Synthesis of Unsymmetric Monosubstituted and Disubstituted Dinaphthothiophenes

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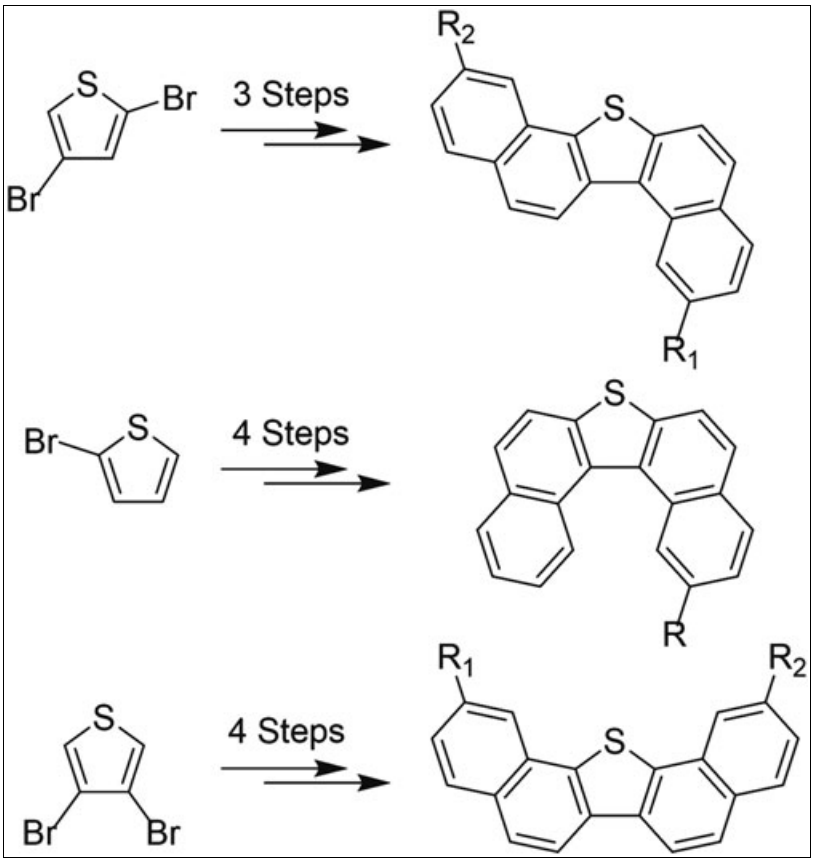

Dinaphthothiophenes (DNTs) are a class of compounds with potential uses in organic semiconductors and the synthesis of unsymmetric catalysts. Symmetrical or asymmetrical addition of functional groups to the DNT structure may be desired for steric bulk in binaphthyl catalyst synthesis or tuning the electronic properties of semiconductors. Thus, versatility of functional group addition is a great asset in DNT synthesis. Until now, no versatile and concise methods for the synthesis of unsymmetrically substituted DNTs have been reported. Herein, we report three synthetic routes for the creation of three different classes of DNTs. Each route involves the successive addition of two functionalized styryl groups to a thiophene ring, followed by a photocyclization to form the desired asymmetric DNT. Various novel unsymmetrically monosubstituted and disubstituted dinaphtho[2,1-*b*:1′,2′-*d*]thiophenes, dinaphtho[1,2-*b*:1′,2′-*d*]thiophenes, and dinaphtho[1,2-*b*:2′,1′-*d*]thiophenes were synthesized from 2-bromothiophene,2,4-dibromothiophene, and 3,4-dibromothiophene in three or four steps. These methods can be used to synthesize a wide variety of unsymmetrically functionalized DNTs.

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INTRODUCTION

Dinaphthothiophenes (DNTs) are a class of compounds structurally similar to thiophene-based organic semiconductors [1–3] and have shown promise for use in p-type organic semiconductors [4,5]. DNTs have also been used as precursors to axially chiral 1,1′-binaphthyl catalysts, which play a large role in asymmetric synthesis [6–8]. In addition, dibenzothiophene S-oxide (DBTO) and its derivatives are a class of compounds suggested to release $O(^3P)$ upon irradiation with UV light [9–12]. DNT S-oxides and other fused ring thiophene S-oxides

have been investigated for their potential to release atomic oxygen during irradiation at longer wavelengths [13]. Despite their potential applications, few efficient ways to prepare asymmetrically substituted DNTs have been reported.

Dinaphtho[2,1-*b*:1′,2′-*d*]thiophene "**DNT-2112**," dinaphtho[1,2-*b*:1′,2′-*d*]thiophene "**DNT-1212**," and dinaphtho[1,2-*b*:2′,1′-*d*]thiophene "**DNT-1221**" (Fig. 1) are three types of DNTs whose syntheses have previously been reported. **DNT-2112** has been synthesized from the Newman–Kwart rearrangement of dithiocarbamates by heating the dimethylthiocarbamate of binaphthol neat at 285–310°C to give the **DNT-2112** in 20–40% yield [8,14,15], from dinaphthyl sulfide using an iodine-catalyzed photocyclization in 85% yield [16] and from cyclization of alkynes by heating ethynyl sulfides in benzene at 200°C in a cascade cycloaromatization with 10% yield [17]. Rabindran and Tilak performed the condensation of 2-bromo-1-tetralone with 2-naphthalenethiol or 1-naphthalenethiol, followed by cyclization with P_2O_5 in phosphoric acid and dehydrogenation with selenium, giving **DNT-2112** and **DNT-1212** in 78% and 76% overall yield, respectively [18]. Morrison and Musgrave used the condensation of thiophene with 1,2-diphenylethanone to give (E,E)-2,5-bis(α-phenylstyryl)thiophene [19]. The phenyl-substituted distyrylthiophene was then photocyclized with iodine to give the diphenyl substituted **DNT-2112** in 10% yield. The drawbacks of these preparations of **DNT-2112** are that they give low overall yields and take two to four steps to prepare.

DNT-1221 derivatives have been made from the reaction of naphthalene-1-sulfonic acid dimethylamide with *n*-butyllithium and S_8 in 29–37% yield [20]. In addition, both **DNT-2112** and **DNT-1221** have been synthesized from dinaphthyl sulfides using a potassium *tert*-butoxide or *n*-butyllithium induced cyclodehydrogenation in 18–31% yield [21,22]. These syntheses of **DNT-1221** suffer from low overall yields. While the final cyclization reactions to synthesize all three varieties of DNTs usually require only one step, anywhere from one to six steps may be required to synthesize the precursors needed for the cyclization reaction from commercially available materials. In addition, the methods requiring fewer steps to reach the cyclization precursor tend to have a more limited scope of synthesis. For example, the method of Morrison and Musgrave, which is the sole method to require only one step to achieve the cyclization precursor, lacks the ability to generate unsubstituted DNTs and has only been used to make diphenyl substituted **DNT-2112** [19].

All three classes of DNTs have been synthesized from the flash vacuum pyrolysis of diethynyl/dichlorovinyl-diphenylthiophenes in 7–89% yield [23]. The flash pyrolysis method has some capability for functionalization of the DNT structure. However, it is only able to functionalize symmetrically, which limits the potential for the tuning of the electronic properties of DNTs by tuning functional groups. Furthermore, Tedjamulia *et al*. prepared all three classes of DNTs from formyl-benzonaphthothiophenes [24]. A Horner–Wadsworth–Emmons reaction was used to add a styrene unit, followed by an iodine-catalyzed photocyclization that gave yields of 45–76%. The synthetic route created here has potential for use in asymmetric DNT substitution, because the DNT core structure is assembled one-half at a time; yet controlling the final position of the functional group would be difficult because of the variability of products of the final photocyclization step. This method also has the disadvantage of requiring three to six steps to reach the photocyclization precursor.

While DNTs have previously been asymmetrically functionalized, this has always been carried out after the synthesis of the DNT structure (Fig. 2). Cho *et al*. have taken **DNT-2112** and functionalized the 6-position of **DNT-2112** by use of *tert*-butyllithium and iodine [8]. In addition, *n*-butyllithium and dimethylformamide (DMF) have been used to add an aldehyde group, also at the

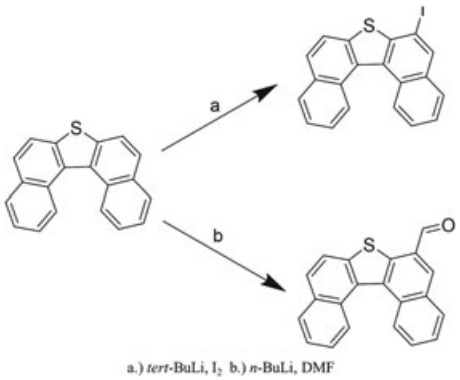


Figure 2. Dinaphthothiophene functionalization.

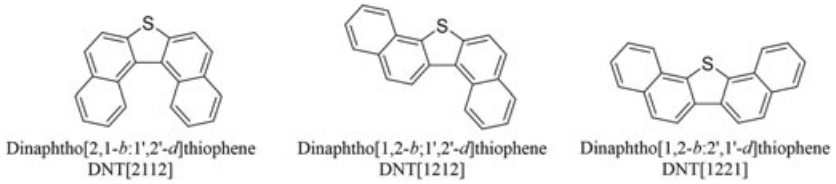
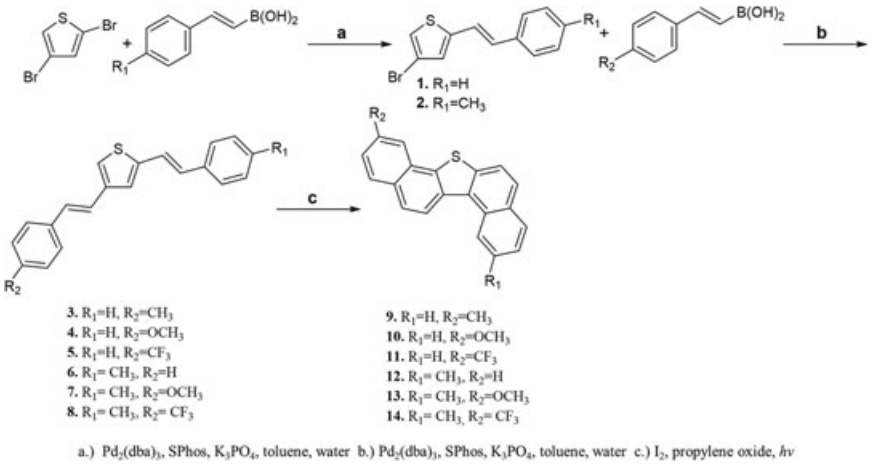


Figure 1. Example dinaphthothiophenes.

Scheme 1. Synthesis of dinaphtho[1,2-*b*:1′,2′-*d*]thiophenes.



a.) $Pd_2(dba)_3$, SPhos, K_3PO_4, toluene, water b.) $Pd_2(dba)_3$, SPhos, K_3PO_4, toluene, water c.) I_2, propylene oxide, *hv*

Table 1

Suzuki–Miyaura reactions.

Entry	Bromothiophene	Product	Equiv R—$B(OH)_2$	Temp. (°C)	Water (% of solvent)	Yield (%)
1	2,4-Dibromothiophene	**1**	1.1	95	11	81
2	2,4-Dibromothiophene	**2**	1.1	90	20	29
3	**1**	**3**	1.2	80	17	46
4	**1**	**4**	1.1	85	17	10
5	**1**	**5**	1.2	55	17	48
6	**2**	**6**	1.2	80	20	36
7	**2**	**7**	1.1	75	17	30
8	**2**	**8**	1.1	75	17	31
9	2-Bromothiophene	**15**	1.2	80	11	78

6-position [25,26]. This indicates that the six (and eight) positions of **DNT-2112** are selectively deprotonated by bases such as *n*-butyllithium, leaving the other positions on the DNT rings unable to be so similarly substituted.

In short, a variety of synthetic routes have previously been reported to produce unfunctionalized and a few functionalized DNTs. However, none of these methods begin with the thiophene ring and, therefore, require a greater number of steps to reach the DNT structure. Furthermore, these methods do not provide a simple way to unsymmetrically incorporate functional groups onto the DNT structure. Herein are shown straightforward novel synthetic routes by which to synthesize unsymmetrically substituted derivatives of **DNT-2112**, **DNT-1212**, and **DNT-1221** (Fig. 1), from monosubstituted and dibromine-substituted thiophenes. Each of these routes involves only three to four steps in total and contains the potential for the symmetric and unsymmetric introduction of a wide variety of functional groups.

RESULTS AND DISCUSSION

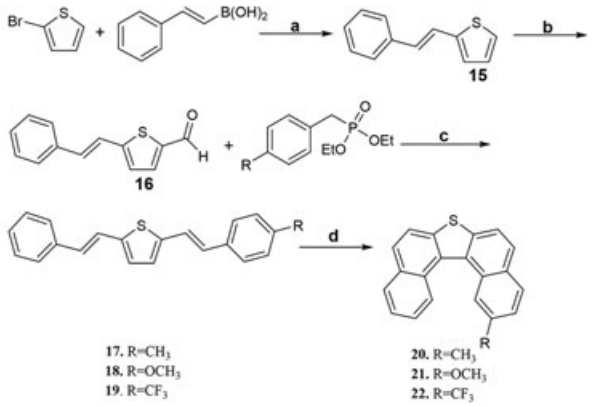
The derivatives of three different classes of thiophenes: dinaphtho[2,1-*b*:1′,2′-*d*]thiophene, dinaphtho[1,2-*b*:1′,2′-*d*]thiophene, and dinaphtho[1,2-*b*:2′,1′-*d*]thiophene were created by three different routes. Each route followed the same general strategy, beginning with two styrene groups being sequentially added to a monosubstituted or dibrominated thiophene by either Suzuki coupling or Horner–Wadsworth–Emmons reaction. Once the thiophene was doubly substituted with styrene units, a photocyclization reaction with iodine as an oxidant was used to create the final DNT structure. In contrast to other synthetic routes, unsymmetrically substituted functional groups have been incorporated into the synthesis of the DNT structure itself. Methoxy, trifluoromethyl, and methyl functional groups were used to create substituted

Table 2

2,4-Distyrylthiophene cyclization.

Entry	Distyrylthiophene	Product	Time (days)	Yield (%)
1	**3**	**9**	0.8	24
2	**4**	**10**	0.6	66
3	**5**	**11**	1.9	24
4	**6**	**12**	0.8	31
5	**7**	**13**	0.8	20
6	**8**	**14**	0.7	28

Scheme 2. Synthesis of dinaphtho[2,1-*b*:1′,2′-*d*]thiophenes.



a.) $Pd_2(dba)_3$, SPhos, K_3PO_4, toluene, water b.) n-BuLi, DMF, THF c.) NaH, THF d.) I_2, propylene oxide, *hv*

DNTs. These three groups were chosen for their differences in electronegativity, which could be used to tune the electronic properties of DNTs.

DNT-1212 derivatives were synthesized using the path shown in Scheme 1. This synthetic path used for the synthesis of unsymmetrically substituted **DNT-1212** derivatives began with 2,4-dibromothiophene. The bromine in the 2-position is preferentially substituted over the 4-position in carbon–carbon coupling reactions to give an unsymmetric product [27–32]. Therefore, a Suzuki–Miyaura reaction with one equivalent of [(E)-2-phenylethenyl]boronic acid or [(E)-2-[4-(methyl)phenyl] ethenyl]boronic acid was performed to add the first styrene unit to give 4-bromo-2-styrylthiophenes **1** and **2** (Table 1). A second Suzuki–Miyaura reaction was used to add a second styryl group in the 4-position. This second coupling was successful with 4-substituted styrylboronic acids to give 2,4-distyrylthiophenes **3**–**8** (Table 1). These Suzuki–Miyaura couplings gave yields anywhere from 10 to 81% depending on the substituent on the boronic acid. Reactions were performed at temperatures ranging from 55°C to 95°C; however, no significant change in yield was noticed. Unsubstituted styrylboronic acids gave the highest yields, followed by trifluoromethyl-substituted styrylboronic acids. Methoxy-substituted boronic acids gave the lowest yields. In the next step of this synthetic route, 2,4-distyrylthiophenes **3**–**8** were irradiated with short-wave ultraviolet (UVC) light in the presence of iodine and propylene oxide to fuse the rings, giving DNTs **9**–**14** (Table 2). This photoreaction proceeds by an oxidative mechanism: first, a photoinduced electrocyclization to create the C─C bond, followed by an oxidative dehydrogenation to regain aromaticity [33]. The photocyclization of 2,4-distyrylthiophenes gave yields of 20–31%, with the exception of **4**. The photocyclization resulting in compound **4** gave a higher yield of 66%, in contrast to most other reactions performed involving methoxy-substituted reactants, which had significantly lower yields on average than those involving different substituents.

Table 3

Horner–Wadsworth–Emmons reaction with 2-formyl-5-styrylthiophene.

Entry	Aldehyde	Product	Equiv phosphonic ester	Equiv NaH	Yield (%)
1	**16**	**17**	1.3	5.5	58
2	**16**	**18**	1.9	3.3	14
3	**16**	**19**	1.2	4.7	76

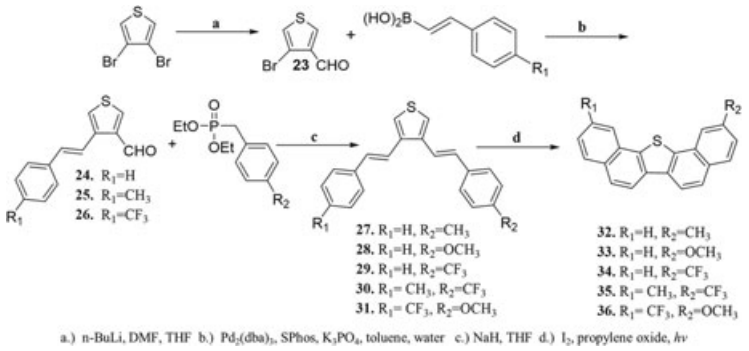
Table 4

2,5-Distyrylthiophene cyclization.

Entry	Distyrylthiophene	Product	Time (days)	Yield (%)
1	**17**	**20**	6	1
2	**18**	**21**	27	<1%
3	**19**	**22**	1.5	29

The 4-substituted *trans*-2-(phenylethenyl)boronic acids used in the Suzuki–Miyaura coupling step often underwent self-coupling rather than coupling with the bromothiophene. This created a side product, which was detected by gas chromatography/mass spectrometry (GCMS), with an *m*/*z* dependent on the boronic acid used. For example, Suzuki reactions involving unsubstituted styrylboronic acids in Table 1 (entries 1, 3, and 6), gave a product with a *m*/*z* of 206 and was believed to be 1,4-diphenyl-1,3-butadiene. The purification of the desired products was complicated by the presence of these self-coupled byproducts, especially in the case of **7**. Propylene oxide was added to all photocyclizations to quench the hydrogen iodide resulting from the reaction. In every photoreaction, care was taken not to irradiate the solution past completion, which would result in both a decreased yield and a white precipitate

Scheme 3. Synthesis of dinaphtho[2,1-*b*:2′,1′-*d*]thiophenes.



that was insoluble in organic solvents. In the absence of iodine, most photoreactions still occurred; however, they proceeded more slowly. In the synthesis of both **DNT-1221** derivatives and **DNT-2112** derivatives, reactions involving a methoxy substituent gave lower yields than those involving other substituents.

DNT-2112 derivatives **20**–**22** were created using the synthetic route shown in Scheme 2. First, 2-bromothiophene was coupled with a trans-2-(4-Phenyl) vinylboronic acid using a Suzuki–Miyaura coupling to create compound **15** in 76% yield [34]. The 5-position of the thiophene ring was then formylated using *n*-butyllithium and DMF, giving compound **16** in 36% yield [35,36]. The 5-position is preferentially formylated because of its relatively low pK_a (~33) compared with the three or four positions (~39) resulting from its location next to the sulfur in the thiophene ring [37]. A Horner–Wadsworth–Emmons reaction using a 4-substituted phosphonic acid diethyl ester was used to add a second styryl group to the other side of the thiophene ring (Table 3) to create 2,5-distyrylthiophenes**17–19.** The methoxy-substituted phosphonic acid diethyl ester gave a 14% yield that was significantly lower than the methyl and trifluoromethyl-substituted phosphonic esters, which gave yields of 58 and 76%, respectively. The DNTs **20**–**22** were created via the same oxidative photocyclization used in the synthesis of DNTs **9**–**14** (Table 4).

The Horner–Wadsworth–Emmons reaction of **16** to yield 2,5-distyrylthiophenes**17–19** gave unreliable yields. Different variables, such as molar equivalents of sodium hydride and temperature during reagent addition, were changed with no consistent improvement in yield. The Horner–Wadsworth–Emmons reaction to give **18** (14% yield) and the subsequent photocyclization to give **21** (<1% yield) gave significantly lower yields than the reactions to produce **17** and **19**. The photocyclization of **18** did not yield enough **21** to completely characterize, although it was detected by GCMS. This follows the trend seen in Table 1 (entries 4 and 7) and Table 2 (entry 5) where the Suzuki coupling and photocyclization of reactants containing the methoxy group gave lower yields. In addition, 2,5-distyrylthiophenes gave lower photocyclization yields than other distyrylthiophenes. Because **DNT-2112** is known to adopt a twisted conformation, these lower yields are likely due to sterics hindering the cyclization of 2,5-distyrylthiophene into the planar shape of other fused thiophene structures [13].

DNT-1221 derivatives **32**–**36** were synthesized using the route shown in Scheme 3. Formylation with *n*-butyllithium and DMF was used to convert 3,4-

Table 5

Suzuki–Miyaura reaction with 3-bromo-4-formylthiophene.

Entry	Bromothiophene	Product	Equiv R─B $(OH)_2$	Temp. (°C)	Yield (%)
1	**23**	**24**	1.2	95	77
2	**23**	**25**	1.5	70	46
3	**23**	**26**	1.2	86	86

Table 6

Horner–Wadsworth–Emmons reaction with 3-formyl-4-styrylthiophene.

Entry	Aldehyde	Product	Equiv phosphonic ester	Yield (%)
1	**24**	**27**	1.5	16
2	**24**	**28**	1.2	21
3	**24**	**29**	1.5	95
4	**25**	**30**	1.5	32
5	**26**	**31**	1.0	40

Table 7

3,4-Distyrylthiophene cyclization.

Entry	Distyrylthiophene	Product	Time (h)	Yield (%)
1	**27**	**32**	8	7
2	**28**	**33**	6.5	20
3	**29**	**34**	7.5	18
4	**30**	**35**	5	9
5	**31**	**36**	3.5	6

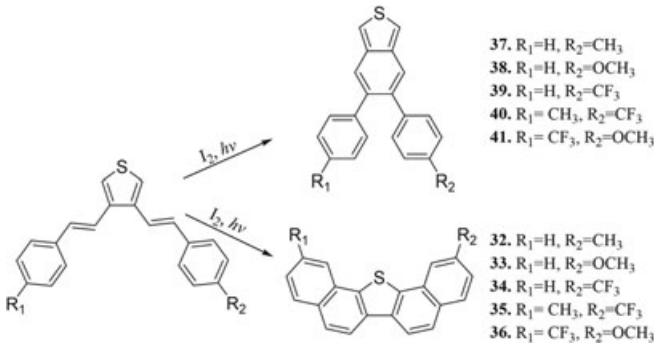


Figure 3. Products of 3,4-distyrylthiophene photoreaction.

dibromothiophene to 3-bromothiophene-4-carbaldehyde**23** in 77% yield [38,39]. Suzuki–Miyaura coupling was then used to add the first styryl group to one side of the thiophene ring to give 3-formyl-4-styrylthiophenes**24**–**26** in 10–77% yield (Table 5), from which unsymmetric distyrylthiophenes could easily be synthesized. The trifluoromethyl-substituted styrylboronic acid gave the highest yields. A Horner–Wadsworth–Emmons reaction was used to add the second substituted styryl group to the other side of the thiophene ring (Table 6), creating 3,4-distyrylthiophenes**27–31** in yields from 16% to 95%. The CF_3-substituted benzylphosphonic esters used in the creation of **29** and **30** gave higher yields compared with those with other substituents. The Suzuki–Miyaura coupling was performed before the Horner–Wadsworth–Emmons reaction in this route because the 1,4-diphenyl-1,3-butadiene byproducts formed from the Suzuki–Miyaura reaction were easier to separate from the more polar 3-formyl-4-styrylthiophenes than from 3,4-distyrylthiophenes. An oxidative photocyclization was then used in the same manner as the previous routes to fuse the rings together to give **DNT-1221** derivatives **32**–**36** in yields of 6–20% (Table 7).

A side product of the photoreaction of **27**–**31** is suspected to form by the ring closure of the thiophene ring as shown in Figure 3, forming side products **37**–**41**. The side product for Table 7, entry 1 (**37**), was analyzed by GCMS and shown to have a *m*/*z* of 300, corresponding to the loss of H_2 by the oxidative dehydrocyclization mechanism. Solvents such as toluene, hexanes, and a mixture of dichloromethane and hexanes were tried in the photocyclization reaction, but there was no significant change in the product ratios. Preparative thin-layer chromatography was used to separate the photocyclization reaction products.

CONCLUSION

In summary, short synthetic routes have been created for making unsymmetrically substituted dinaphtho[1,2-*b*:1′,2′-*d*]thiophenes, dinaphtho[2,1-*b*:1′,2′-*d*]thiophenes, and dinaphtho[1,2-*b*:2′,1′-*d*]thiophenes from 2,4-dibromothiophene,2-bromothiophene, and 3,4-dibromothiophene, respectively. Distyrylthiophenes were created unsymmetrically by adding one styrene unit to the thiophene ring at a time. The first styrene was added by Suzuki–Miyaura coupling, while the second styrene as added by either Suzuki–Miyaura or Horner–Wadsworth–Emmons reactions, depending on the desired DNT. The 2,4-distyrylthiophenes,2,5-distyrylthiophenes, and 3,4-distyrylthiophenes were fused to form their respective DNTs using an oxidative photocyclization. Using these methods, a variety of asymmetric monosubstituted and disubstituted DNTs can be synthesized in three to four straightforward steps. Whereas only symmetrically substituted DNTs had been synthesized before, now a wide variety of unsymmetrically substituted DNTs can be easily made and tuned for use in organic semiconductors and chiral 1,1′-binaphthyl catalysts.

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SUPPORTING INFORMATION

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