Phosphine-catalyzed [3+2] cycloaddition of Morita—Baylis—Hillman carbonates to isothiocyanates in the synthesis of adamantane-containing trisubstituted aminothiophenes*

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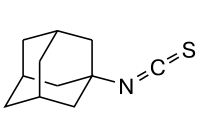
An addition of the Morita—Baylis—Hillman (MBH) carbonates to adamantane-containing isothiocyanates was studied. The MBH carbonates react with 1-(4-isothiocyanatophenyl)-adamantane in the presence of triphenylphosphine to give 5-aryl-substituted adamantane-containing 2-amino-4-methoxycarbonylthiophenes in 31—59% yields, while 1- and 2-isothiocyanatoadamantanes were found unreactive in this reaction.

Key words: thiophene, organocatalysts, phosphines, adamantane, isothiocyanates, Morita—Baylis—Hillman carbonates.

In recent years, organocatalysis, the use of small molecules to catalyze organic transformations, have been widely developed. In this field of chemistry, special attention is given to the reactions catalyzed by phosphines[1,2] and *N*-heterocyclic carbenes (NHCs).[3,4] It is of note that phosphines and NHCs were previously widely known to the researchers mainly as the ligands for metal complex catalysis. The recent studies showed that phosphine-catalyzed reactions could be an efficient tool for the synthesis of a wide range of organic compounds,[2,5—7] *e.g.*, carbo- and heterocycles.[8] For instance, phosphine-catalyzed reactions provide a one-step access to polysubstituted thiophenes.[9—11] The thiophene derivatives possess different valuable properties, they are extensively studied as the biologically active compounds[12—18] and the components for organic electronic materials.[19,20] Adamantane derivatives exhibit different biological activities since rigid lipophilic adamantane moiety enhances cell membrane penetration. Therefore, the development of new approaches of introduction of the adamantane moiety into organic molecules is of interest for the search of potential drugs.[21—23] The aim of the present work is the study of phosphine-catalyzed approach to substituted 2-aminothiophenes bearing the adamantane moiety.

Previously, we investigated different synthetic approaches to a wide range of adamantane-containing heterocyclic compounds by palladium-[24] and copper-catalyzed[25] amination of the corresponding halo derivatives. We demonstrated the possibility to synthesize *N*-substituted adamantane-containing succinimides by phosphine-catalyzed cycloaddition of ethyl buta-2,3-dienoate to the appropriate maleimides.[26] Catalytic amination of halothiophenes has a number of significant limitations, *e.g.*, by the nature of the amination agent and the substituents.[27—30] Therefore, we synthesized adamantane-containing 2-aminothiophenes by the phosphine catalyzed cycloaddition of the Morita—Baylis—Hillman (MBH) carbonates to isothiocyanates.

To study the possibility to synthesize 2-amino thiophenes by phosphine-catalyzed cycloaddition, we selected isothiocyanates **1**—**3**. In these compounds, the isothio-



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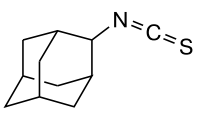

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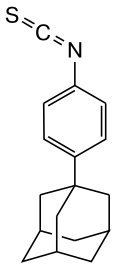

3

* Dedicated to Academician of the Russian Academy of Sciences V. N. Charushin on the occasion of his 70th birthday.

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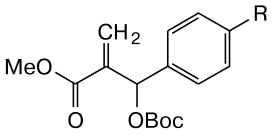
cyanate moiety is attached to different positions of the adamantane core either directly (compounds **1** and **2**) or *via para*-phenylene spacer (compound **3**).

To reveal the effects of the substituents on the course of the organocatalytic reactions, we employed the 4-substituted MBH carbonates **4a—e** as the substrates.

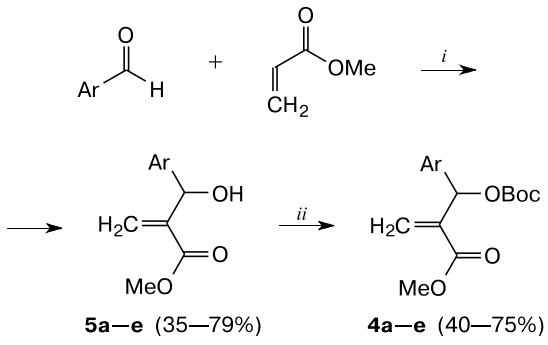


4a—e

R = Cl (**a**), H (**b**), Br (**c**), F (**d**), OMe (**e**)

The MBH carbonates were synthesized from the corresponding benzaldehydes and methyl acrylate in two steps by common approach[31] (Scheme 1). The first step was the Morita—Baylis—Hillman reaction catalyzed by DABCO, namely, the addition to the aldehyde carbonyl group to give the corresponding allylic alcohols **5a—e**. The synthesized compounds were protected by the *tert*-butoxycarbonyl group under the standard conditions that gave compounds **4a—e** in the yields from moderate to high. Both stages are complicated by polymerization of the product in the presence of DABCO and DMAP. This leads to a significant dependence of the product yield on the nature of the substituent in the aromatic cycle that determines the tendency of the compound to polymerization.

Scheme 1



5a—e (35—79%) **4a—e** (40—75%)

Ar = 4-ClC_6H_4 (**a**), Ph (**b**), 4-BrC_6H_4 (**c**), 4-FC_6H_4 (**d**), 4-$MeOC_6H_4$ (**e**)

Reagents and conditions: *i*. DABCO, MeOH, ~20 °C, 72 h; *ii*. Boc_2O, DMAP, CH_2Cl_2, ~20 °C, 3 h.

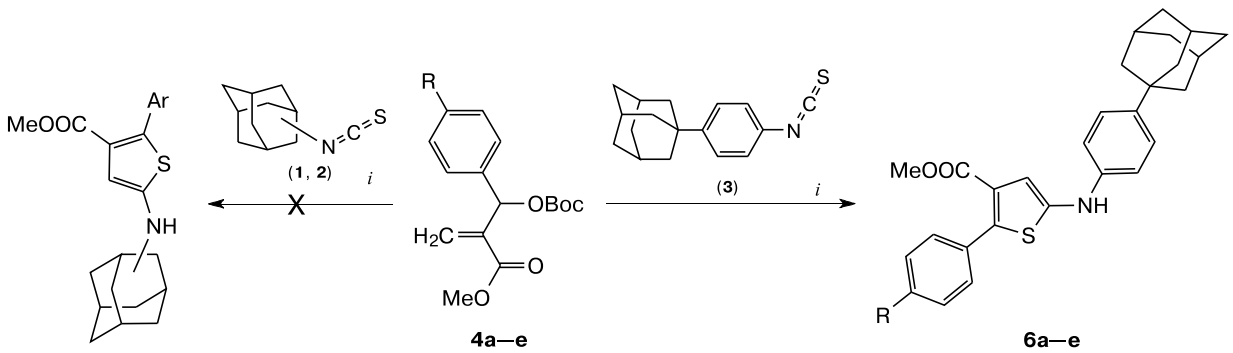
The conditions of the cycloaddition were chosen based on the published data.[10] The reaction was carried out in refluxing toluene in the presence of phosphine (20 mol.%) as a catalyst using 0.4 mol L^{-1} concentrations of the reagents (Scheme 2).

In the case of isothiocyanates **1** and **2**, under any conditions used no formation of the target products was observed. The reaction in the presence of either triphenylphosphine or other phosphines (tributylphosphine, methyldiphenylphosphine) proceeded with complete conversions of the MBH carbonates and partial conversion of isothiocyanates but gave only complex mixtures of the polymerization products.

In the case of isothiocyanate **3**, the target aminothiophenes **6** were obtained in the presence of triphenylphosphine (Table 1, entries *1* and *6—9*). The use of other phosphines resulted only in the polymerization products (entries *2* and *3*) but gave no target products. No improvement in the yield of the target products was observed under microwave irradiation conditions (entries *4* and *5*). The reaction was completed within 1.5 h, the products were isolated by preparative column chromatography. The target thiophenes **6a,b,d,e** were obtained in 48—59% yields (entries *1*, *6*, *8*, and *9*) and only in the case of 4-bromo-substituted thiophene **6c** the isolated yield was lower (31%, entry *7*). Thus, the nature of the substituent in the phenyl ring has no substantial effect on the reaction course.

The same reaction conditions were used to synthesize 2-amino-4-acetylated thiophenes from the MBH carbon-

Scheme 2



4a—e **6a—e**

4, 6: R = Cl (**a**), H (**b**), Br (**c**), F (**d**), OMe (**e**)

Reagents and conditions: *i*. PPh_3 (20 mol.%), toluene, 110 °C, 1.5 h.

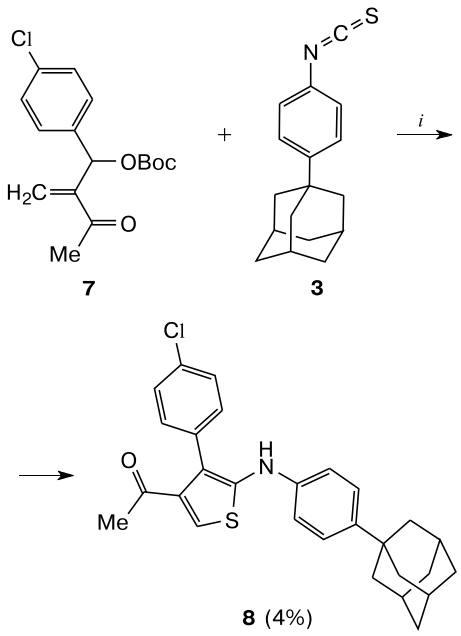
Table 1. Phosphine-catalyzed [3+2] cycloaddition of Morita—Baylis—Hillman carbonates to isothiocyanate **3**

Entry	R	Catalyst	MBH carbonate	Product	Yield (%)
1	Cl	PPh_3	**4a**	**6a**	53
2	Cl	PBu_3	**4a**	**6a**	0
3	Cl	$PMePh_2$	**4a**	**6a**	0
4[a]	Cl	PPh_3	**4a**	**6a**	50
5[b]	Cl	PPh_3	**4a**	**6a**	0
6	H	PPh_3	**4b**	**6b**	50
7	Br	PPh_3	**4c**	**6c**	31
8	F	PPh_3	**4d**	**6d**	59
9	OMe	PPh_3	**4e**	**6e**	48

[a] Reaction was carried out under microwave irradiation at 110 °C.
[b] Reaction was carried out under microwave irradiation at 70 °C.

ate **7** (Scheme 3), which was prepared by the known procedure.[9] However, this reaction gave only isomeric product **8** in very low yield of 4%.

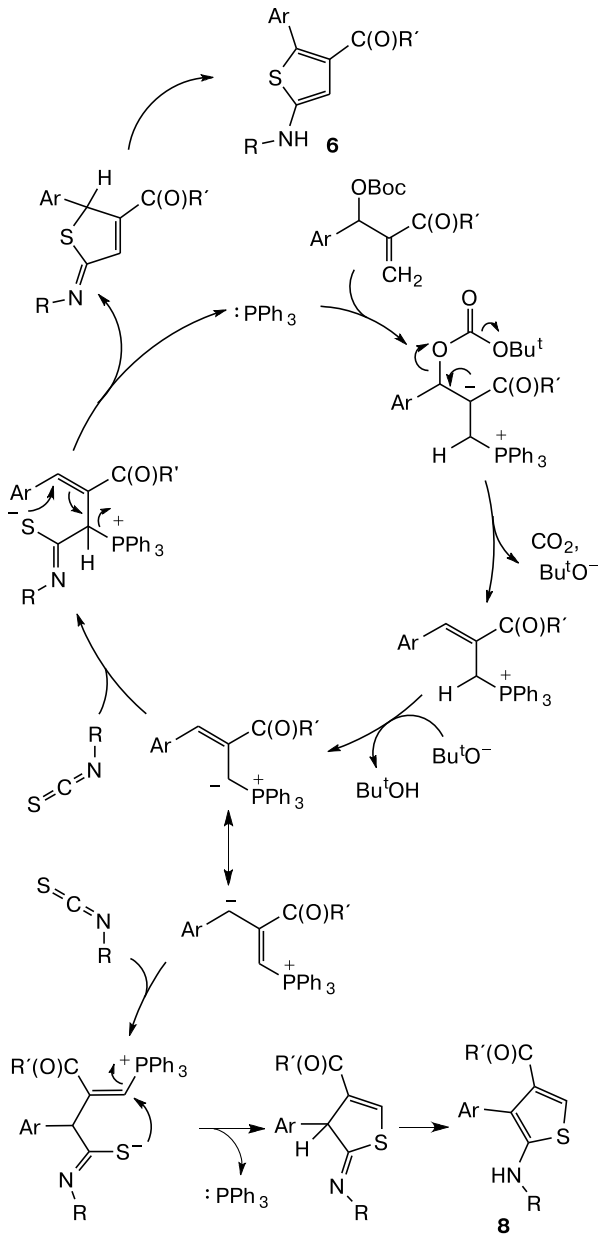
Scheme 3



Reagents and conditions: PPh_3 (20 mol.%), toluene, 110 °C, 1.5 h.

Formation of products **6** and **8** can be rationalized by analyzing the reaction mechanism (Scheme 4). Addition of phosphine to the MBH carbonate gave ylide that eliminated *tert*-butylcarbonate anion decomposing into CO_2 and *tert*-butylate anion. Further, the *tert*-butylate anion acted as a base that resulted in the formation of the conjugated ylide.[32] This intermediate has two nucleophilic centers, therefore two directions of the attack by isothiocyanate could be realized. Apparently, the reactivity of the nucleophilic centers strongly depends on the nature of the substituents. In the case of the MBH carbonates **4a—e**, the nucleophilic attack resulted in 5-aryl-2-aminothiophene **6**, while in the case of the MBH **7** another nucleophilic center is involved. It seems that the methyl group due to the proton transfer and formation of the stable enolate promoted the numerous side reactions.

Scheme 4



In summary, in the present work we revealed the possibilities and limitations of phosphine-catalyzed [3+2] cycloaddition in the synthesis of adamantane-

containing 5-aryl-4-methoxycarbonyl-2-aminothiophenes. These peculiarities were used to synthesize a broader range of adamantane-containing thiophene derivatives.

Experimental

^{1}H and ^{13}C NMR spectra were run on a Bruker Avance-400 instrument (working frequencies of 400 (^{1}H) and 100 MHz (^{13}C)) at 298 K. ^{19}F NMR spectra were recorded with an Agilent 400-MR instrument (working frequency of 376 MHz). Matrix-assisted laser desorption/ionization (MALDI) mass spectrometry was performed with a Bruker Ultraflex-II instrument using 1,8,9-trihydroxyanthracene as a matrix and polyethylene glycols as the internal standards. Microwave experiments were carried out using an Anton Paar Monowave 400 high-performance microwave reactor. For preparative column chromatography (CC), Merck silica gel (40/60) was used. Adamantane-containing isothiocyanates **1—3** were synthesized from the corresponding amines as earlier described.[33] The Morita—Baylis—Hillman carbonates **4a—e** were synthesized from methyl acrylate and the appropriate aldehydes following the known procedure.[31] Compound **7** was synthesized in two steps from methyl vinyl ketone and 4-chlorobenzaldehyde as earlier described.[9] Triphenylphosphine (Aldrich) was used as purchased. Toluene was distilled over sodium metal under an argon atmosphere. Petroleum ether (PE), dichloromethane, and methanol were distilled prior to use.

Phosphine-catalyzed [3+2] cycloaddition of the Morita—Baylis—Hillman carbonate 4a—c to 1-(4-isothiocyanatophenyl)-adamantane 3 (general procedure). A two-neck flask equipped with a magnetic stirrer and a reflux condenser was charged with the MBH carbonate **4** (0.20 mmol), PPh_3 (20 mol.%, 11 mg, 0.04 mmol), and 1-(4-isothiocyanatophenyl)adamantane **3** (65 mg, 0.24 mmol). The flask was flushed with argon and anhydrous toluene (0.5 mL) was added under stream of argon. The mixture was stirred at 110 °C for 1.5 h under argon. The solvent was removed *in vacuo*, product was purified by CC using gradient elution with PE→PE—CH_2Cl_2 (4 : 1 → 1 : 1).

Methyl 5-{[4-(adamantan-1-yl)phenyl]amino}-2-(4-chlorophenyl)thiophene-3-carboxylate (6a) was synthesized from MBH carbonate **4a** (65.3 mg, 0.20 mmol) and compound **3** (65 mg, 0.24 mmol) in the presence of PPh_3 (10.5 mg, 0.04 mmol) in toluene (0.5 mL). Eluent for CC was PE—CH_2Cl_2 (2 : 1) → → PE—CH_2Cl_2 (1 : 1). Yield 50.6 mg (53%), yellow powder. M.p. 108—109 °C. ^{1}H NMR ($CDCl_3$), δ: 1.73—1.81 (m, 6 H, H_{Ad}); 1.88—1.91 (m, 6 H, H_{Ad}); 2.09 (br.s, 3 H, H_{Ad}); 3.73 (s, 3 H, OMe); 5.75 (br.s, 1 H, NH); 6.96 (d, 2 H, H_{Ar}, ^3J = 8.6 Hz), 7.03 (s, 1 H, H_{Thio}); 7.25—7.28 (m, 2 H, H_{Ar}); 7.33—7.36 (m, 2 H, H_{Ar}); 7.41—7.44 (m, 2 H, H_{Ar}). ^{13}C NMR ($CDCl_3$), δ: 28.9 (3 C, C_{Ad}); 35.6 (1 C, C_{Ad}); 36.7 (3 C, C_{Ad}); 43.3 (3 C, C_{Ad}); 51.6 (1 C, OMe); 115.2 (2 C), 117.9 (1 C), 125.8 (2 C), 128.1 (2 C), 128.4 (1 C), 131.1 (2 C), 131.5 (1 C), 132.2 (1 C), 134.3 (1 C), 141.8 (1 C), 144.2 (1 C), 145.5 (1 C), 163.5 (1 C, C(O)). MS (MALDI, PEG-300), found: *m/z* 478.163 $[M + H]^+$. Calculated for $C_{28}H_{29}ClNO_2S$: 478.161.

Methyl 5-{[4-adamantan-1-yl)phenyl]amino}-2-phenylthiophene-3-carboxylate (6b) was synthesized from MBH carbonate **4b** (58 mg, 0.2 mmol) and compound **3** (65 mg, 0.24 mmol) in the presence of PPh_3 (11 mg, 0.04 mmol) in toluene (0.5 mL). Eluent for CC was PE—CH_2Cl_2 (2 : 1) → PE—CH_2Cl_2 (1 : 1). Yield 44 mg (50%), yellow powder. M.p. 146—147 °C. ^{1}H NMR ($CDCl_3$), δ: 1.74—1.82 (m, 6 H, H_{Ad}); 1.89—1.91 (m, 6 H, H_{Ad}); 2.10 (br.s, 3 H, H_{Ad}); 3.73 (s, 3 H, OMe); 6.98 (d, 2 H, H_{Ar}, ^3J = 8.6 Hz); 7.07 (s, 1 H, H_{Thio}); 7.28 (d, 2 H, H_{Ar}, ^3J = 8.3 Hz); 7.37—7.42 (m, 3 H, H_{Ar}); 7.49—7.52 (m, 2 H, H_{Ar}); the NH group proton signal was not ambiguously attributed. ^{13}C NMR ($CDCl_3$), δ: 28.9 (3 C, C_{Ad}); 35.6 (1 C, C_{Ad}); 36.8 (3 C, C_{Ad}); 43.3 (3 C, C_{Ad}); 51.5 (1 C, OMe); 115.2 (2 C), 118.4 (1 C), 125.8 (2 C), 125.9 (1 C), 127.9 (2 C), 128.3 (1 C), 129.7 (2 C), 133.3 (1 C), 141.9 (1 C), 142.5 (1 C), 144.1 (1 C), 144.9 (1 C), 163.7 (1 C, C(O)). MS (MALDI, PEG-400), found: *m/z* 444.201 $[M + H]^+$. Calculated for $C_{28}H_{30}NO_2S$: 444.199.

Methyl 5-{[4-(adamantan-1-yl)phenyl]amino}-2-(4-bromophenyl)thiophene-3-carboxylate (6c) was synthesized from MBH carbonate **4c** (74 mg, 0.20 mmol) and compound **3** (65 mg, 0.24 mmol) in the presence of PPh_3 (11 mg, 0.04 mmol) in toluene 0.5 mL. Eluent for CC was PE—CH_2Cl_2 (1 : 1). Yield 32 mg (31%), yellow oil. ^{1}H NMR ($CDCl_3$), δ: 1.72—1.80 (m, 6 H, H_{Ad}); 1.87—1.89 (m, 6 H, H_{Ad}); 2.09 (br.s, 3 H, H_{Ad}); 3.73 (s, 3 H, OMe); 6.96 (d, 2 H, H_{Ar}, ^3J = 8.6 Hz); 7.03 (s, 1 H, H_{Thio}); 7.27 (m, 2 H, H_{Ar}); 7.36 (m, 2 H, H_{Ar}); 7.48—7.53 (m, 2 H, H_{Ar}); the NH group proton signal was not ambiguously attributed. ^{13}C NMR ($CDCl_3$), δ: 28.9 (3 C, C_{Ad}); 35.6 (1 C, C_{Ad}); 36.8 (3 C, C_{Ad}); 43.3 (3 C, C_{Ad}); 51.6 (1 C, OMe); 115.3 (2 C), 118.0 (1 C), 122.6 (1 C), 125.8 (2 C), 131.1 (2 C), 131.3 (2 C), 132.3 (1 C), 133.3 (1 C), 140.5 (1 C), 141.7 (1 C), 144.3 (1 C), 145.5 (1 C), 163.5 (1 C, C(O)). MS (MALDI, PEG-400), found: *m/z* 522.110 $[M + H]^+$. Calculated for $C_{28}H_{29}BrNO_2S$: 522.110.

Methyl 5-{[4-(adamantan-1-yl)phenyl]amino}-2-(4-fluorophenyl)thiophene-3-carboxylate (6d) was synthesized from MBH carbonate **4d** (62.0 mg, 0.20 mmol) and compound **3** (65 mg, 0.24 mmol) in the presence of PPh_3 (11 mg, 0.04 mmol) in toluene (0.5 mL). Eluent for CC was PE—CH_2Cl_2 (1 : 1). Yield 54.4 mg (59%), yellow powder. M.p. 162—163 °C. ^{1}H NMR ($CDCl_3$), δ: 1.76—1.84 (m, 6 H, H_{Ad}); 1.92—1.94 (m, 6 H, H_{Ad}); 2.13 (br.s, 3 H, H_{Ad}); 3.77 (s, 3 H, OMe); 5.71 (br.s, 1 H, NH); 6.99 (d, 2 H, H_{Ar}, ^3J = 8.6 Hz); 7.07—7.13 (m, 3 H, H_{Thio}, H_{Ar}); 7.30 (d, 2 H, H_{Ar}, ^3J = 8.6 Hz); 7.49—7.52 (m, 2 H, H_{Ar}). ^{13}C NMR ($CDCl_3$), δ: 29.0 (3 C, C_{Ad}); 35.6 (1 C, C_{Ad}); 36.8 (3 C, C_{Ad}); 43.3 (3 C, C_{Ad}); 51.6 (1 C, OMe); 115.0 (d, 2 C, $^2J_{C-F}$ = 21.9 Hz), 115.2 (2 C), 118.2 (1 C), 125.8 (2 C), 128.6 (1 C), 129.3 (1 C), 131.6 (d, 2 C, $^3J_{C-F}$ = 8.4 Hz), 132.2 (1 C), 141.3 (1 C), 143.1 (d, 1 C, $^1J_{C-F}$ = 225.9 Hz), 145.1 (1 C), 163.5 (1 C), 164.0 (1 C). ^{19}F NMR ($CDCl_3$), δ: −113.30 (tt, 1 F, J = 8.6 Hz, J = 5.4 Hz). MS (MALDI, PEG-400), found: *m/z* 462.191 $[M + H]^+$. Calculated for $C_{28}H_{29}FNO_2S$: 462.190.

Methyl 5-{[4-(adamantan-1-yl)phenyl]amino}-2-(4-methoxyphenyl)thiophene-3-carboxylate (6e) was synthesized from MBH carbonate **4e** (65 mg, 0.20 mmol) and compound **3** (65 mg, 0.24 mmol) in the presence of PPh_3 (11 mg, 0.04 mmol) in toluene (0.5 mL). Eluent for CC was PE—CH_2Cl_2 (1 : 1) → → CH_2Cl_2. Yield 45.4 mg (48%), yellow powder. M.p. 158—159 °C. ^{1}H NMR ($CDCl_3$), δ: 1.73—1.81 (m, 6 H, H_{Ad}); 1.89—1.91 (m, 6 H, H_{Ad}); 2.10 (br.s, 3 H, H_{Ad}); 3.74 (s, 3 H, OMe); 3.84 (s, 3 H, OMe); 5.66 (br.s, 1 H, NH); 6.90—6.96 (m, 4 H, H_{Ar}); 7.04 (s, 1 H, H_{Thio}); 7.26 (d, 2 H, H_{Ar}, ^3J = 8.6 Hz); 7.44 (d, 2 H, H_{Ar}, ^3J = 8.6 Hz). ^{13}C NMR ($CDCl_3$), δ: 28.9 (3 C, C_{Ad}); 35.6 (1 C, C_{Ad}); 36.8 (3 C, C_{Ad}); 43.3 (3 C, C_{Ad}); 51.5 (1 C, OMe); 55.3 (1 C, OMe); 113.4 (2 C), 114.9 (2 C), 118.7 (1 C), 125.3 (1 C), 125.6 (1 C), 125.7 (2 C), 131.0 (2 C),

142.2 (1 C), 143.0 (1 C), 143.8 (1 C), 144.2 (1 C), 159.7 (1 C, C—O); 163.7 (1 C, C(O)). MS (MALDI, PEG-400), found: *m/z* 474.210 $[M + H]^+$. Calculated for $C_{29}H_{32}NO_3S$: 474.195.

1-[5-{[4-(Adamantan-1-yl)phenyl]amino}-4-(4-chlorophenyl)thiophen-3-yl]ethanone (8) was synthesized from MBH carbonate **7** (62 mg, 0.20 mmol) and compound **3** (65 mg, 0.24 mmol) in the presence of PPh_3 (11 mg, 0.04 mmol) in toluene (0.5 mL). Eluent for CC was PE—CH_2Cl_2 (1 : 1). Yield 4 mg (4%), yellow oil. ^{1}H NMR ($CDCl_3$), δ: 1.72—1.80 (m, 6 H, H_{Ad}); 1.85—1.92 (m, 6 H, H_{Ad}); 2.09 (br.s, 3 H, H_{Ad}); 2.33 (s, 3 H, CH_3); 6.91 (d, 2 H, H_{Ar}, ^3J = 8.6 Hz); 7.18—7.20 (m, 2 H, H_{Ar}); 7.23—7.25 (m, 2 H, H_{Ar}); 7.36 (d, 2 H, H_{Ar}, ^3J = 8.6 Hz); 7.59 (s, 1 H, H_{Thio}); the NH group proton signal was not ambiguously attributed. ^{13}C NMR ($CDCl_3$), δ: 28.5 (1 C, CH_3); 28.9 (3 C, C_{Ad}); 36.7 (3 C, C_{Ad}); 43.3 (3 C, C_{Ad}); 115.5 (2 C), 123.8 (1 C), 125.8 (2 C), 128.8 (2 C), 131.2 (2 C), the quaternary carbon signals were nor attributed due to their low intensity. MS (MALDI, PEG-400), found: *m/z* 462.173 $[M + H]^+$. Calculated for $C_{28}H_{29}ClNOS$: 462.166.

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This work does not involve human participants and animal subjects.

The authors declare no conflicts of interests.

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