{23}H_{16}N_2O_2S$: C, 71.85; H, 4.19; N, 7.29%. Found C, 71.91; H, 4.23; N, 7.34%.

*5.1.9. 2-(1-Naphthyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3i**)*

Pale yellow solid; Yield: 93%, m.p. 194–195 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 4.06–4.21 (m, 2H, 3-CH_2), 5.84 (t, 1H, J = 7.5 Hz, H-2), 7.41–7.71 (m, 11H, Ar-H), 7.79 (t, 2H, J = 8.1 Hz, Ar-H), 7.90 (d, 1H, J = 7.8 Hz, Ar-H), 8.13 (d, 1H, J = 8.4 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 44.1, 45.8, 122.8, 123.5, 124.5, 125.4, 125.8, 126.4, 126.5, 126.6, 127.9, 128.4, 128.5, 128.7, 128.9, 129.0, 130.9, 133.7, 133.9, 136.2, 136.7, 139.0, 146.5, 162.3. Anal. Calcd for $C_{27}H_{19}NS$: C, 83.26; H, 4.92; N, 3.60%. Found C, 83.30; H, 4.88; N, 3.64%.

*5.1.10. 7-Chloro-2,9-diphenyl-2,3-dihydrothieno[3,2-b]quinoline (**3j**)*

Pale yellow solid; Yield: 98%, m.p. 179–180 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.79 (dd, 1H, J = 16.8, 8.3 Hz, H-3), 3.91 (dd, 1H, J = 16.8, 8.3 Hz, H-3), 5.06 (t, 1H, J = 8.3 Hz, H-2), 7.24–7.34 (m, 3H, Ar-H), 7.40–7.57 (m, 8H, Ar-H), 7.93 (d, 1H, J = 8.7 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 45.7, 50.4, 123.4, 127.1, 127.4, 128.0, 128.6, 128.9, 129.0, 130.4, 132.4, 135.4, 136.2, 137.6, 140.4, 144.8, 162.6. Anal. Calcd for $C_{23}H_{16}ClNS$: C, 73.88; H, 4.31; N, 3.75%. Found C, 73.94; H, 4.36; N, 3.82%.

*5.1.11. 7-Chloro-2-(4-methylphenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3k**)*

Pale yellow solid; Yield: 80%, m.p. 115–116 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 2.32 (s, 3H, CH_3), 3.78 (dd, 1H, J = 16.7, 8.3 Hz, H-3), 3.90 (dd, 1H, J = 16.7, 8.3 Hz, H-3), 5.05 (t, 1H, J = 8.3 Hz, H-2), 7.12 (d, 2H, J = 7.8 Hz, Ar-H), 7.31 (d, 2H, J = 8.1 Hz, Ar-H), 7.44–7.64 (m, 7H, Ar-H), 7.94 (d, 1H, J = 9.0 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 21.1, 45.7, 50.2, 123.4, 127.0, 127.4, 128.3, 128.5, 128.8, 129.0, 129.4, 130.4, 132.3, 135.6, 136.2, 137.4, 137.5, 137.8, 144.8, 162.7. Anal. Calcd for $C_{24}H_{18}ClNS$: C, 74.31; H, 4.68; N, 3.61%. Found C, 74.36; H, 4.73; N, 3.65%.

*5.1.12. 7-Chloro-2-(4-chlorophenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3l**)*

Pale yellow solid; Yield: 78%, m.p. 119–120 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.71 (dd, 1H, J = 16.8, 8.1 Hz, H-3), 3.90 (dd, 1H, J = 16.8, 8.1 Hz, H-3), 5.00 (t, 1H, J = 8.1 Hz, H-2), 7.25 (d, 2H, J = 8.4 Hz, Ar-H), 7.32 (d, 2H, J = 8.4 Hz, Ar-H), 7.40–7.56 (m, 7H, Ar-H), 7.92 (d, 1H, J = 9.0 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 45.6, 49.5, 123.4, 127.4, 128.5, 128.7, 128.9, 129.0, 130.4, 132.5, 133.7, 135.0, 136.0, 137.8, 139.1, 144.9, 162.1. Anal. Calcd for $C_{23}H_{15}Cl_2NS$: C, 67.65; H, 3.70; N, 3.43%. Found C, 67.74; H, 3.78; N, 3.48%.

*5.1.13. 7-Chloro-2-(4-fluorophenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3m**)*

Pale yellow solid; Yield: 78%, m.p. 158–159 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.77 (dd, 1H, J = 16.8, 8.3 Hz, H-3), 3.94 (dd, 1H, J = 16.8, 8.3 Hz, H-3), 5.07 (t, 1H, J = 8.3 Hz, H-2), 7.02 (t, 2H, J = 8.4 Hz, Ar-H), 7.39–7.61 (m, 8H, Ar-H), 7.97 (d, 1H, J = 9.0 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 45.8, 49.6, 115.5, 115.8, 123.4, 127.4, 128.7, 128.8, 128.9, 129.0, 130.4, 132.5, 135.1, 136.1, 136.2, 136.3, 137.7, 144.9, 162.2. Anal. Calcd for $C_{23}H_{15}ClFNS$: C, 70.49; H, 3.86; N, 3.57%. Found C, 70.52; H, 3.94; N, 3.64%.

*5.1.14. 7-Chloro-2-(2,4-dichlorophenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3n**)*

Pale yellow solid; Yield: 65%, m.p. 145–146 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.72 (dd, 1H, J = 17.1, 8.5 Hz, H-3), 4.06 (dd, 1H, J = 17.1, 8.5 Hz, H-3), 5.40 (dd, 1H, J = 8.5, 5.4 Hz, H-2), 7.17–7.20 (m, 1H, Ar-H), 7.41–7.60 (m, 9H, Ar-H), 7.97 (d, 1H, J = 8.7 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 43.9, 45.3, 123.5, 127.4, 127.7, 128.2, 128.6, 128.9, 129.0, 129.6, 129.9, 130.4, 132.7, 134.0, 134.1, 134.4, 136.0, 137.5, 138.4, 144.9, 161.9. Anal. Calcd for $C_{23}H_{14}Cl_3NS$: C, 62.39; H, 3.19; N, 3.16%. Found C, 62.46; H, 3.25; N, 3.23%.

*5.1.15. 7-Chloro-2-(2-chlorophenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3o**)*

Pale yellow solid; Yield: 76%, m.p. 134–135 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.74 (dd, 1H, J = 17.1, 8.3 Hz, H-3), 4.03 (dd, 1H, J = 17.1, 8.3 Hz, H-3), 5.44 (dd, 1H, J = 8.3, 5.7 Hz, H-2), 7.15–7.18 (m, 2H, Ar-H), 7.33–7.65 (m, 9H, Ar-H), 7.95 (d, 1H, J = 8.7 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 43.9, 45.7, 123.4, 127.2, 127.3, 128.1, 128.6, 128.9, 129.7, 130.4, 132.4, 133.3, 134.6, 136.0, 138.0, 138.7, 144.8, 162.3. Anal. Calcd for $C_{23}H_{15}Cl_2NS$: C, 67.65; H, 3.70; N, 3.43%. Found C, 67.73; H, 3.76; N, 3.48%.

*5.1.16. 7-Chloro-2-(3-nitrophenyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3p**)*

Pale yellow solid; Yield: 88%, m.p. 160–161 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.79 (dd, 1H, J = 16.8, 8.0 Hz, H-3), 4.03 (dd, 1H, J = 16.8, 8.0 Hz, H-3), 5.15 (t, 1H, J = 8.0 Hz, H-2), 7.44–7.61 (m, 7H, Ar-H), 7.77 (d, 1H, J = 7.5 Hz, Ar-H), 7.96 (d, 1H, J = 7.5 Hz, Ar-H), 8.13–8.16 (m, 1H, Ar-H), 8.32 (d, 1H, J = 1.5 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 45.5, 49.1, 122.3, 123.0, 123.5, 127.3, 128.8, 128.9, 129.0, 129.1, 129.9, 130.5, 132.6, 133.2, 134.3, 135.9, 138.1, 143.0, 145.0, 148.3, 161.4. Anal. Calcd for $C_{23}H_{15}ClN_2O_2S$: C, 65.95; H, 3.61; N, 6.69%. Found C, 66.01; H, 3.69; N, 6.74%.

*5.1.17. 7-Chloro-2-(1-naphthyl)-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3q**)*

Pale yellow solid; Yield: 93%, m.p. 222–223 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 4.02–4.17 (m, 2H, 3-CH_2), 5.84 (t, 1H, J = 7.5 Hz, H-2), 7.41–7.63 (m, 10H, Ar-H), 7.75 (d, 1H, J = 7.2 Hz, Ar-H), 7.81 (d, 1H, J = 8.1 Hz, Ar-H), 7.89–7.92 (m, 1H, Ar-H), 8.01 (d, 1H, J = 8.7 Hz, Ar-H), 8.10 (d, 1H, J = 8.1 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 44.0, 45.9, 122.7, 123.4, 123.5, 125.4, 125.9, 126.6, 127.5, 128.6, 128.9, 129.0, 129.1, 130.5, 130.9, 132.4, 133.9, 135.2, 135.9, 136.1, 138.1, 144.9, 162.7. Anal. Calcd for $C_{27}H_{18}ClNS$: C, 76.49; H, 4.28; N, 3.30%. Found C, 76.53; H, 4.25; N, 3.28%.

*5.1.18. 7-Chloro-9-phenyl-2,3-dihydrothieno[3,2-b]quinoline (**3r**)*

Pale yellow solid; Yield: 90%, m.p. 138–139 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 3.34 (t, 2H, J = 7.5 Hz, CH_2, CH_2), 3.58 (t, 2H, J = 7.5 Hz, CH_2), 7.39–7.57 (m, 5H, Ar-H), 7.90 (d, 1H, J = 8.7 Hz, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 29.4, 37.4, 123.2, 127.1, 128.4, 128.7, 128.8, 128.9, 130.3, 132.2, 135.3, 136.3, 137.7, 144.5, 163.5. Anal. Calcd for $C_{17}H_{12}ClNS$: C, 68.56; H, 4.06; N, 4.70%. Found C, 68.63; H, 4.12; N, 4.77%.

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Appendix. Supporting information

Supplementary data associated with this article can be found in online version at doi:10.1016/j.ejmech.2009.11.011.

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