Synthesis of some thieno[3,2-*b*]thiophene derivatives via alkynylation reaction

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Abstract

Using the Sonogashira cross-coupling reactions synthesized seven new thieno[3,2-*b*]thiophen derivatives in moderate yield. The procedure was optimized and found that triphenylphosphine (0.2 eq.), Pladium diacetate (0.1 eq), copper (I) iodide (0.2 eq.), THF, *i*Pr_2NH was the best in these cases of **6a**, **6b**, **6c**, **6d** and **6e**. The structures of these compounds were elucidated by ^1H and ^{13}C NMR and mass spectral analysis.

Keywords. Thieno[3,2-*b*]thiophen, Sonogashira reaction, cross-coupling reaction.

1. INTRODUCTION

Thieno[3,2-*b*]thiophene (**TT**) is a core to construct organic semiconductors of different conjugation lengths [1, 2, 3,4]. The functionalization of thieno[3,2-*b*]thiophene in particular plays an important role in making organic materials, Figure 1. In 2006, McCulloch *et al.* reported a liquid-crystalline semiconducting polymer (PBTTT) containing thieno[3,2-*b*]thiophene moieties with a very high charge-carrier mobility (Figure 1) [5]. Recently, dinaphtho[2,3-*b*:2′,3′-*f*]thieno[3,2-*b*]thiophene (DNTT) and alkylated benzothieno[3,2-*b*][1]benzothiophene (C13BTBT) were shown to demonstrate a very high thin film mobility of 3.1 cm^2/Vs and 17.2 cm^2/Vs, respectively, in VD-OFETs [6,7]. Shi *et al.* reported Sn-TIPS as a new high performance semiconductor [8].

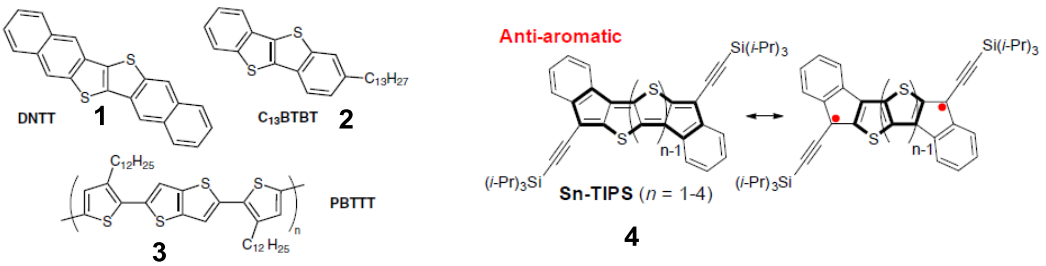


Figure 1: Some organic materials containing thieno[3,2-*b*]thiophene

Recently, the Sonogashira reaction of alkynes and **TT** has become one of the most popular C-C bond forming processes in organic synthesis that might fill up the band gap of organic semiconducting polymers, extension of the π system by increasing the conjugated length of the molecule is one of the most promising method. It helps not only decreased band gap but also increased charge carrier mobility [9-16]. In order to make a long conjugated system, palladium-assisted C−Br activation appears to be straightforward for functionalization into **TT**. Taking these facts into considerations, we report here new results that allows selective and controllable C-Br functionalization of 2,3,5,6-tetrabromthieno[3,2-*b*]thiophene to monoalkynyl derivatives.

2. EXPERIMENTAL

2.1. Experimental section

Solvents and other chemicals were purchased from Sigma-Aldrich, Merck were used as received, unless indicated. The ^1H NMR and ^{13}C NMR spectra were recorded on the Bruker Avance 500 NMR spectrometer in $CDCl_3$. Chemical-shift data for each

signal was reported in ppm units. Mass spectra were obtained from Mass Spectrometry Facility of The Vietnam Academy of Science and Technology on LC-MSD-Trap-SL spectrometer.

2.2. Synthetic procedure

2.2.1. General procedure: To the argon degassed solution of THF (6 mL) and iPr_2NH (6 mL) was added 2,3,5,6-tetrabromthieno[3,2-*b*]thiophene (114 mg, 1 eq.), Ph_3P (13.1 mg; 0.2 eq.), $Pd(OAc)_2$ (5.6 mg; 0.1 eq.), CuI (10 mg; 0.2 eq). The resulting solution was refluxed to dissolve all substrates and reagents. The the reaction solution was added slowly alkynes (1.2 eq.) then refluxed at 75 °C for 2-3 h. The progress of reaction was monitored by TLC (eluent: *n*-hexane). The mixture was concentrated *in vacou*. The products were purified with column chromatography.

*Synthesis of 2,3,6-tribromo-5-(2-p-tolylethynyl)thieno[3,2-b]thiophene (**6a**)*
Following the general procedure, using 2,3,5,6-tetrabromthieno[3,2-*b*]thiophene (114 mg, 0.25 mmol, 456 g/mol), Ph_3P (13.1 mg; 0.2 eq., 262 g/mol), $Pd(OAc)_2$ (5.6 mg; 0.1 eq., 225 g/mol), CuI (10 mg; 0.2 eq., 190 g/mol) and 1-ethynyl-4-methylbenzene (29.0 mg, 0.25 mmol, 116 g/mol) gave **6a** as a white powder (30 mg, 491 g/mol, 25 %), mp. 194 °C; ^{13}C NMR ($CDCl_3$, 125 MHz) δ (ppm): 139.6, 138.0, 131.7, 131.5, 129.2, 119.0, 114.8, 107.9, 107.1, 99.9, 80.4, 21.6.

*Synthesis of 2,3,6-tribromo-5-(2-(4-methoxyphenyl)ethynyl)thieno[3,2-b]thiophene (**6b**)*
Following the general procedure, using 2,3,5,6-tetrabromthieno[3,2-*b*]thiophene (114 mg, 0.25 mmol, 456 g/mol), Ph_3P (13.1 mg; 0.2 eq., 262 g/mol), $Pd(OAc)_2$ (5.6 mg; 0.1 eq., 225 g/mol), CuI (10 mg; 0.2 eq., 190 g/mol) and 1-ethynyl-4-methoxybenzene (33.0 mg, 0.25 mmol, 132 g/mol) gave **6b** as a white powder (32 mg, 507 g/mol, 25 %), mp. 182 °C. ^{13}C NMR ($CDCl_3$, 125 MHz) δ (ppm): 160.4, 138.0, 137.1, 133.2, 122.6, 114.6, 114.2, 107.6, 107.0, 99.9, 79.9, 55.3.

*Synthesis of 2,3,6-tribromo-5-(2-phenylethynyl)thieno[3,2-b]thiophene (**6c**)*
Following the general procedure, using 2,3,5,6-tetrabromthieno[3,2-*b*]thiophene (114 mg, 0.25 mmol, 456 g/mol), Ph_3P (13.1 mg; 0.2 eq., 262 g/mol), $Pd(OAc)_2$ (5.6 mg; 0.1 eq., 225 g/mol), CuI (10 mg; 0.2 eq., 190 g/mol) and 1-ethynylbenzene (25.5 mg, 0.25 mmol, 102 g/mol) gave **6c** as a white powder (50 mg, 477 g/mol, 42 %), mp. 190 °C. ^{13}C NMR ($CDCl_3$, 125 MHz) δ (ppm): 137.5, 131.6, 129.2, 128.5, 122.1, 122.0, 115.0, 108.2, 107.1, 99.6, 81.0.

*Synthesis of 2,3,6-tribromo-5-(2-m-tolylethynyl)thieno[3,2-b]thiophene (**6d**)*
Following the general procedure, using 2,3,5,6-tetrabromthieno[3,2-*b*]thiophene (114 mg, 0.25 mmol, 456 g/mol), Ph_3P (13.1 mg; 0.2 eq., 262 g/mol), $Pd(OAc)_2$ (5.6 mg; 0.1 eq., 225 g/mol), CuI (10 mg; 0.2 eq., 190 g/mol) and 1-ethynyl-3-methylbenzene (29.0 mg, 0.25 mmol, 116 g/mol) gave **6d** as a white powder (44 mg, 491 g/mol, 36 %), mp. 180 °C. ^{13}C NMR ($CDCl_3$, 125 MHz) δ (ppm): 138.2, 137.4, 132.1, 130.1, 128.7, 128.4, 122.2, 121.6, 114.9, 108.1, 107.1, 99.9, 80.7, 21.2.

*Synthesis of 3-(2-(2,3,6-tribromothieno[3,2-b]thiophen-5-yl)ethynyl)pyridine (**6e**)*
Following the general procedure, using 2,3,5,6-tetrabromthieno[3,2-*b*]thiophene (114 mg, 0.25 mmol, 456 g/mol), Ph_3P (13.1 mg; 0.2 eq., 262 g/mol), $Pd(OAc)_2$ (5.6 mg; 0.1 eq., 225 g/mol), CuI (10 mg; 0.2 eq., 190 g/mol) and 3-ethynylpyridine (25.75 mg, 0.25 mmol, 103 g/mol) gave **6e** as a white powder (35 mg, 478 g/mol, 30 %), mp. 210 °C. ^{13}C NMR ($CDCl_3$, 125 MHz) δ (ppm): 153.2, 149.4, 139.7, 130.3, 130.1, 122.1, 121.8, 123.7, 123.0, 116.4, 109.0, 78.2 ; MS (ESI): calcd. for $[M+H]^+$, $[C_{13}H_5Br_3NS_2]^+$ 479, found 479; calcd. for $[M-H]^-$, $[C_{13}H_3Br_3NS_2]^-$, 477, found 477.

*Synthesis of 3,6-dibromo-2-(2-p-tolylethynyl)thieno[3,2-b]thiophene (**7a**)*
Following the general procedure, using 2,3,5,6-tetrabromthieno[3,2-*b*]thiophene (114 mg, 0.25 mmol, 456 g/mol), Ph_3P (13.1 mg; 0.2 eq., 262 g/mol), $Pd(OAc)_2$ (5.6 mg; 0.1 eq., 225 g/mol), CuI (10 mg; 0.2 eq., 190 g/mol) and 1-ethynyl-4-methylbenzene (51.0 mg, 0.50 mmol, 116 g/mol) gave **7a** as a pale yellow powder (41 mg, 412 g/mol, 42 %), mp. 130 °C. ^{13}C NMR ($CDCl_3$, 125 MHz) δ (ppm): 137.5, 131.6, 129.2, 128.5, 122.1, 122.0, 115.0, 108.2, 107.1, 99.6, 81.0, 21.1; MS (ESI): calcd. for $[M+H]^+$, $[C_{15}H_9Br_2S_2]^+$: 413, found 413; calcd. for $[M-H]^-$, $[C_{15}H_7Br_2S_2]^-$ 411, found 411.

*Synthesis of 3,6-dibromo-2-(2-(4-methoxyphenyl)ethynyl)thieno[3,2-b]thiophene (**7b**)*
Following the general procedure, using 2,3,5,6-tetrabromthieno[3,2-*b*]thiophene (114 mg, 0.25 mmol, 456 g/mol), Ph_3P (13.1 mg; 0.2 eq., 262 g/mol), $Pd(OAc)_2$ (5.6 mg; 0.1 eq., 225 g/mol), CuI (10 mg; 0.2 eq., 190 g/mol) and 1-ethynyl-4-methoxybenzene (66.0 mg, 0.50 mmol, 132 g/mol)

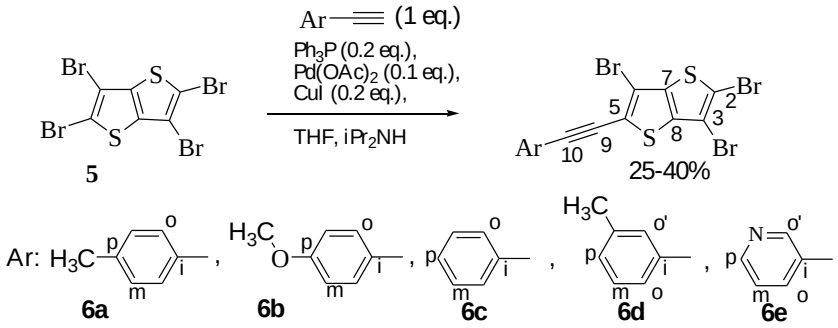
gave **7b** as a white powder (45 mg, 428 g/mol, 42 %), mp. 125 °C. ^{13}C NMR ($CDCl_3$, 125 MHz) δ (ppm): 139.2, 138.0, 131.2, 131.0, 129.5, 119.0, 114.9, 108.0, 107.2, 99.9, 80.4, 55.4.

3. RESULTS AND DISCUSSION

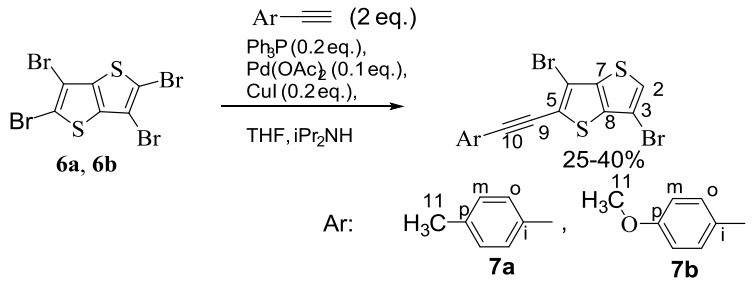
3.1. Synthesis

The 2,3,5,6-tetrabromthieno[3,2-*b*]thiophene (**5**) was synthesized following by Wynberg *et al.* and Turbiez *et al.* [16,17] with some modification such as ratio of thieno[3,2-b]thiophene-2-carboxylic acid and bromine. It was found that the best ratio of thieno[3,2-b]thiophene-2-carboxylic acid and bromine was 1:1.8 at ambient temperature 30 ºC for 1 hour; 110 ºC for 3.5 hours and 70 ºC for 12 hours forming 2,3,5,6-tetrabromthieno[3,2-*b*]thiophene in 60 % yield.

Monoalkylation via the Sonogashira reaction was carried out successfully, Scheme 1 and 2, but the yields were quite low since the diyne products were formed along with. Surprisingly, when the aryl alkynes were increased 2 equivalents gave more monoalkynyl products **7a** and **7b** with removal of a bromine atom. That observation could be explained that H-insertion happened. The bromine atoms at position 2 or 5 were selective for substitution since they were more reactive than the others [18]. As mentioned above, the formation of diyne products called Glaser products [19] decreased the yield of expected products. Therefore, amount of Cu(I) was changed from 0.05 eq.; 0.1 eq.; 0.2 eq. to 0.3 eq. It was found that amount Cu(I) iodide of 0.2 eq. was the best for **6a**. Then other reactions were carried under this condition.



Scheme 1: Monoalkylation via the Sonogashira reaction



Scheme 2: Attempt in synthesis of dialkynylation *via* the Sonogashira reaction

3.2. Structure determination

Mass spectral data of compounds **6e** and **7a** gave complicated molecular ion peaks because of the presence of 3 or 2 bromine atoms based on its isotope abundant. So it seemed that MS method was not a good choice for them. Anyway, MS of compound **6e** had a peak at *m/z* 479 indicating that molecular formula of **6e** must be $C_{13}H_4Br_3NS_2$ with molecular weight was 478 g/mol. Compound **7a** also gave molecular weight peak at 413 au that associated with molecular formula $C_{15}H_8Br_2S_2$.

Compounds **6a**, **6b**, **6c**, **6d** and **6e** were recorded ^{1}H NMR and ^{13}C NMR spectra. All compounds were resembled by alkynyl moieties and thieno[3,2-*b*]thiophen. However, the alkynyl moieties have protons but no protons for thieno[3,2-*b*]thiophen. Consequently, the ^{1}H NMR spectral analysis was quite simple as shown in the table 1. Compounds **6a**, **6b** and **6c** have two pairs of identical protons that have same chemical shift values. Since ortho protons were split by Hm/Hm' and Hp with J = 8.0-9.0 Hz and J = 1.5-2.0 Hz their shape ware as doublet or doublet doublet shape peaks. Then other aromatic protons were indicated for Hm/Hm'. Apart from these aromatic protons, peak at δ 2.38 ppm (s,

3H) was assigned for methyl group in compound **6a**; 3.86 ppm (s, 3H) for methyl group in the methoxy one in the compound **6b**. All aromatic protons of compounds **6d** and **6e** were not identical; thus, they appeared on the ^{1}H NMR as separately peaks. For example, in the ^{1}H NMR spectrum of compound **6d**, the peak at δ 7.40 ppm (s, 1H) was for Ho', but at δ 7.37 ppm (d, J = 8.0 Hz, 1H) must have been for Ho. Because of pyridine ring, Ho' of compound **6e** had appearance of a single peak at δ 8.8 ppm and Ho was a doublet peak at δ 8.6 ppm with J = 5.5 Hz. Both compounds **6d** and **6e** have only one proton Hm. The triplet peak at δ 7.26 ppm was assigned for Hm of compound **6d**, while the multiplet peak at δ 7.88 showed the appearance of Hm in the structure of compound **6e** since splitting with three different protons in the pyridine ring. Compounds **6c**, **6d** and **6e** have para protons that were very specific shape. Hp of compound **6c** was a multiplet peak at δ 7.37 ppm, doublet peak at δ 7.19 ppm with J = 7.5 Hz was for Hp of compound 6d and the doublet peak at δ 7.33 ppm with J = 5.5 Hz was fit with Hp of compound **6e**. Similar to compound **6a**, the peak at δ 2.38 ppm must have been for protons of methyl group on the benzene ring.

Table 1: ^{1}H NMR analysis of compound **6a-e** (δ (ppm))

	Ho/Ho'	Hm/Hm'	Hp	H11
6a	7.46 (d, J = 8.0 Hz, 2H)	7.18 (d, J = 8.0 Hz, 2H)	-	2.38 (s, 3H)
6b	7.53 (d, J = 9.0 Hz, 2H)	6.92 (d, J = 8 Hz, 2H)	-	3.86 (s, 3H)
6c	7.38 (dd, J = 8.0, 1.5 Hz, 2H)	7.58 (dd, J = 8.0, 7.5 Hz, 2H)	7.37 (m, 1H)	-
6d	7.40 (s, 1H) 7.37 (d, J = 8.0 Hz, 1H),	7.26 (t, J = 7.5 Hz, 1 H)	7.19 (d, J = 7.5 Hz, 1H)	2.37 (s, 3H)
6e	8.8 (s, 1H) 8.6 (d, J = 5.5 Hz, 1H)	7.86 (m, 1H)	7.33 (d, J = 5.5 Hz, 1H)	-

^{13}C NMR spectral analysis was listed in the experimental section. It is clearly that the framework of the expected structures. For example, all compounds showed 6 carbons of thieno[3,2-*b*]thiophene part. In addition, compounds **6a**, **6b** and **6c** ^{13}C NMR spectra showed 4 signals for 6 carbons in the benzene ring due to the para-position substituent themselves. In contrast, compounds **6d** and **6e** ^{13}C NMR spectra appeared 6 peaks according to unidentical 6 carbons. Each compounds **6a**, **6b** and **6d** gave an extra at strong field. Those were defined for methyl group attached directly to benzene rings or oxygen atom. Unfortunately, all ^{13}C NMR spectra of these compounds only showed one signal for C9 and C10. It was thought one might be in the peak blocks of $CDCl_3$.

In attempt synthesizing dialkynyl derivatives, the Sonogashira gave monoalkynation product **7a** and **7b**. These ^{1}H NMR analysis was shown in the table 2. The ^{1}H NMR spectrum of compound **7a** showed a new peak at δ 7.40 ppm and ^{1}H NMR spectrum of compound **7b** showed almost at the same position at δ 7.39 ppm. This observation indicated that the Sonogashira did proton reductive insertion instead of the second alkylation reaction. Moreover, both compound **7a** and **7b** contained alkynyl moiety including para-substituted groups. Therefore, protons of ortho or meta positions were doublet with coupling constant from 7.0 Hz to 8.0 Hz indicating the ortho positions each other in the benzene ring [20]. Signals at δ 2.38 ppm in the ^{1}H NMR spectrum of compound **7a** illustrated for the presence of the methyl group attached with benzene group. Similarly, the ^{1}H NMR spectrum of compound **7b** also had a peak at δ 3.84 ppm of methyl in the methoxy group.

Table 2: ^{1}H NMR spectral analysis of compounds **7a** and **7b**

	H2	Ho	Hm	H11
7a	7.40 (s, 1H)	7.47 (d, J = 8.0 Hz, 2H)	7.18 (d, J = 8.0 Hz, 2H)	2.38 (s, 3H)
7b	7.39 (s, 1H)	7.52 (d, J = 7.0 Hz, 2H)	6.90 (d, J = 7.0 Hz, 2H)	3.84 (s, 3H)

Compounds **7a** and **7b** were also recorded ^{13}C NMR spectra. The analysis was shown in the experiment section. Compound **7a** has 13 signals of carbons; nevertheless its ^{13}C NMR spectrum showed only 12

signals included 10 aromatic carbon atoms from δ 99.6 ppm to 137.5 ppm; a peak at δ 81.0 ppm was for C9 or C10 (C≡C), similarly, one was hidden in the solvent peaks. In addition, the signal at δ 21.0 ppm must be for methyl group. Instantly, the ^{13}C NMR spectrum of compound **7b** was quite similar to that of compound **7a** case except the signal at δ 55.4 ppm that belonged to the methyl group of methoxy one.

4. CONCLUSION

Seven new thieno[3,2-*b*]thiophen derivatives were synthesized successfully via the Sonogashira cross-coupling reactions in moderate yield. The equivalent of CuI was 0.2 that was good for producing monoalkynation products **6a**, **b**, **c**, **d**, **e**; to reduce diyne products but increasing the equivalence of alkynes monoalkynation companied by H- insertion to form compound **7a** and **7b**. The structures of these compounds were confirmed by ^{1}H and ^{13}C NMR and mass spectral analyzes.

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