

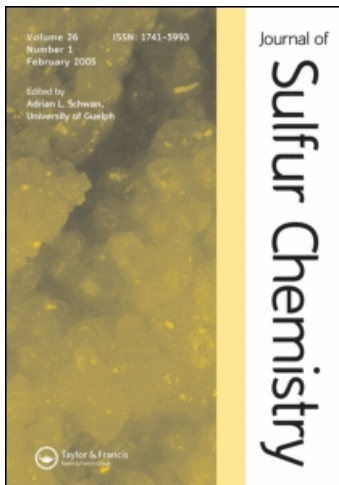
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F. M. Moghaddam^a; H. Saeidian^a; Z. Mirjafary^a; S. Taheri^a

^a Department of Chemistry, Sharif University of Technology, Tehran, Iran

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RESEARCH ARTICLE

A simple and efficient synthesis of new indeno- and naphtho-fused thiophenes using arylthioacetamides

F. M. MOGHADDAM*, H. SAEIDIAN, Z. MIRJAFARY and S. TAHERI

Department of Chemistry, Sharif University of Technology, PO Box 11365-9516, Tehran, Iran

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New indeno- and naphtho-fused thiophenes were synthesized from reaction of 2-bromo-1-indanone and 2-bromo-1-tetralone with arylthioacetamides in good yields.

Keywords: Tetra substituted thiophenes; 2-Aminothiophenes; Thioamides; Interamolecular condensation

1. Introduction

Thiophene derivatives are important chemical building blocks. A variety of molecules containing the thiophene ring display a wide range of biological activity and find applications as pharmaceuticals [1]. Highly substituted thiophenes have attracted a great deal of interest, due to their presence in natural products [2], as novel conducting polymers [3, 4], isosteric replacements for phenyl group in medicinal chemistry [5] and as optical chromophores [6].

However, the synthesis of highly substituted thiophenes is restricted by the lack of enough available methods to construct the desired ring bearing functionality in a controlled fashion. The most convenient method for preparing thiophene with a high degree of functionality is by the Gewald method in which elemental sulfur is reacted with an activated acetonitrile and an aldehyde, ketone or 1,3-dicarbonyl compound in the presence of a base [7]. A modification of the Gewald method has been reported in which an alkoxyacetone is reacted with ethyl cyanoacetate, sulfur and morpholine producing most sources of alkoxy thiophene derivatives in poor yields (19–39%) [8].

Recently syntheses of substituted thiophenes based on Pd-catalyzed cycloisomerization of *Z*-2-en-4-yne-1-thiols have been reported [9]. But this procedure suffers from poor substituents tolerance and reaction needs an expensive catalyst. New and improved methodologies have also been developed [10, 11].

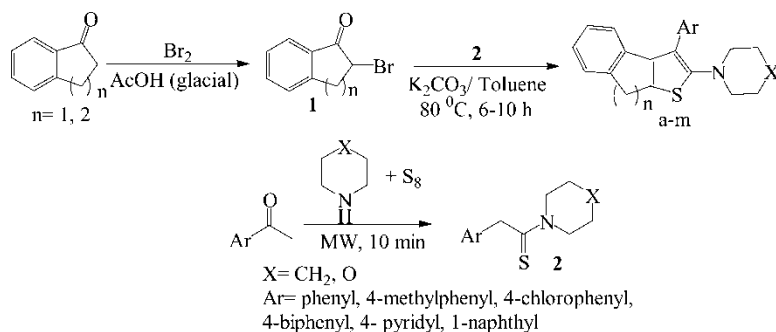
Thioamides have been used as useful synthons in the substrates of heterocycles [12–14]. The tertiary thioamides having an activated methylene group could react with α -halo carbonyl

*Corresponding author. Email: matloubi@sharif.edu

compounds in the presence of a base leading to the synthesis of 2-aminothiophene derivatives. For example, thioacetamides in benzene react with α -bromoketones in the presence of DBU yielding 2-amino-3-nitrothiophene [15]. These reactions are restricted to the tertiary nitro thioacetamides as starting materials. Earlier we have reported a simple microwave-induced method for the preparation of thiomorpholides including aryl thioacetomorpholides [16, 17]. The availability of such thiomorpholides provided a unique opportunity of examining their synthetic utility.

On the other hand, recently a versatile one-pot synthesis of trisubstituted thiophenes from thiomorpholides via S-Claisen rearrangement has been reported [18]. In continuation of the research in this area [19, 20] and aiming to find new biologically active thiophene heterocyclic compounds, such as 2-aminothiophene derivatives, the authors report their results on the reaction of aryl thioamides with 2-bromo-1-indanone and 2-bromo-1-tetralone (scheme 1).

When thioamide **2** was treated with **1** in toluene and stirred for 6–10 h at 80 °C in the presence of anhydrous K_2CO_3 , the highly substituted 2-aminothiophenes were obtained in good yields. Several examples have been investigated and table 1 summarizes the results.



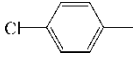
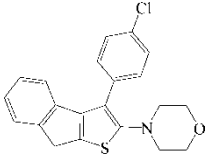
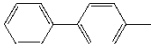
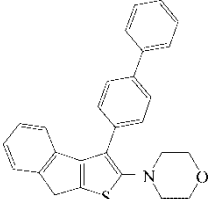
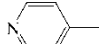
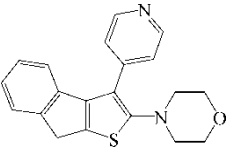

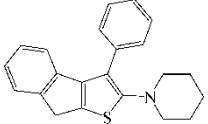
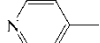
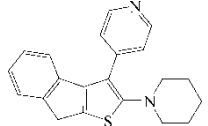

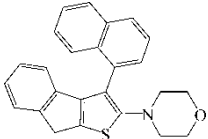

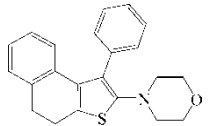
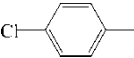
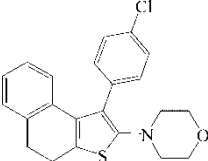
SCHEME 1

Table 1. Synthesis of indane and tetralone fused thiophenes using thioamides.

Entry	<i>n</i>	X	Ar	Product	Time (h)	Yield (%) ^a
a	1	O			8	64
b	1	O			8	57

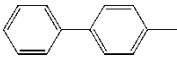
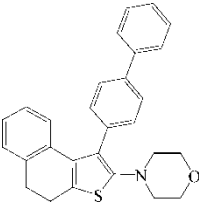

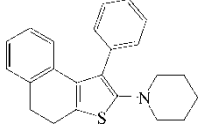

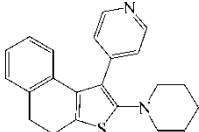
(continued)

Table 1. Continued.

Entry	<i>n</i>	X	Ar	Product	Time (h)	Yield (%) ^a
c	1	O			8	64
d	1	O			8	72
e	1	O			6	75
f	1	CH ₂			7	77
g	1	CH ₂			7	84
h ^b	1	O			10	42
i	2	O			7	63
j	2	O			7	60

(continued)

Table 1. Continued.

Entry	<i>n</i>	X	Ar	Product	Time (h)	Yield (%) ^a
k	2	O			7	71
l	2	CH ₂			6	83
m	2	CH ₂			6	90

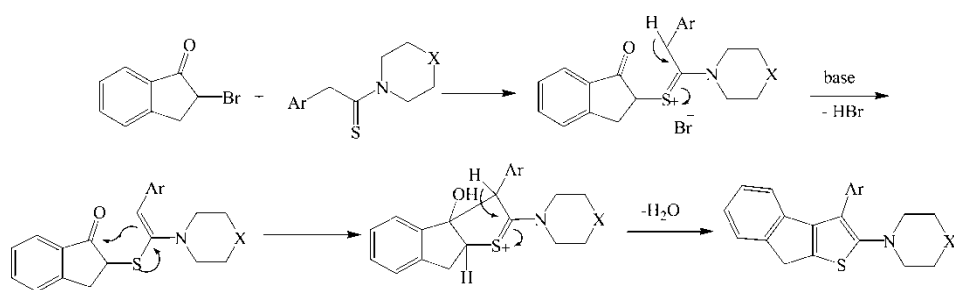
^aIsolated yield.^bReaction temperature was 110 °C.

A mechanism is proposed for the reaction course and is shown in scheme 2. Arylthioacetamides undergoes first an S-alkylation with haloketone, then subsequent intermolecular aldol-type condensation and finally dehydration which could be supported by the literature analogy [21].

The products were characterized by spectroscopic methods (¹H-NMR, ¹³C-NMR, DEPT 90, DEPT 135 and GC-MS). Interestingly, in ¹H-NMR of compounds (**a–g**) the benzylic CH₂ group appears as a singlet for all compounds except for compound **h** which shows an AB Pople's system (figure 1).

This observation may suggest that one of the hydrogens of CH₂ should be spatially closer to the naphthyl group and thus it experiences more anisotropic effect from naphthyl group. The simulation performed by "CS Chem 3D Pro 5.0", confirms this suggestion (figure 1).

Synthesis of tetra-substituted thiophenes proceeded more conveniently using thioamides with heteroaromatic substitutes such as pyridyl (entries **3e**, **3g**, **3m**, table 1) in high yields and short reaction times. Thiophenes contained the pyridyl substituent are interesting compounds in organic chemistry.



SCHEME 2

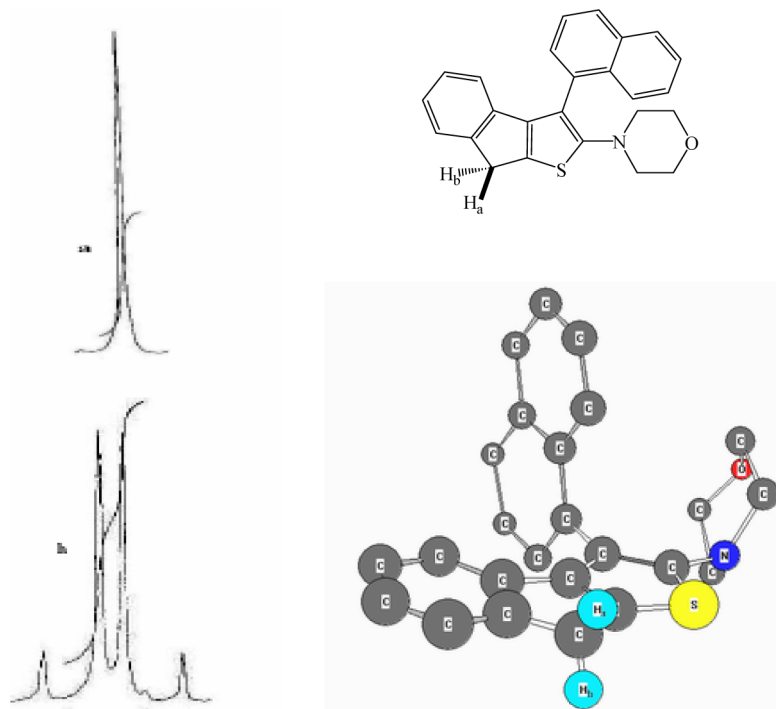


Figure 1. The $^1\text{H-NMR}$ of benzylic CH_2 for compounds **a** and **h** with structure of compound **h**.

In conclusion, we have developed an efficient and simple method for preparation of highly substituted 2-aminoindane and 2-aminotetralane fused thiophenes. The generality of the method has been demonstrated by the successful conversion of thirteen substrates into fully substituted 2-aminothiophenes in good yields. These materials have the potential to be used as new reagents for synthesis of heterocyclic compounds. The methodology described here seems to be the simplest one for the synthesis of these compounds.

2. Experimental

The compounds all gave satisfactory spectroscopic data. A Bruker (DRX-500 Avanes) NMR was used to record the $^1\text{H-NMR}$, $^{13}\text{C-NMR}$ and DEPT 90 and DEPT 135 spectra. All NMR spectra were determined in CDCl_3 at ambient temperature. Melting points were determined on a Buchi B540 apparatus. GC-MS (EI), 70 eV, HP6890 Column: HP-5 (30 m \times 0.25 mm \times 0.2 μm MSD: HP5793) was used to record the mass spectra.

2.1 Preparation of thiophenes (3a–3m)

To a stirred solution of a thioamide [16, 17] (4 mmol) in toluene (5 ml), anhydrous K_2CO_3 (0.552 g, 4 mmol) was added. Then a solution of 2-bromo-1-indanone or 2-bromo-1-tetralone [22] (4 mmol) in toluene (2 ml) was added dropwise over 10 min. The reaction mixture was heated at 80°C for about 6–10 hours. Then, the solvent was evaporated and the residue was subjected to column chromatography (EtOAc/Hexane; 1:4 on silica gel) to obtain pure products.

2.2 Spectroscopic data for compounds (3a–3m)

2.2.1 4-(3-Phenyl-8*H*-indeno[2,1-*b*]thien-2-yl)morpholine (3a). Light yellow solid. Mp: 156–158 °C. ¹H-NMR (500 MHz, CDCl₃): δ = 7.65 (d, *J* = 8.9 Hz, 2H), 7.53–7.49 (m, 3H), 7.43 (t, *J* = 5.7 Hz, 1H), 7.21–7.15 (m, 3H), 3.86 (s, 2H), 3.72 (t, *J* = 4.6 Hz, 4H), 2.69 (t, *J* = 4.6 Hz, 4H). ¹³C-NMR (125 MHz, CDCl₃): δ = 156.1(C), 146.3(C), 143.9(C), 140.3(C), 135.6(C), 135.5(C), 130.2(CH), 128.8(CH), 127.6(CH), 126.7(CH), 125.4(C), 124.9(CH), 124.7(CH), 119.9(CH), 67.1(CH₂), 54.2(CH₂), 35.5(CH₂). MS (EI) *m/z*: 333 (M⁺, 100), 274 (30), 202 (7), 136 (7).

2.2.2 4-(3-*p*-tolyl-8*H*-indeno[2,1-*b*]thien-2-yl)morpholine (3b). Light yellow solid. Mp: 159–161 °C. ¹H-NMR (500 MHz, CDCl₃): δ = 7.52 (d, *J* = 7.8 Hz, 2H), 7.48 (t, *J* = 7.8 Hz, 1H), 7.31 (d, *J* = 7.8 Hz, 2H), 7.23–7.14 (m, 3H), 3.84 (s, 2H), 3.71 (t, *J* = 4.5 Hz, 4H), 2.94 (t, *J* = 4.5 Hz, 4H), 2.48 (s, 3H). ¹³C-NMR (125 MHz, CDCl₃): δ = 156.8(C), 146.3(C), 144.0(C), 140.5(C), 137.0(C), 135.2(C), 132.6(C), 130.1(CH), 129.4(CH), 126.6(CH), 125.2(C), 124.7(CH), 124.5(CH), 119.9(CH), 67.3(CH₂), 54.1(CH₂), 35.5(CH₂), 21.8(CH₃). MS (EI) *m/z*: 347 (M⁺, 100), 288 (27), 256 (12), 202 (9), 136 (11).

2.2.3 4-{3-(4-chloro Phenyl)-8*H*-indeno[2,1-*b*]thien-2-yl}morpholine (3c). Light yellow solid. Mp: 192–194 °C. ¹H-NMR (500 MHz, CDCl₃): δ = 7.58 (d, *J* = 6.5 Hz, 2H), 7.51–7.48 (m, 3H), 7.20–7.16 (m, 3H), 3.58 (s, 2H), 3.72 (t, *J* = 4.6 Hz, 4H), 2.93 (t, *J* = 4.6 Hz, 4H). ¹³C-NMR (125 MHz, CDCl₃): δ = 157.4(C), 146.2(C), 143.5(C), 140.1(C), 135.7(C), 134.1(C), 133.2(C), 131.5(CH), 128.9(CH), 126.7(CH), 124.9(CH), 124.7(CH), 124.1(C), 119.7(CH), 67.2(CH₂), 54.2(CH₂), 35.5(CH₂). MS (EI) *m/z*: 369 (M+2, 35), 367 (M⁺, 100), 308 (25), 274 (12), 256 (12), 202 (9).

2.2.4 4-{3-(1-biphenyl-4-yl)-8*H*-indeno[2,1-*b*]thien-2-yl}morpholine (3d). Light yellow solid. Mp: 168–170 °C. ¹H-NMR (500 MHz, CDCl₃): δ = 7.78–7.72 (m, 6H), 7.55–7.41 (m, 4H), 7.32 (t, *J* = 5.5 Hz, 1H), 7.18–7.16 (m, 2H), 3.87 (s, 2H), 3.74 (t, *J* = 4.6 Hz, 4H), 2.98 (t, *J* = 4.6 Hz, 4H). ¹³C-NMR (125 MHz, CDCl₃): δ = 157.3(C), 146.3(C), 143.8(C), 141.1(C), 140.4(C), 140.0(C), 135.5(C), 134.7(C), 130.6(CH), 129.3(CH), 127.7(CH), 127.4(CH), 127.2(CH), 126.7(CH), 124.9(C), 124.8(CH), 124.6(CH), 120.0(CH), 67.3(CH₂), 54.2(CH₂), 35.6(CH₂). MS (EI) *m/z*: 409 (M⁺, 100), 350 (28), 323 (11), 256 (10).

2.2.5 4-{3-(4-pyridyl)-8*H*-indeno[2,1-*b*]thien-2-yl}morpholine (3e). Light yellow solid. Mp: 183–185 °C. ¹H-NMR (500 MHz, CDCl₃): δ = 8.76 (d, *J* = 6 Hz, 2H), 7.61 (d, *J* = 6 Hz, 2H), 7.51 (t, *J* = 3 Hz, 1H), 7.30–7.18 (m, 3H), 3.85 (s, 2H), 3.73 (t, *J* = 4.6 Hz, 4H), 2.93 (t, *J* = 4.6 Hz, 4H). ¹³C-NMR (125 MHz, CDCl₃): δ = 158.9(C), 150.3(CH), 146.2(C), 144.0(C), 142.8(C), 139.7(C), 136.5(C), 126.8(CH), 125.1(CH), 125.0(CH), 124.8(CH), 122.7(C), 119.7(CH), 67.1(CH₂), 54.4(CH₂), 35.5(CH₂). MS (EI) *m/z*: 334 (M⁺, 100), 275 (30), 248 (20), 137 (9).

2.2.6 4-(3-Phenyl-8*H*-indeno[2,1-*b*]thien-2-yl)piperidine (3f). Light yellow solid. Mp: 125–127 °C. ¹H-NMR (500 MHz, CDCl₃): δ = 7.68 (d, *J* = 6.9 Hz, 2H), 7.54–7.49 (m, 3H), 7.41 (t, *J* = 6.2 Hz, 1H), 7.28 (t, *J* = 4.1 Hz, 1H), 7.18 (d, *J* = 4.2 Hz, 1H), 7.15 (d, *J* = 4.1 Hz, 1H), 3.85 (s, 2H), 2.91 (t, *J* = 5.3 Hz, 4H), 1.62–1.50 (m, 6H). ¹³C-NMR (125 MHz, CDCl₃): δ = 146.4(C), 143.6(C), 140.7(C), 136.1(C), 134.9(C), 130.4(C),

130.2(CH), 128.5(CH), 127.1(CH), 126.6(CH), 124.8(CH), 124.4(CH), 124.3(C), 119.9(CH), 55.5(CH₂), 35.6(CH₂), 26.4(CH₂), 24.2(CH₂). MS (EI) m/z: 331 (M⁺, 100), 288 (7), 274 (27), 254 (19), 221 (20), 202 (14).

2.2.7 4-{3-(4-pyridyl)-8H-indeno[2,1-b]thien-2-yl}piperidine (3g). Light yellow solid. Mp: 193–195 °C. ¹H-NMR (500 MHz, CDCl₃): δ = 8.76 (m, 2H), 7.65 (d, *J* = 4.0 Hz, 2H), 7.50 (t, *J* = 3.5 Hz, 1H), 7.29 (t, *J* = 4.9 Hz, 1H), 7.19 (m, 2H), 3.84 (s, 2H), 2.88 (t, *J* = 5.2 Hz, 4H), 1.59 (d, *J* = 4.8 Hz, 4H), 1.52 (d, *J* = 4.8 Hz, 2H). ¹³C-NMR (125 MHz, CDCl₃): δ = 161.0(C), 150.1(CH), 146.3(C), 144.3(C), 142.6(C), 140.0(C), 135.9(C), 126.8(CH), 125.1(CH), 124.9(CH), 124.8(CH), 121.7(C), 119.7(CH), 55.7(CH₂), 35.5(CH₂), 26.3(CH₂), 24.1(CH₂). MS (EI) m/z: 332 (M⁺, 100), 275 (23), 254 (17), 221 (15).

2.2.8 4-{3-(1-naphthyl)-8H-indeno[2,1-b]thien-2-yl}morpholine (3h). Light yellow oil. ¹H-NMR (500 MHz, CDCl₃): δ = 7.97 (m, 2H), 7.84 (d, *J* = 8.5 Hz, 1H), 7.64–7.61 (m, 2H), 7.54 (t, *J* = 8.1 Hz, 1H), 7.45 (d, *J* = 7.3 Hz, 1H), 7.40 (t, *J* = 8.1 Hz, 1H), 7.07 (t, *J* = 7.4 Hz, 1H), 6.90 (t, *J* = 7.6 Hz, 1H), 6.34 (d, *J* = 7.6 Hz, 1H), 3.94 (d, *J* = 20 Hz, 1H, AB system), 3.88 (d, *J* = 20 Hz, 1H, AB system), 3.52–3.49 (m, 4H), 2.93–2.89 (m, 4H). ¹³C-NMR (125 MHz, CDCl₃): δ = 157.8(C), 146.1(C), 145.2(C), 140.3(C), 134.2(C), 134.1(C), 133.7(C), 132.5(C), 128.6(CH), 128.4(CH), 128.2(CH), 126.9(CH), 126.6(CH), 126.4(CH), 126.3(CH), 125.9(CH), 124.6(CH), 124.4(CH), 122.0(C), 119.7(CH), 67.1(CH₂), 53.8(CH₂), 35.6(CH₂). MS (EI) m/z: 383 (M⁺, 100), 324 (26), 310 (5), 297 (10), 277 (4), 265 (4), 252 (7).

2.2.9 4-(1-phenyl-4,5-dihydronaphtho[2,1-b]thien-2-yl)morpholine (3i). Light yellow solid. Mp: 165–167 °C. ¹H-NMR (500 MHz, CDCl₃): δ = 7.43–7.36 (m, 5H), 7.23 (d, *J* = 7.4 Hz, 1H), 7.07 (t, *J* = 7.4 Hz, 1H), 6.90 (t, *J* = 7.7 Hz, 1H), 6.66 (d, *J* = 7.8 Hz, 1H), 3.66 (t, *J* = 4.5 Hz, 4H), 3.04 (t, *J* = 6.8 Hz, 2H), 2.90 (t, *J* = 6.8 Hz, 2H), 2.85 (t, *J* = 4.5 Hz, 4H). ¹³C-NMR (125 MHz, CDCl₃): δ = 151.9(C), 137.1(C), 136.1(C), 132.7(C), 132.6(C), 131.8(C), 130.5(CH), 129.6(C), 128.7(CH), 128.3(CH), 127.2(CH), 126.4(CH), 126.1(CH), 125.2(CH), 67.3(CH₂), 54.2(CH₂), 30.7(CH₂), 24.9(CH₂). MS (EI) m/z: 347 (M⁺, 100), 345 (10), 288 (16), 260 (5), 215 (3).

2.2.10 4-{1-(4-chlorophenyl)-4,5-dihydronaphtho[2,1-b]thien-2-yl)morpholine (3j). Light yellow solid. Mp: 131–133 °C. ¹H-NMR (500 MHz, CDCl₃): δ = 7.45–7.35 (m, 4H), 7.23 (d, *J* = 7.3 Hz, 1H), 7.08 (t, *J* = 7.4 Hz, 1H), 6.94 (t, *J* = 7.5 Hz, 1H), 6.95 (d, *J* = 7.8 Hz, 1H), 3.67 (t, *J* = 4.5 Hz, 4H), 3.02 (t, *J* = 6.8 Hz, 2H), 2.88 (t, *J* = 6.8 Hz, 2H), 2.83 (t, *J* = 4.5 Hz, 4H). ¹³C-NMR (125 MHz, CDCl₃): δ = 171.7(C), 152.3(C), 136.2(C), 135.4(C), 133.0(C), 132.5(C), 132.4(C), 131.9(CH), 130.0(C), 128.9(CH), 128.4(CH), 126.5(CH), 126.3(CH), 125.1(CH), 67.3(CH₂), 54.3(CH₂), 30.6(CH₂), 24.8(CH₂). MS (EI) m/z: 383 (M+2, 33), 381 (M⁺, 100), 379 (9), 322 (9), 288 (14), 258 (10).

2.2.11 4-(1-Biphenyl-4-yl-4,5-dihydronaphtho[2,1-b]thien-2-yl)morpholine (3k). Light yellow solid. Mp: 191–193 °C. ¹H-NMR (500 MHz, CDCl₃): δ = 7.75 (d, *J* = 7.8 Hz, 2H), 7.70 (d, *J* = 8.2 Hz, 2H), 7.59–7.47 (m, 4H), 7.41 (t, *J* = 7.4 Hz, 1H), 7.25 (d, *J* = 7.4 Hz, 1H), 7.07 (t, *J* = 7.4, 1H), 6.92 (t, *J* = 7.6, 1H), 6.77 (d, *J* = 7.8, 1H), 3.68 (t, *J* = 4.4 Hz, 4H), 3.05 (t, *J* = 6.8 Hz, 2H), 2.90–2.87 (m, 6H). ¹³C-NMR (125 MHz, CDCl₃): δ = 152.1(C), 144.9(C), 141.1(C), 139.6(C), 136.2(C), 136.0(C), 132.7(C), 132.0(C), 130.9(CH), 129.2(CH), 129.1(C), 128.3(CH), 127.7(CH), 127.3(CH), 127.2(CH), 126.5(CH),

126.1(CH), 125.3(CH), 67.3(CH₂), 54.2(CH₂), 30.6(CH₂), 24.9(CH₂). MS (EI) m/z: 423 (M⁺, 100), 421 (14), 364 (14).

2.2.12 4-(1-phenyl-4,5-dihydronaphtho[2,1-*b*]thien-2-yl) piperidine (3l). Light yellow solid. Mp: 110–112 °C. ¹H-NMR (500 MHz, CDCl₃): δ = 7.43–7.33 (m, 5H), 7.23 (d, *J* = 7.3 Hz, 1H), 7.05 (t, *J* = 7.4 Hz, 1H), 6.90 (t, *J* = 7.7 Hz, 1H), 6.67 (d, *J* = 7.8 Hz, 1H), 3.03 (t, *J* = 6.8 Hz, 2H), 2.86 (t, *J* = 6.8 Hz, 2H), 2.80 (t, *J* = 4.6 Hz, 4H), 1.56–1.46 (m, 6H). ¹³C-NMR (125 MHz, CDCl₃): δ = 153.9(C), 137.4(C), 136.2(C), 133.0(C), 132.5(C), 131.5(C), 130.6(CH), 128.9(C), 128.5(CH), 128.3(CH), 127.0(CH), 126.5(CH), 125.9(CH), 125.2(CH), 55.6(CH₂), 30.8(CH₂), 26.4(CH₂), 25.0(CH₂), 24.3(CH₂). MS (EI) m/z: 345 (M⁺, 100), 343 (17), 288 (4), 215 (5).

2.2.13 4-(1-(4-pyridyl)-4,5-dihydronaphtho[2,1-*b*]thien-2-yl)piperidine (3m). Light yellow solid. Mp: 187–189 °C. ¹H-NMR (500 MHz, CDCl₃): δ = 8.65 (d, *J* = 6.0 Hz, 2H), 7.40 (d, *J* = 6.0 Hz, 2H), 7.24 (d, *J* = 7.4 Hz, 1H), 7.08 (t, *J* = 7.4 Hz, 1H), 6.93 (t, *J* = 7.6, 1H), 6.66 (d, *J* = 7.8 Hz, 1H), 3.01 (t, *J* = 6.8 Hz, 2H), 2.84 (t, *J* = 6.8 Hz, 2H), 2.78 (t, *J* = 4.6 Hz, 4H), 1.55–1.48 (m, 6H). ¹³C-NMR (125 MHz, CDCl₃): δ = 155.6(C), 150.1(CH), 145.5(C), 136.2(C), 132.8(C), 132.2(C), 131.8(C), 128.5(CH), 126.5(CH), 126.3(CH), 126.2(CH), 125.6(C), 125.2(CH), 55.9(CH₂), 30.6(CH₂), 26.3(CH₂), 24.9(CH₂), 24.1(CH₂). MS (EI) m/z: 346 (M⁺, 100), 345 (22), 289 (4), 230 (6).

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