Lewis Acid-Catalyzed Propargylic Etherification and Sulfanylation from Alcohols in $MeNO_2$–H_2O

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Direct scandium- and lanthanum-catalyzed etherifications of propargyl alcohols 1 and 6 in $MeNO_2$–H_2O provided propargyl ethers 3, 4 and 7 in high yields. In addition, reactions of 1 and 6 with thiols exclusively yielded the corresponding propargyl sulfides.

Key words C–O bond formation; C–S bond formation; propargyl alcohol

Ethers are one of the most fundamental structures of natural products[1—3] and carbohydrates[4]; thus, the development of general, straightforward synthetic methods for their preparation has received much attention. Methods whereby only H_2O is produced as a byproduct, such as dehydrative etherification, are desirable yet challenging synthetic process. Dehydrative etherifications generally employ two alternative mechanistic pathways. In one case, alcohol is used as a precursor for carbocations. Alternatively, the alcohol can function as a nucleophile (Chart 1). However, substitution of hydroxyl group in an alcohol generally requires the prior activation of the hydroxyl group because of the poor leaving ability of this group.[5,6] An alcohol used as a precursor for carbocations is usually activated by transforming it to the corresponding acetate,[7—17] carbonate,[18,19] halide,[20] phosphonate.[21,22] Prior activation of an alcohol is also necessary if it has to be used as a nucleophile because of its low nucleophilicity. This is usually achieved by converting the hydroxyl group to a metal alkoxide.[23—26] Various other useful methods employing transition metals have been reported[27—38]; however, these methods have limited applicability. To achieve one-pot etherification, the following four processes need to be performed: i) consider the alcohol as an electrophile and activate the hydroxyl group, ii) generate a carbocation, iii) consider the alcohol as a nucleophile and form an ether, and iv) control further reactions that lead to fission of the newly formed C–O bond.

In this context, we previously devised a new method for C–O bond formation with propargyl alcohols. This method involves Lewis acid-catalyzed C–C bond formation and thiazole synthesis *via* a propargyl cation stabilized by a unique structure.[39] A more versatile propargylation using the alcohol directly as a carbocation precursor would be a powerful tool leading to some useful compounds. In the present study, we report a unique Lewis acid-catalyzed etherification using both γ-sulfur substituted and non-substituted propargyl alcohols in $MeNO_2$–H_2O.

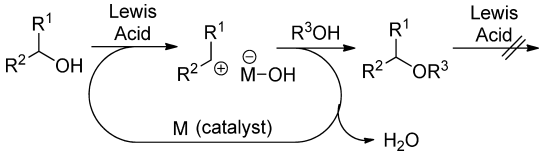


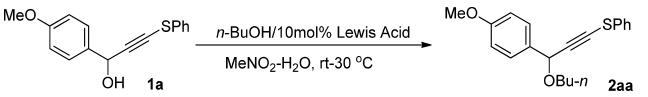
Chart 1. Lewis Acid-Catalyzed Etherification of Alcohols

Results and Discussion

First, we examined the reaction of **1a** with *n*-butanol in $MeNO_2$ under the previously reported propargylation conditions, which were described only as the synthetic procedure for the 4-oxahepta-1,6-diynes.[40—43] Scandium-catalyzed etherifications in nitromethane resulted in a complex mixture; however, the addition of H_2O drastically improved the reaction and provided ether **3a** in 84% yield (entries 1 and 2). Results of screening of other etherification conditions using **1a** are shown in Table 1. Various metals, mainly lanthanoids, were found to be effective for etherifications in $MeNO_2$–H_2O. Next, we selected propargyl alcohols bearing other aromatic substituents as starting materials and performed etherifications under the suitable condition using $Sc(OTf)_3$/$MeNO_2$–H_2O at 30 °C. With the $Sn(OTf)_2$ system, the product **2aa** was also obtained in the satisfactory yield, however, a small amount of unknown compounds were contaminated. Results of these comparisons are shown in Table 1.

Using the same substrate, 3-*p*-methoxyphenylpropargyl alcohol **1a**, etherification with 1° alcohols including methanol, dodecanol and geraniol as nucleophiles produced products **2ab**, **2ac** and **2af**, respectively, in good yields (entries 1, 2

Table 1. Discovering Reaction Conditions for Lewis Acid-Catalyzed C–O Bond Formation of 3-Sulfanyl Propargyl Alcohol **1a**



Entry	Lewis acid	Condition	Product[a] (% yield)
1	$Sc(OTf)_3$	$MeNO_2$, 30 °C, 10 min	0
2		$MeNO_2$–H_2O, 30 °C, 20 min	84
3		$MeNO_2$–H_2O, 15 °C, 1 h	81
4	BF_3–Et_2O	$MeNO_2$, 30 °C, 10 min	0
5		$MeNO_2$–H_2O, 30 °C, 18 h	73
6	$SnCl_4$	$MeNO_2$, 30 °C, 10 min	56
7		$MeNO_2$–H_2O, 30 °C, 7 h	82
8	$TiCl_4$	$MeNO_2$, 30 °C, 20 min	68
9		$MeNO_2$–H_2O, 30 °C, 6 h	78
10	$Yb(OTf)_3$	$MeNO_2$–H_2O, 30 °C, 30 min	83
11	$La(OTf)_3$	$MeNO_2$–H_2O, 30 °C, 15 min	85
12	$Hf(OTf)_4$	$MeNO_2$–H_2O, 30 °C, 50 min	64
13	TMSOTf	$MeNO_2$, 30 °C, 1 h	0
14	$Sn(OTf)_2$	$MeNO_2$–H_2O, 30 °C, 1 h	85

a) The reaction was carried out with alcohol (10 eq) in the presence of 10 mol% of Lewis acid.

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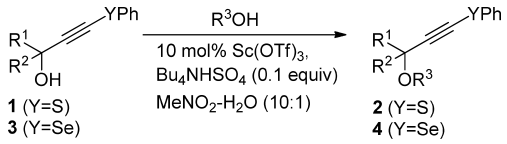
Table 2. Propargylic Etherifications of **1** and **3** with Some Alcohols



Entry	Y	Alcohol **1** R^1	R^2	R^3	Reaction time	Product (% yield)[a]
1	**1a** (S)	*p*-$MeOC_6H_4$	H	Me	20 min	**2ab** (83)
2				$Me(CH_2)_{11}$	20 min	**2ac** (59)
3				Cyclopentyl	20 min	**2ad** (70)
4				Cyclohexyl	20 min	**2ae** (75)
5				Geranyl	3 h	**2af** (71)
6	**1b** (S)	2-Thienyl	H	Me	1 h	**2ba** (82)
7				*n*-Bu	1 h	**2bb** (75)
8				Cinnamyl	1 h	**2bc** (79)
9				Cyclopentyl	1 h	**2bd** (82)
10				Cyclohexyl	1.5 h	**2be** (74)
11	**1c** (S)	1-Naphthyl	H	*n*-Bu	4 h	**2ca** (80)
12	**1d** (S)	2,4,6-$Me_3C_6H_2$	H	*n*-Bu	1.5 h	**2da** (91)
13	**1e** (S)	*p*-FC_6H_4	H	*n*-Bu	9 h	**2ea** (78)
14			H	Me	10 h	**2eb** (73)
15	**1f** (S)		H	Allyl	20 min	**2fa** (78)
16			H	Me	10 min	**2fb** (84)
17				Cyclopentyl	1 h	**2fc** (72)
18	**1g** (S)	*n*-Pent	*n*-Pent	Bn	3.5 h	**2ga** (44)
19	**1h** (S)	Ph	Ph	Bu	3.5 h	**2ha** (41)
20	**1i** (S)	$(CH_2)_5$		$Me(CH_2)_{11}$	10 min	**2ia** (88)[b]
21	**1j** (S)	$(CH_2)_4$		$Me(CH_2)_{11}$	10 min	**2ja** (58)[b]
22	**1k** (S)	$(CH_2)_6$		$Me(CH_2)_{11}$	10 min	**2ka** (79)[b]
23	**3a** (Se)	*p*-$MeOC_6H_4$	H	Me	10 min	**4aa** (80)
24	**3b** (Se)		H	Bn	10 min	**4ba** (99)
25	**3c** (Se)	1-Naphthyl	H	Bn	3 h	**4ca** (99)
26	**3d** (Se)	$(CH_2)_5$		Bn	1.5 h	**4da** (99)

a) The reaction was carried out with alcohol (3—10 eq) in $MeNO_2$–H_2O (10 : 1) at 30 °C. *b*) The reactions of cycloalkanols gave a small amount of phenylsulfanylethynylcycloalkenes **5**.[46]

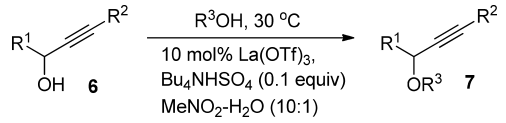
and 5). In contrast, 2° alcohols such as cyclopentanol and cyclohexanol afforded **2ad** and **2ae** (entries 3 and 4). Surprisingly, both 1° and 2° alcohols are also nucleophilic toward propargyl cations in $MeNO_2$–H_2O. Alcohols bearing other aromatic substituents at the R^1-position were examined with the 1° and 2° alcohols (entries 6—17). Alcohol **1d**, bearing the bulky aromatic substituent, 2,4,6-trimethylphenyl, also produced propargylic ether **2da** in excellent yield (entry 12). Di(*n*-pentyl)alcohol **1g** and diphenyl derivative **1h** produced ether **2ga** and **2ha** in moderate yields (entries 18 and 19, respectively). 1,3-Benzodioxol-5-yl derivative **1f** provided **2fa**—**c** (entries 15—17). Interestingly, phenylsulfanylethynylcycloalkanols, which were expected to undergo exclusive β-elimination, gave ethers **2ia**—**2ka** in good yields (entries 20—22).[44] Using similar conditions, the selenium analogs underwent etherification to produce **4aa**—**da** (entries 23—26). 1-Aryl propargyl alcohols (R^1=Ar, R^2=H) underwent etherification to yield a wide variety of ethers **2a**—**f**; however, reactions of the dialkyl derivatives were found to be sensitive. Cycloalkanols (R^1–R^2=$(CH_2)_n$) provided ethers in high yields, while the dipentyl alcohol gave low yields of products. On the other hand, most 1° and 2° alcohols, but not 3° alcohols, were also found to be effective as electrophiles.

These successful results suggested the possibility that etherifications of non-sulfur-substituted propargyl alcohols with both 1° and 2° alcohols in $MeNO_2$–H_2O might be productive (Table 3). When alcohol **6a** bearing an electron-donating substituent (Ar=*p*-$MeOC_6H_4$) was used, it took 12—24 h to complete the reaction because of the absence of the sulphur-accelerating component in the propargyl alcohols. Therefore, we further optimized the reaction conditions for non-sulphur-substituted propargyl alcohols and found that the use of lanthanum triflate in $MeNO_2$–H_2O at room temperature was suitable. Using the optimized conditions, we performed reactions with *n*-dodecanol and allyl alcohol, as 1° alcohols, and isopropyl alcohol, as 2° alcohol (entries 2—4). Etherifications of the propargyl alcohols **6b** (R^1=Ph, R^2=H), **6c** (R^1=R^2=Ph), and **6d** (R^1=Ph, R^2=*n*-Bu) proceeded in high yields. Thus, we have found that lanthanum triflate-catalyzed propargylic etherifications in $MeNO_2$–H_2O represent a powerful tool for C–O bond formations.

Sulphur-substituted dipropargyl ethers were previously reported to be good precursors for methoxide- and ethoxide-mediated cyclizations giving the furan derivatives.[45] Therefore, we investigated the sulfanylation of propargyl alcohol **1a** with non-activated thiols, and the results of these investigations are shown in Table 4.

Compared to alcohols, reactions with thiols were completed more rapidly. The reaction of **1a** with decanethiol gave propargyl decyl sulphide **8aa** in 91% yield. We also per-

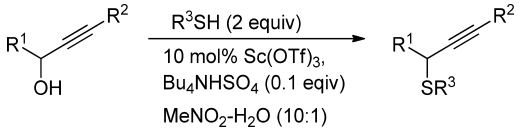
Table 3. Propargylic Etherifications of **6** with Some Alcohols



Entry		Alcohol **6** R^1	R^2	R^3 (eq)	Reaction time	Product **7** (% yield)[a]
1	**6a**	*p*-$MeOC_6H_4$	H	Bn (3)	10 min	**7aa** (63)
2				$Me(CH_2)_{11}$ (3)	18 h	**7ab** (79)
3				Allyl (10)	10 min	**7ac** (92)
4				*i*-Pr (10)	20 min	**7ad** (64)
5	**6b**	Ph	H	Bn (5)	2 h	**7ba** (81)
6				Propargyl (2)	48 h	**7bb** (75)
7				Me (5)	24 h	**7bc** (82)
8	**6c**	Ph	Ph	Allyl (10)	72 h	**7ca** (96)
9				Propargyl (7)	15 h	**7cb** (79)
10				Cyclopentyl (3)	15 h	**7cc** (73)
11				Cyclohexyl (3)	21 h	**7cd** (46)
12	**6d**	Ph	*n*-Bu	Ph-propargyl (3)	19 h	**7da** (92)
13				Bn (3)	5 h	**7db** (88)

a) The reaction was carried out with alcohol (5—10 eq) in $MeNO_2$–H_2O (10 : 1) at 30 °C.

Table 4. C–S Bond Formation of Propargyl Alcohols with Some Thiols



Entry		Alcohol **1** R^1	R^2	R^3 (eq)	Reaction time	Product (% yield)[a]
1	**1a**	*p*-$MeOC_6H_4$	SPh	$Me(CH_2)_9$ (1.3)	1 h	**8aa** (91)
2				2-Naphthyl (2)	15 min	**8ab** (85)
3				2-Thienyl (2)	15 min	**8ac** (61)
4				*p*-ClC_6H_4 (2)	20 min	**8ad** (99)
5				Cyclohexyl (2)	30 min	**8ae** (94)
6	**1b**	2-Thienyl	SPh	$Me(CH_2)_9$ (2)	15 min	**8ba** (95)
7				*p*-ClC_6H_4 (2)	15 min	**8bb** (67)
8	**1c**	1-Naphthyl	SPh	2-Naphthyl (1.3)	15 min	**8ca** (65)
9	**1g**	Benzodioxol-5-yl	SPh	2-Naphthyl (2)	20 min	**8ga** (64)
10				$Me(CH_2)_9$ (2)	15 min	**8gb** (82)
11				*p*-ClC_6H_4 (2)	30 min	**8gc** (89)
12	**6b**	Ph	H	2-Naphthyl (2)	12 h	**9ba** (—)
13	**6c**	Ph	Ph	$Me(CH_2)_9$ (3)	30 min	**9ca** (82)
14				2-Naphthyl (1.5)	20 min	**9cb** (72)
15				*p*-ClC_6H_4 (1.5)	12 h	**9cc** (48)
16	**6d**	Ph	*n*-Bu	$Me(CH_2)_9$ (2)	1 h	**9da** (52)

a) The reaction was carried out in $MeNO_2$–H_2O (10 : 1) at 30 °C.

formed the reaction with other thiols such as 2-thienyl, naphthyl, *p*-chlorophenyl and cyclohexylthiol and obtained satisfactory results (entries 2—5, Table 4). A variety of propargyl alcohols bearing the substituents such as R^1=2-thienyl, 1-naphthyl and benzodioxol-5-yl produced some propargyl sulphides **8ba**—**8gc** in moderate to high yields (entries 6—11). Surprisingly, Lewis acid-catalyzed C–S bond formation with thiols generally proceeded without further addition of thiols to the alkynyl group, and the desired propargyl sulphides were obtained in good to high yields (entries 1—11). We expected that this is due to the strong stabilizing effect of the sulfanyl group on acetylene. On the other hand, propargyl alcohols bearing no sulphur functional group at the terminal acetylene afforded some of the desired sulphides in reasonable yields (entries 13—16). However, the reaction of 1-phenylpropargyl alcohol resulted in complete recovery of the starting alcohol (entry 12). Therefore, this protocol provided a wide variety of sulphides directly from propargyl alcohols.

In summary, we developed a powerful and novel protocol for dehydrative etherifications from propargyl alcohols using a variety of simple alcohols as nucleophiles and a combination of commercially available Lewis acids–Bu_4NHSO_4 in $MeNO_2$–H_2O. Since the removable organosulfanyl functional groups can be easily converted to other functional groups, these propargyl ethers and sulfides will become the leading compounds by some transformations. We also described the sulfanylation of propargyl alcohols with thiols. Further work, aims at development of useful organic synthesis using sulfur-

substituted propargyl ethers and sulfides, and the synthesis of new catalysts used in conjunction with $MeNO_2$–H_2O, is in progress. The results will be reported elsewhere.

Experimental

Melting points were determined by a Yanagimoto micro-melting point apparatus and uncorrected. Elemental analyses were determined by using Micro Corder (MT-6) of J Science Lab. at the Life Science Research Center, Gifu University. ^{1}H- and ^{13}C-NMR spectra were determined on JEOL ECA-600 (600 MHz), ECA-500 (500 MHz) and ECA-400 (400 MHz) spectrometer. IR spectra were recorded with a JASCO Fourier Transform-Infrared (FT-IR) 460PLUS infrared spectrometer and are expressed in reciprocal centimeters. Electron ionization (EI) mass spectra (MS) were obtained using a JEOL MS-700 spectrometer with a direct-insertion probe at 70 eV. All high-resolution mass spectra were obtained using a JMSD300 JMA2000 online system. 1-(Benzyloxy-2-propyn-1-yl)benzene (**7e**) (Registry No: 129758-80-1). 1-(Methoxy-2-propyn-1-yl)benzene (**7g**) (Registry No: 50874-13-0).

Typical Procedure for the Synthesis of 1-Methoxy-4-[1-butoxy-3-(phenylthio)-2-propyn-1-yl]benzene (3a), Typical Procedure Scandium triflate (55 mg, 0.11 mmol) was added to a mixture of **1a** (0.60 g, 2.22 mmol), 1-butanol (0.82 g, 11.0 mmol), tetrabutylammonium hydrogensulfate (75.3 mg, 0.22 mmol) in $MeNO_2$ (6.0 ml)–H_2O (0.6 ml) at 30 °C. The reaction mixture was stirred for 10 min and poured into a saturated $NaHCO_3$ (50 ml). The organic layer was separated and the aqueous layer was extracted with ether. The combined organic layer was dried over $MgSO_4$. The solvent was removed under reduced pressure. The residue was purified by the preparative TLC on silica gel eluting with AcOEt–*n*-hexane (1 : 50) to give the title compound **2aa** (0.52 g, 71%) as a yellow oil.

1-Methoxy-4-[1-butoxy-3-(phenylthio)-2-propyn-1-yl]benzene (2aa) A yellow oil, IR (KBr, cm^{-1}) *ν*: 2957, 2932, 2869, 2178, 1710, 1610, 1584, 1511, 1478, 1442, 1304, 1250, 1173, 1084, 1034, 831, 740, 688, 585, 519; ^{1}H-NMR (600 MHz, $CDCl_3$) *δ*: 0.91 (3H, t, *J*=7 Hz, Me), 1.36—1.43 (2H, m, CH_2), 1.59—1.64 (2H, m, CH_2), 3.49—3.53 (1H, m, OCH_2), 3.67—3.70 (1H, m, OCH_2), 3.80 (3H, s, OMe), 5.32 (1H, s, CH), 6.90 (2H, br d, *J*=9 Hz, ArH), 7.20—7.22 (1H, m, ArH), 7.30—7.33 (2H, m, ArH), 7.39—7.41 (2H, m, ArH), 7.45 (2H, brd, *J*=9 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) *δ*: 13.8 (q), 19.3 (t), 31.6 (t), 55.3 (q), 68.1 (t), 72.0 (d), 73.7 (s), 97.4 (s), 113.8 (d×2), 126.2 (d×2), 126.5 (d), 128.7 (d×2), 129.2 (d×2), 130.8 (s), 132.6 (s), 159.6 (s); MS *m/z*: 253 (M^+−Bu). *Anal.* Calcd for $C_{20}H_{22}O_2S$: C, 74.04; H, 6.46. Found: C, 73.95; H, 6.47.

1-Methoxy-4-[1-methoxy-3-(phenylthio)-2-propyn-1-yl]benzene (2ab) A yellow oil, IR (KBr, cm^{-1}) *ν*: 3428, 2929, 2360, 1610, 1510, 1478, 1442, 1304, 1250, 1173, 1079, 1033, 831, 740, 688; ^{1}H-NMR (600 MHz, $CDCl_3$) *δ*: 3.44 (3H, s, OMe), 3.81 (3H, s, OMe), 5.25 (1H, s, CH), 6.91 (2H, d, *J*=8 Hz, ArH), 7.20—7.24 (1H, m, ArH), 7.32 (2H, br t, *J*=8 Hz, ArH), 7.41 (2H, d, *J*=8 Hz, ArH), 7.44 (2H, d, *J*=8 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) *δ*: 55.3 (q), 55.7 (q), 73.5 (d), 74.4 (s), 96.7 (s), 113.9 (d×2), 126.3 (d×2), 126.6 (d), 128.8 (d×2), 129.2 (d×2), 130.3 (s), 132.5 (s), 159.8 (s); MS *m/z*: 269 (M^+−Me), 253 (M^+−MeO). *Anal.* Calcd for $C_{17}H_{16}O_2S$: C, 71.80; H, 5.67. Found: C, 71.95; H, 5.58.

1-Methoxy-4-[1-(dodecyloxy)-3-(phenylsulfanyl)-2-propyn-1-yl]benzene (2ac) A yellow oil, IR (KBr, cm^{-1}) *ν*: 2925, 2853, 2179, 1713, 1609, 1584, 1510, 1478, 1465, 1442, 1304, 1250, 1173, 1083, 1035, 832, 739, 687, 582, 418, 409; ^{1}H-NMR (600 MHz, $CDCl_3$) *δ*: 0.87 (3H, t, *J*=7 Hz, Me), 1.24—1.37 (14H, m, CH_2), 1.55—1.58 (6H, m, CH_2), 3.64 (2H, t, *J*=7 Hz, CH_2), 3.81 (3H, s, OMe), 5.65 (1H, s, CH), 6.92 (2H, d, *J*=8 Hz, ArH), 7.21 (1H, t, *J*=7 Hz, ArH), 7.32—7.34 (2H, m, ArH), 7.40 (2H, d, *J*=8 Hz, ArH), 7.45 (2H, d, *J*=9 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) *δ*: 14.1 (q), 22.7 (t), 26.2 (t), 29.3 (t), 29.4 (t), 29.6 (t×3), 29.6 (t×2), 31.9 (t), 55.3 (q), 68.4 (t), 72.0 (d), 73.8 (s), 97.5 (s), 113.9 (d×2), 126.2 (d×2), 126.5 (d), 128.7 (d×2), 129.2 (d×2), 130.9 (s), 132.7 (s), 159.7 (s); MS *m/z*: 438 (M^+), 329 (M^+−PhS), 253 (M^+−$OC_{12}H_{25}$). High resolution mass Calcd for $C_{28}H_{38}O_2S$: 438.2592, Found: 438.2584.

1-Methoxy-4-[1-(cyclopentyloxy)-3-(phenylsulfanyl)-2-propyn-1-yl]-benzene (2ad) A pale yellow oil, IR (KBr, cm^{-1}) *ν*: 2956, 2870, 2836, 2178, 1887, 1712, 1610, 1583, 1510, 1478, 1442, 1329, 1303, 1249, 1173, 1079, 1025, 831, 771, 740, 688, 580, 421; ^{1}H-NMR (600 MHz, $CDCl_3$) *δ*: 1.51—1.56 (2H, m, CH_2), 1.72—1.84 (6H, m, CH_2), 3.80 (3H, s, OMe), 4.30—4.31 (1H, m, CH), 5.32 (1H, s, CH), 6.90 (2H, d, *J*=9 Hz, ArH), 7.19—7.21 (1H, m, ArH), 7.29—7.32 (2H, m, ArH), 7.39—7.40 (2H, m, ArH), 7.44—7.45 (2H, m, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) *δ*: 23.6 (t×2), 32.0 (t), 32.8 (t), 55.3 (q), 70.1 (d), 73.2 (s), 79.5(d), 98.1 (s), 113.8 (d×2), 126.2 (d×2), 126.4 (d), 128.6 (d×2), 129.2 (d×2), 131.3 (s), 132.7 (s), 159.6 (s); MS *m/z*: 269 (M^+−C_5H_9), 253 (M^+−C_5H_9O). *Anal.* Calcd for $C_{21}H_{22}O_2S$: C, 74.52; H, 6.55. Found: C, 74.31; H, 6.53.

1-Methoxy-4-[1-(cyclohexyloxy)-3-(phenylsulfanyl)-2-propyn-1-yl]-benzene (2ae) Yellow powders, mp 46—49 °C, IR (KBr, cm^{-1}) *ν*: 2932, 2857, 2178, 1713, 1610, 1583, 1511, 1478, 1442, 1361, 1304, 1249, 1221, 1173, 1074, 1034, 950, 892, 831, 772, 740, 688; ^{1}H-NMR (600 MHz, $CDCl_3$) *δ*: 1.20—1.30 (3H, m, CH_2), 1.34—1.45 (2H, m, CH_2), 1.53—1.55 (1H, m, CH_2), 1.74—1.77 (2H, m, CH_2), 1.96 (2H, br s, CH_2), 3.67—3.70 (1H, m, CH), 3.80 (3H, s, OMe), 5.43 (1H, s, CH), 6.90 (2H, d, *J*=9 Hz, ArH), 7.20 (1H, t, *J*=7 Hz, ArH), 7.29—7.31 (2H, m, ArH), 7.38—7.40 (2H, m, ArH), 7.46 (2H, d, *J*=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) *δ*: 24.1 (t), 24.2 (t), 25.7 (t), 31.7 (t), 33.0 (t), 55.3 (q), 68.9 (d), 73.0 (s), 75.5 (d), 98.2 (s), 113.8 (d×2), 126.2 (d×2), 126.4 (d), 128.6 (d×2), 129.1 (d×2), 131.5 (s), 132.7 (s), 159.5 (s); MS *m/z*: 352 (M^+), 269 (M^+−cyclohexyl), 253 (M^+−OC_6H_{11}). *Anal.* Calcd for $C_{22}H_{24}O_2S$: C, 74.96; H, 6.86. Found: C, 75.10; H, 6.64.

1-Methoxy-4-[1-(geranyloxy)-3-(phenylsulfanyl)-2-propyn-1-yl]benzene (2af) A pale yellow oil, IR (KBr, cm^{-1}) *ν*: 3435, 2928, 2177, 1611, 1584, 1511, 1478, 1442, 1304, 1249, 1173, 1033, 831, 740, 688; ^{1}H-NMR (600 MHz, $CDCl_3$) *δ*: 1.60 (3H, s, Me), 1.67 (6H, d, *J*=1 Hz, Mex2), 2.03—2.06 (2H, m, CH_2), 2.09—2.13 (2H, m, CH_2), 3.80 (3H, s, OMe), 4.17 (2H, d, *J*=7 Hz, CH_2), 5.09 (1H, t, *J*=7 Hz, olefinic H), 5.36 (1H, s, OCH), 5.39 (1H, t, *J*=7 Hz, olefinic H), 6.90 (2H, d, *J*=9 Hz, ArH), 7.19—7.22 (1H, m, ArH), 7.31 (2H, br t, *J*=8 Hz, ArH), 7.41 (2H, d, *J*=8 Hz, ArH), 7.45 (2H, d, *J*=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) *δ*: 16.5 (q), 17.7 (q), 25.7 (q), 26.3 (t), 39.6 (t), 55.3 (q), 64.5 (t), 70.7 (d), 73.9 (s), 97.3 (s), 113.9 (d×2), 120.2 (d), 124.9 (d), 126.3 (d×2), 126.5 (d), 128.8 (d×2), 129.2 (d×2), 130.8 (s), 131.7 (s), 132.6 (s), 141.3 (s), 159.7 (s); MS *m/z*: 406 (M^+), 337 (M^+−CH_2CH=CMe_2). *Anal.* Calcd for $C_{26}H_{30}O_2S$: C, 76.81; H, 7.44. Found: C, 76.37; H, 7.41.

2-[1-Methoxy-3-(phenylthio)-2-propyn-1-yl]thiophene (2ba) A yellow oil, IR (KBr, cm^{-1}) *ν*: 3436, 2928, 2178, 1582, 1478, 1442, 1288, 1230, 1079, 1023, 931, 740, 706; ^{1}H-NMR (600 MHz, $CDCl_3$) *δ*: 3.46 (3H, s, OMe), 5.58 (1H, s, OCH), 6.99 (1H, dd, *J*=4 and 5 Hz, ArH), 7.19 (1H, d, *J*=4 Hz, ArH), 7.22—7.28 (1H, m, ArH), 7.33 (3H, br t, *J*=8 Hz, ArH), 7.44 (2H, br d, *J*=7 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) *δ*: 55.3 (q), 69.1 (d), 74.7 (s), 95.4 (s), 126.4 (d), 126.5 (d×2), 126.5 (d), 126.6 (d), 126.7 (d), 129.3 (d×2), 132.1 (s), 141.4 (s); MS *m/z*: 245 (M^+−Me), 229 (M^+−OMe); high resolution mass Calcd for $C_{14}H_{12}OS_2$: 260.0329, Found *m/z* 260.0274.

2-[1-Butoxy-3-(phenylthio)-2-propyn-1-yl]thiophene (2bb) A yellow oil, IR (KBr, cm^{-1}) *ν*: 3425, 2957, 2929, 2870, 2352, 2178, 1732, 1583, 1478, 1442, 1380, 1356, 1287, 1230, 1084, 1039, 1024, 999, 899, 855, 836, 739, 703, 688, ^{1}H-NMR (600 MHz, $CDCl_3$) *δ*: 0.91 (3H, t, *J*=7 Hz, Me), 1.37—1.43 (2H, m, CH_2), 1.58—1.63 (2H, m, CH_2), 3.55—3.59 (1H, m, OCH_2), 3.66—3.70 (1H, m, OCH_2), 5.62 (1H, s, OCH), 6.95 (1H, dd, *J*=1 and 5 Hz, ArH), 7.16 (1H, d, *J*=3 Hz, ArH), 7.19—7.21 (1H, m, ArH), 7.28—7.32 (3H, m, ArH), 7.42—7.43 (2H, m, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) *δ*: 13.8 (q), 19.3 (t), 31.6 (t), 67.8 (t×d), 74.0 (s), 96.1 (s), 126.1 (d), 126.2 (d), 126.4 (d×2), 126.5 (d), 126.6 (d), 129.2 (d×2), 132.2 (s), 142.0 (s); MS *m/z*: 302 (M^+), 245 (M^+−Bu), 229 (M^+−BuO); high resolution mass Calcd for $C_{17}H_{18}OS_2$: 302.0799, Found *m/z* 302.0789.

2-[1-(Cinnamyloxy)-3-(phenylthio)-2-propyn-1-yl]thiophene (2bc) A yellow oil, IR (KBr, cm^{-1}) *ν*: 3060, 3026, 2924, 2852, 2177, 1582, 1478, 1442, 1294, 1230, 1071, 967, 836, 740, 691; ^{1}H-NMR (600 MHz, $CDCl_3$) *δ*: 4.33—4.40 (2H, m, CH_2), 5.74 (1H, s, OCH), 6.30—6.35 (1H, m, olefinic H), 6.66 (1H, d, *J*=16 Hz, olefinic H), 6.99 (1H, dd, *J*=4 and 5 Hz, ArH), 7.17—7.25 (3H, m, ArH), 7.29—7.34 (5H, m, ArH), 7.38 (2H, d, *J*=8 Hz, ArH), 7.45 (2H, d, *J*=8 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) *δ*: 66.9 (d), 68.5 (t), 74.8 (s), 95.8 (s), 125.1 (d), 126.4 (d), 126.5 (d×2), 126.6 (d×2), 126.6 (d), 126.7 (d), 127.8 (d), 128.5 (d×2), 129.3 (d×2), 132.1 (s), 133.4 (d×2), 136.5 (s), 141.7 (s); MS *m/z*: 362 (M^+), 245 (M^+−cinnamyl), 229 (M^+−cinnamyloxy). *Anal.* Calcd for $C_{22}H_{18}OS_2$: C, 72.89; H, 5.00. Found: C, 72.56; H, 5.00.

2-[1-(Cyclopentyloxy)-3-(phenylthio)-2-propyn-1-yl]thiophene (2bd) A yellow oil, IR (KBr, cm^{-1}) *ν*: 2959, 2178, 1583, 1479, 1442, 1355, 1294, 1230, 1174, 1069, 1038, 1023, 972, 854, 738, 704, 687; ^{1}H-NMR (600 MHz, $CDCl_3$) *δ*: 1.49—1.58 (2H, m, CH_2), 1.75—1.85 (6H, m, CH_2), 4.32—4.35 (1H, m, CHO), 5.59 (1H, s, CH_2), 6.96 (1H, t, *J*=4 Hz, ArH), 7.16 (1H, d, *J*=3 Hz, ArH), 7.20—7.24 (1H, m, ArH), 7.29—7.33 (3H, m, ArH), 7.43 (2H, d, *J*=8 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) *δ*: 23.5 (t×2), 32.1 (t), 32.9 (t), 66.2 (d), 73.5 (s), 79.8 (d), 96.9 (s), 125.8 (d), 126.0 (d), 126.3 (d×2), 126.5 (d), 126.6 (d), 129.2 (d×2), 132.3 (s), 142.8 (s); MS *m/z*: 314 (M^+), 245 (M^+−C_5H_9), 229 (M^+−C_5H_9O). *Anal.* Calcd for $C_{18}H_{18}OS_2$: C, 68.75; H, 5.77. Found: C, 68.27; H, 5.77.

2-[1-(Cyclohexyloxy)-3-(phenylthio)-2-propyn-1-yl]thiophene (2be) A yellow oil, IR (KBr, cm^{-1}) ν: 3440, 2930, 2856, 2178, 1583, 1478, 1442, 1287, 1230, 1158, 1074, 1024, 739, 704, 688; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 1.22—1.32 (3H, m, CH_2), 1.38—1.47 (2H, m, CH_2), 1.54 (1H, br s, CH_2), 1.76 (2H, br d, J=10 Hz, CH_2), 1.95 (2H, br s, CH_2), 3.71—3.74 (1H, m, OCH), 5.70 (1H, s, OCH), 6.96 (1H, dd, J=5 and 3 Hz, ArH), 7.17 (1H, d, J=3 Hz, ArH), 7.22 (1H, q, J=8 Hz, ArH), 7.29 (1H, dd, J=4 and 5 Hz, ArH), 7.32 (2H, t, J=8 Hz, ArH), 7.42 (2H, d, J=8 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 24.0 (t), 24.1 (t), 25.7 (t), 31.7 (t), 32.9 (t), 65.1 (d), 73.2 (s), 75.8 (d), 97.0 (s), 125.7 (d), 125.9 (d), 126.3 (d×2), 126.5 (d), 126.6 (d), 129.2 (d×2), 132.3 (s), 142.9 (s); MS m/z: 245 (M^+−cyclohexyl), 229 (M^+−cyclohexyloxy). *Anal.* Calcd for $C_{19}H_{20}OS_2$: C, 69.47; H, 6.14. Found: C, 69.17; H, 6.13.

1-[1-Butoxy-3-(phenylthio)-2-propyn-1-yl]naphthalene (2ca) A yellow oil, IR (KBr, cm^{-1}) ν: 3059, 2957, 2869, 2176, 1582, 1510, 1478, 1442, 1300, 1256, 1165, 1083, 1045, 1024, 801, 780, 739, 687, 630, 586, 510, 424; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.88 (3H, t, J=7 Hz, ArH), 1.36—1.42 (2H, m, CH_2), 1.61—1.65 (2H, m, CH_2), 3.57—3.61 (1H, m, OCH_2), 3.76—3.79 (1H, m, OCH_2), 5.92 (1H, s, OCH), 7.15—7.18 (1H, m, ArH), 7.22—7.24 (2H, m, ArH), 7.33—7.35 (2H, m, ArH), 7.43—7.45 (1H, m, ArH), 7.46—7.52 (1H, m, ArH), 7.53—7.56 (1H, m, ArH), 7.75 (1H, d, J=7 Hz, ArH), 7.83 (1H, d, J=8 Hz, ArH), 7.87 (1H, d, J=8 Hz, ArH), 8.35 (1H, br d, J=9 Hz, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 13.8 (q), 19.4 (t), 31.8 (t), 68.5 (t), 71.4 (d), 74.6 (s), 97.2 (s), 124.5 (d), 125.1 (d), 125.8 (d), 125.9 (d), 126.2 (d), 126.2 (d×2), 126.5 (d), 128.6 (d), 129.1 (d×2), 129.3 (d), 130.8 (s), 132.6 (s), 133.9 (s), 134.1 (s); MS m/z: 346 (M^+), 289 (M^+−Bu), 273 (M^+−OBu). *Anal.* Calcd for $C_{23}H_{22}OS$: C, 79.73; H, 6.40. Found: C, 79.54; H, 6.37.

1,3,5-Trimethyl-2-[1-butoxy-3-(phenylthio)-2-propyn-1-yl]benzene (2da) A yellow oil, IR (KBr, cm^{-1}) ν: 2957, 2929, 2870, 2170, 1610, 1582, 1478, 1442, 1378, 1308, 1087, 1024, 851, 738, 687; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.90 (3H, t, J=7 Hz, Me), 1.36—1.42 (2H, m, CH_2), 1.56—1.64 (2H, m, CH_2), 2.25 (3H, s, Me), 2.46 (6H, s, Me×2), 3.38—3.41 (1H, m, OCH_2), 3.68—3.72 (1H, m, OCH_2), 5.66 (1H, s, OCH), 6.84 (2H, s, ArH), 7.17—7.20 (1H, m, ArH), 7.27—7.30 (2H, m, ArH), 7.35—7.36 (2H, m, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 13.8 (q), 19.4 (t), 20.4 (q×2), 20.9 (q), 31.8 (t), 68.1 (d), 68.5 (t), 72.8 (s), 97.2 (s), 126.3 (d×2), 126.4 (d), 129.1 (d×2), 129.8 (d×2), 132.0 (s), 132.9 (s), 136.7 (s×2), 137.6 (s); MS m/z: 281 (M^+−Bu), 265 (M^+−BuO). *Anal.* Calcd for $C_{22}H_{26}OS$: C, 78.06; H, 7.74. Found: C, 77.59; H, 7.55.

1-Fluoro-4-[1-butoxy-1-phenyl-3-(phenylthio)-2-propyn-1-yl]benzene (2ea) A yellow oil, IR (KBr, cm^{-1}) ν: 3062, 2958, 2932, 2871, 2179, 1731, 1604, 1583, 1508, 1478, 1442, 1412, 1381, 1296, 1226, 1156, 1086, 836, 787, 739, 687, 569, 509; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.91 (3H, t, J=7 Hz, Me), 1.36—1.44 (2H, m, CH_2), 1.61—1.65 (2H, m, CH_2), 3.47—3.65 (1H, m, OCH_2), 3.70—3.74 (1H, m, OCH_2), 5.34 (1H, s, OCH), 7.04—7.08 (2H, m, ArH), 7.20—7.24 (1H, m, ArH), 7.30—7.32 (2H, m, ArH), 7.38—7.40 (2H, m, ArH), 7.49—7.52 (2H, m, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 13.8 (q), 19.4 (t), 31.6 (t), 68.4 (t), 71.0 (d), 76.8 (s), 96.8 (s), 115.3 (d), 115.4 (d), 126.3 (d×2), 126.6 (d), 129.0 (d), 129,1 (d), 129.2 (d×2), 132.4 (s), 134.5 (s), 162.6 (d, J=247 Hz); MS m/z: 314 (M^+), 257 (M^+−Bu); high resolution mass Calcd for $C_{19}H_{19}FOS$: 314.1140, Found m/z 314.1086.

1-Fluoro-4-[1-methoxy-1-phenyl-3-(phenylthio)-2-propyn-1-yl]benzene (2eb) A yellow oil, IR (KBr, cm^{-1}) ν: 2931, 2179, 1604, 1583, 1508, 1478, 1327, 1226, 1184, 1157, 1082, 837, 789, 740, 688, 752; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 3.47 (3H, s, OMe), 5.28 (1H, s, OCH), 7.05—7.08 (2H, m, ArH), 7.21—7.24 (1H, m, ArH), 7.32 (2H, br t, J=7 Hz, ArH), 7.40 (2H, d, J=8 Hz, ArH), 7.49—7.50 (2H, m, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 56.0 (q), 73.2 (d), 75.2 (s), 96.1 (s), 115.4 (d), 115.5 (d), 126.4 (d×2), 126.7 (d), 129.1 (d), 129.2 (d), 129.3 (d×2), 132.3 (s), 134.1 (s), 162.8 (d, J=247 Hz); MS m/z: 272 (M^+), 241 (M^+−OMe); high resolution mass Calcd for $C_{16}H_{13}FOS$: 272.0671, Found m/z 272.0601.

5-[1-Allyloxy-3-(phenylsulfanyl)-2-propyn-1-yl]-1,3-benzodioxole (2fa) A pale yellow oil, IR (KBr, cm^{-1}) ν: 3292, 2898, 2178, 1582, 1503, 1488, 1442, 1379, 1303, 1247, 1103, 1040, 937, 863, 813, 740, 687; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 4.10—4.13 (1H, m, OCH_2), 4.19—4.22 (1H, m, OCH_2), 5.23 (1H, br d, J=11 Hz, olefinic H), 5.32 (1H, s, CHO), 5.35 (1H, dd, J=2, 17 Hz, olefinic H), 5.92—5.98 (3H, m, olefinic H, OCH_2O), 6.79 (1H, d, J=8 Hz, ArH), 6.99 (1H, dd, J=2, 8 Hz, ArH), 7.04 (1H, d, J=2 Hz, ArH), 7.21—7.25 (1H, m, ArH), 7.32 (2H, t, J=8 Hz, ArH), 7.41 (2H, d, J=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 69.1 (t), 71.3 (d), 74.5 (s), 96.7 (s), 101.2 (t), 108.0 (d), 108.1 (d), 117.9 (t), 121.1 (d), 126.3 (d×2), 126.6 (d), 129.2 (d×2), 132.2 (s), 132.4 (s), 134.1 (d), 147.8 (s), 147.9 (s); MS m/z: 267 (M^+−$OCH_2CH{=}CH_2$). *Anal.* Calcd for $C_{19}H_{16}O_3S$: C, 70.35; H, 4.97. Found: C, 70.43; H, 5.07.

5-[1-Methoxy-3-(phenylsulfanyl)-2-propyn-1-yl]-1,3-benzodioxole (2fb) A pale yellow oil, IR (KBr, cm^{-1}) ν: 3430, 2926, 2179, 1582, 1503, 1488, 1442, 1246, 1102, 1082, 1039, 936, 864, 812, 740, 688; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 3.44 (3H, s, OMe), 5.20 (1H, s, OCH), 5.96 (2H, s, OCH_2O), 6.80 (1H, d, J=8 Hz, ArH), 6.99 (1H, dd, J=1, 8 Hz, ArH), 7.02 (1H, d, J=1 Hz, ArH), 7.21—7.23 (1H, m, ArH), 7.31—7.34 (2H, m, ArH), 7.41 (2H, d, J=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 55.8 (q), 73.6 (d), 74.7 (s), 96.3 (s), 101.2 (t), 107.9 (d), 108.0 (d), 121.1 (d), 126.4 (d×2), 126.6 (d), 129.2 (d×2), 132.1 (s), 132.4 (s), 147.8 (s), 147.9 (s); MS m/z: 283 (M^+−Me), 267 (M^+−OMe). *Anal.* Calcd for $C_{17}H_{14}O_3S$: C, 68.44; H, 4.73. Found: C, 67.96; H, 4.74.

5-[1-Cyclopentyloxy-3-(phenylsulfanyl)-2-propyn-1-yl]-1,3-benzodioxole (2fc) A pale yellow oil, IR (KBr, cm^{-1}) ν: 2957, 1714, 1583, 1504, 1488, 1442, 1246, 1079, 1039, 933, 812, 740, 688; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 1.53—1.55 (2H, m, CH_2), 1.71—1.85 (6H, m, CH_2), 4.28—4.31 (1H, m, OCH), 5.26 (1H, s, OCH), 5.95 (2H, s, OCH_2O), 6.78 (1H, d, J=8 Hz, ArH), 6.98 (1H, dd, J=1, 8 Hz, ArH), 7.02 (1H, d, J=1 Hz, ArH), 7.19—7.24 (1H, m, ArH), 7.29—7.32 (2H, m, ArH), 7.39 (2H, dd, J=1, 9 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 23.6 (t×2), 32.0 (t), 32.8 (t), 70.3 (d), 73.4 (s), 79.6 (d), 97.7 (s), 101.1 (t), 107.9 (d), 108.0 (d), 120.9 (d), 126.3 (d×2), 126.5 (d), 129.2 (d×2), 132.6 (s), 133.1 (s), 147.6 (s), 147.8 (s); MS m/z: 352 (M^+), 283 (M^+−C_5H_9), 267 (M^+−C_5H_9O). *Anal.* Calcd for $C_{21}H_{20}O_3S$: C, 71.57; H, 5.72. Found: C, 71.07; H, 5.93.

6-Benzyloxy-6-(plenylsulfanylethynyl)undecane (2ga) A colorless oil, IR (KBr, cm^{-1}) ν: 3063, 3031, 2954, 2862, 2164, 1942, 1868, 1799, 1716, 1583, 1496, 1478, 1455, 1442, 1378, 1360, 1309, 1245, 1217, 1139, 1086, 1026, 930, 737, 695, 688; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.90 (6H, t, J=7 Hz, Me×2), 1.33 (8H, br s, CH_2), 1.50—1.54 (5H, m, CH_2), 1.82 (4H, t, J=8 Hz, CH_2), 4.64 (2H, s, CH_2), 7.21 (1H, br t, J=7 Hz, ArH), 7.25 (1H, br t, J=7 Hz, ArH), 7.30—7.33 (3H, m, ArH), 7.36 (2H, br d, J=7 Hz, ArH), 7.41 (2H, br d, J=7 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 14.1 (q×2), 22.6 (t×2), 23.7 (t×2), 32.1 (t×2), 38.6 (t×2), 66.2 (t), 72.3 (s), 78.0 (s), 100.6 (s), 126.0 (d×2), 126.4 (d), 127.2 (d), 127.6 (d×2), 128.2 (d×2), 129.2 (d×2), 133.0 (s), 139.2 (s); MS m/z: 394 (M^+), 303 (M^+−Bn), 286 (M^+−BnOH), 261 (M^+−C_2SPh). *Anal.* Calcd for $C_{26}H_{34}OS$: C, 79.14; H, 8.68. Found: C, 79.03; H, 8.68.

1-[1-Butoxy-1-phenyl-3-(phenylsulfanyl)-2-propyn-1-yl]benzene (2ha) A pale yellow oil, IR (KBr, cm^{-1}) ν: 3060, 3025, 2957, 2930, 2871, 2171, 1583, 1489, 1478, 1448, 1380, 1194, 1177, 1079, 1065, 1041, 1027, 968, 931, 902, 739, 699, 666, 651; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.91 (3H, t, J=7 Hz, Me), 1.41—1.48 (2H, m, CH_2), 1.63—1.67 (2H, m, CH_2), 3.53 (2H, t, J=7 Hz, OCH_2), 7.18—7.21 (1H, m, ArH), 7.24—7.28 (4H, m, ArH), 7.30—7.36 (6H, m, ArH), 7.57 (4H, d, J=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 13.9 (q), 19.5 (t), 32.0 (t), 64.6 (t), 76.0 (s), 81.0 (s), 99.1 (s), 126.2 (d×2), 126.5 (d), 126.7 (d×4), 127.6 (d×2), 128.2 (d×4), 129.2 (d×2), 132.8 (s), 143.6 (s×2); MS m/z: 372 (M^+), 315 (M^+−Bu), 299 (M^+−BuO). *Anal.* Calcd for $C_{25}H_{24}OS$: C, 80.61; H, 6.49. Found: C, 80.63; H, 6.71.

1-(Dodecyloxy)-1-(phenylsulfanylethynyl)cyclohexane (2ia) A colorless oil, IR (KBr, cm^{-1}) ν: 2928, 2854, 1709, 1634, 1584, 1479, 1465, 1442, 1415, 1363, 1341, 1258, 1227, 1160, 1069, 1035, 1024, 965, 903, 738, 687; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.88 (3H, t, J=7 Hz, Me), 1.23—1.30 (18H, m, CH_2), 1.54—1.74 (10H, m, CH_2), 1.98—2.04 (2H, m, CH_2), 3.62 (2H, t, J=6 Hz, OCH_2), 7.21 (1H, t, J=7 Hz, ArH), 7.32 (2H, t, J=7 Hz, ArH), 7.40 (2H, dd, J=1, 8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 14.1 (q), 22.6 (t), 23.2 (t), 25.1 (t), 25.7 (t), 29.3 (t), 29.4 (t), 29.6 (t×2), 29.7 (t×3), 31.9 (t), 32.7 (t), 39.8 (t×2), 63.0 (t), 69.6 (s), 70.4 (s), 102.6 (s), 125.9 (d×2), 126.4 (d), 129.2 (d×2), 132.8 (s); MS m/z: 400 (M^+), 215 (M^+−$OC_{12}H_{25}$); high resolution mass Calcd for $C_{26}H_{40}OS$: 400.2799, Found m/z 400.2783.

1-(Dodecyloxy)-1-(phenylsulfanylethynyl)cyclopentane (2ja) A pale yellow oil, IR (KBr, cm^{-1}) ν: 3076, 3062, 2956, 2925, 2871, 2853, 2363, 2170, 1703, 1584, 1478, 1467, 1441, 1378, 1314, 1210, 1181, 1075, 1024, 997, 945, 919, 738, 687; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.88 (3H, t, J=7 Hz, Me), 1.26—1.35 (18H, m, CH_2), 1.53—1.58 (2H, m, CH_2), 1.77—1.80 (2H, m, CH_2), 1.85—1.88 (2H, m, CH_2), 2.02—2.07 (4H, m, CH_2), 3.63 (2H, t, J=7 Hz, CH_2), 7.21 (1H, t, J=7 Hz, ArH), 7.33 (2H, t, J=8 Hz, ArH), 7.39—7.41 (2H, m, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 14.1 (q), 22.6 (t), 23.5 (t×2), 25.7 (t), 29.3 (t), 29.4 (t), 29.6 (t×2, t), 29.6 (t), 31.9 (t), 32.7 (t), 42.5 (t×2), 63.0 (t), 69.6 (s), 75.3 (s), 102.6 (s), 125.9 (d×2), 126.4 (d×2), 129.2 (d), 132.8 (s); MS m/z: 386 (M^+), 277 (M^+−SPh), 201 (M^+−$OC_{12}H_{25}$). *Anal.* Calcd for $C_{25}H_{38}OS$: C, 77.66; H, 9.91. Found: C, 77.55; H, 9.90.

1-(Dodecyloxy)-1-(phenylsulfanylethynyl)cycloheptane (2ka) A pale yellow oil, IR (KBr, cm^{-1}) ν: 2926, 2854, 2364, 2174, 1938, 1706, 1584, 1479, 1461, 1442, 1378, 1350, 1200, 1123, 1024, 957, 909, 846, 738, 723, 687, 667; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.88 (3H, t, J=6 Hz, Me), 1.30 (18H, br s, CH_2), 1.53—1.62 (8H, m, CH_2), 1.69 (2H, br s, CH_2), 1.90—1.94 (2H, m, CH_2), 2.04—2.13 (2H, m, CH_2), 3.62 (2H, t, J=7 Hz, CH_2), 7.21 (1H, t, J=7 Hz, ArH), 7.32 (2H, t, J=8 Hz, ArH), 7.41 (2H, d, J=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 14.1 (q), 22.2 (t×2), 22.6 (t), 25.7 (t), 28.0 (t×2), 29.3 (t), 29.4 (t), 29.6 (t×2), 29.6 (t×2), 31.9 (t), 32.7 (t), 42.9 (t×2), 63.0 (t), 69.6 (s), 72.7 (s), 103.7 (s), 125.9 (d×2), 126.3 (d), 129.1 (d×2), 132.9 (s); MS m/z: 414 (M^+), 305 (M^+-SPh), 229 ($M^+-OC_{12}H_{25}$). *Anal.* Calcd for $C_{27}H_{42}OS$: C, 78.20; H, 10.21. Found: C, 78.24; H, 10.14.

1-Methoxy-4-[3-(phenylselanyl)-1-methoxy-2-propyn-1-yl]benzene (4aa) A pale yellow oil, IR (KBr, cm^{-1}) ν: 2933, 2836, 2168, 1890, 1712, 1675, 1602, 1577, 1510, 1476, 1439, 1359, 1304, 1250, 1173, 1081, 1022, 832, 737, 688, 580; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 3.43 (3H, s, OMe), 3.80 (3H, s, OMe), 5.24 (1H, s, CH), 6.91 (2H, d, J=9 Hz, ArH), 7.24—7.26 (1H, m, ArH), 7.28—7.31 (2H, m, ArH), 7.44 (2H, d, J=9 Hz, ArH), 7.51—7.52 (2H, m, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 55.3 (q), 55.7 (q), 67.8 (s), 73.6 (d), 101.5 (s), 113.9 (d×2), 127.1 (d), 128.5 (s), 128.8 (d×2), 129.1 (d×2), 129.5 (d×2), 130.4 (s), 159.8 (s); MS m/z: 332 (M^+), 301 (M^+-OMe), 175 (M^+-PhSe), 151 (M^+-C_2SePh). *Anal.* Calcd for $C_{17}H_{16}O_2Se$: C, 61.63; H, 4.87. Found: C, 61.33; H, 4.89.

1-[1-Benzyloxy-3-(phenylselanyl)-2-propyn-1-yl]benzodioxole (4ba) A pale yellow oil, IR (KBr, cm^{-1}) ν: 3060, 3030, 2892, 2169, 1952, 1865, 1608, 1577, 1502, 1487, 1441, 1373, 1303, 1247, 1208, 1123, 1100, 1084, 1065, 1039, 999, 935, 864, 811, 784, 736, 698, 688, 666; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 4.64 (1H, d, J=16 Hz, CH_2), 4.71 (1H, d, J=16 Hz, CH_2), 5.30 (1H, s, CH), 5.93 (2H, s, OCH_2O), 6.77 (1H, d, J=8 Hz, ArH), 6.98 (1H, dd, J=1, 8 Hz, ArH), 7.04 (1H, d, J=1 Hz, ArH), 7.21—7.30 (4H, m, ArH), 7.32—7.37 (4H, m, ArH), 7.51 (2H, d, J=7 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 68.1 (s), 69.9 (t), 71.1 (d), 101.1 (t), 101.3 (s), 108.0 (d), 108.1 (d), 121.2 (d), 127.2 (d), 127.7 (d), 128.1 (d×2), 128.3 (s), 128.4 (d×2), 129.1 (d×2), 129.5 (d×2), 132.3 (s), 137.5 (s), 147.7 (s), 147.8 (s); MS m/z: 422 (M^+), 315 (M^+-OCH_2Ph), 265 (M^+-PhSe). *Anal.* Calcd for $C_{23}H_{18}O_3Se$: C, 65.60; H, 4.31. Found: C, 65.24; H, 4.29.

2-[Benzyloxy-3-(phenylselanyl)-2-propyn-1-yl]naphthalene (4ca) A pale yellow oil, IR (KBr, cm^{-1}) ν: 3060, 2924, 2856, 2169, 1948, 1875, 1598, 1577, 1509, 1476, 1454, 1440, 1395, 1379, 1359, 1302, 1258, 1233, 1210, 1166, 1083, 1065, 1050, 1021, 999, 932, 905, 802, 780, 735, 698, 687, 666; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 4.76 (2H, s, CH_2), 5.99 (1H, s, CH), 7.18—7.23 (3H, m, ArH), 7.24—7.40 (5H, m, ArH), 7.43—7.52 (5H, m, ArH), 7.76 (1H, d, J=7 Hz, ArH), 7.80—7.87 (2H, m, ArH), 8.28—8.29 (1H, m, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 68.8 (s), 70.1 (t), 70.3 (d), 101.4 (s), 124.5 (d), 125.1 (d), 125.8 (d), 126.2 (d), 126.2 (d), 126.9 (d), 127.1 (d), 127.8 (d), 128.3 (d×2), 128.4 (d×2), 128.5 (s), 128.6 (d), 129.1 (d×2), 129.5 (d×2), 130.8 (s), 133.6 (s), 134.1 (s), 137.5 (s); MS m/z: 428 (M^+), 321 (M^+-OCH_2Ph), 271 (M^+-SePh). *Anal.* Calcd for $C_{26}H_{20}Se$: C, 73.07; H, 4.72. Found: C, 72.97; H, 4.86.

1-Benzyloxy-1-[3-(phenylselanyl)-1-propyn-1-yl]cyclohexane (4da) A pale yellow oil, IR (KBr, cm^{-1}) ν: 3061, 3031, 2934, 2856, 2359, 2341, 2162, 1946, 1864, 1803, 1714, 1577, 1496, 1451, 1441, 1379, 1339, 1286, 1256, 1203, 1149, 1087, 1066, 1022, 999, 967, 937, 905, 853, 734, 696, 687, 667; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 1.32—1.36 (1H, m, CH_2), 1.53—1.62 (4H, m, CH_2), 1.70—1.79 (4H, m, CH_2), 2.04 (2H, t, J=7 Hz, CH), 4.69 (2H, s, CH_2), 7.22—7.33 (5H, m, ArH), 7.37 (2H, d, J=7 Hz, ArH), 7.50—7.52 (2H, m, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 22.8 (t), 25.4 (t×2), 37.3 (t×2), 65.2 (s), 65.7 (t), 75.2 (s), 105.6 (s), 126.9 (d), 127.3 (d), 127.7 (d×2), 128.2 (d×2), 128.7 (d×2), 128.9 (s), 129.5 (d×2), 139.1 (s); MS m/z: 370 (M^+), 263 (M^+-OCH_2Ph), 213 (M^+-PhSe). *Anal.* Calcd for $C_{21}H_{22}OSe$: C, 68.29; H, 6.00. Found: C, 68.54; H, 6.10.

1-Methoxy-4-(1-benzyloxy-2-propyn-1-yl)benzene (7aa) A yellow oil, IR (KBr, cm^{-1}) ν: 3287, 3033, 2933, 2838, 2360, 2112, 1611, 1512, 1455, 1421, 1389, 1305, 1250, 1174, 1060, 1036, 930, 831, 749, 699, 650, 589, 556; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 2.64 (1H, d, J=3 Hz, acetylenic H), 3.74 (3H, s, OMe), 4.60 (1H, d, J=12 Hz, OCH_2), 4.67 (1H, d, J=12 Hz, OCH_2), 5.14 (1H, d, J=2 Hz, OCH), 6.87 (2H, d, J=8 Hz, ArH), 7.24—7.27 (1H, m, ArH), 7.30—7.36 (4H, m, ArH), 7.43 (2H, d, J=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 55.1 (q), 69.7 (t), 75.6 (d), 81.6 (s), 113.8 (d×2), 127.7 (d), 128.0 (d×2), 128.3 (d×2), 128.7 (d×2), 130.2 (s), 137.5 (s), 159.7 (s); MS m/z: 252 (M^+), 221 (M^+-OMe). *Anal.* Calcd for $C_{17}H_{16}O_2$: C, 80.09; H, 6.39. Found: C, 79.75; H, 6.52.

1-Methoxy-4-(1-dodecyloxy-2-propyn-1-yl)benzene (7ab) A yellow oil, IR (KBr, cm^{-1}) ν: 3452, 3309, 2925, 2854, 2360, 2112, 1714, 1610, 1512, 1465, 1360, 1304, 1250, 1173, 1084, 1037, 829, 775, 721, 657, 633, 584, 530; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.88 (3H, t, J=7 Hz, Me), 1.25 (18H, br s, CH_2), 1.58—1.63 (2H, m, CH_2), 2.60 (1H, d, J=3 Hz, acetylenic H), 3.44—3.48 (1H, m, OCH_2), 3.60—3.64 (1H, m, OCH_2), 3.80 (3H, s, OMe), 5.09 (1H, d, J=2 Hz, OCH), 6.89 (2H, d, J=9 Hz, ArH), 7.43 (2H, d, J=9 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 14.1 (q), 22.7 (t), 26.1 (t), 29.3 (t), 29.4 (t), 29.5 (t), 29.6 (t×2), 29.7 (t×2), 31.9 (t), 55.3 (q), 68.4 (t), 70.8 (d), 75.0 (d), 82.2 (s), 113.8 (d×2), 128.6 (d×2), 130.7 (s), 159.7 (s); MS m/z: 330 (M^+); high resolution mass Calcd for $C_{22}H_{34}O_2$: 330.2558, Found m/z 330.2516.

1-Methoxy-4-(1-allyloxy-2-propyn-1-yl)benzene (7ac) A yellow oil, IR (KBr, cm^{-1}) ν: 3290, 3079, 3005, 2957, 2838, 2112, 1713, 1647, 1612, 1578, 1513, 1464, 1422, 1335, 1304, 1250, 1174, 830, 778, 658, 583, 542; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 2.63 (1H, d, J=2 Hz, acetylenic H), 3.81 (3H, s, OMe), 4.08—4.10 (1H, m, OCH_2), 4.15—4.16 (1H, m, OCH_2), 5.16 (1H, d, J=3 Hz, OCH), 5.21 (1H, dd, J=1, 10 Hz, olefinic H), 5.32 (1H, dd, J=1, 15 Hz, olefinic H), 5.91—5.97 (1H, m, olefinic H), 6.90 (2H, d, J=9 Hz, ArH), 7.44 (2H, d, J=9 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 55.3 (q), 69.0 (t), 69.9 (d), 75.3 (d), 81.8 (s), 113.9 (d×2), 117.8 (t), 128.8 (d×2), 130.4 (s), 134.2 (d), 159.8 (s); MS m/z: 202 (M^+); high resolution mass Calcd for $C_{13}H_{14}O_2$: 202.0994, Found m/z 202.0944.

1-Methoxy-4-(1-isopropyloxy-2-propyn-1-yl)benzene (7ad) A yellow oil, IR (KBr, cm^{-1}) ν: 3289, 2972, 2933, 2837, 2359, 2112, 1713, 1613, 1586, 1513, 1465, 1420, 1373, 1334, 1304, 1249, 1173, 1122, 1037, 969, 914, 819, 914, 819, 771, 655, 586, 540; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 1.20 (3H, d, J=7 Hz, Me), 1.23 (3H, d, J=7 Hz, Me), 2.57 (1H, d, J=2 Hz, acetylenic H), 3.80 (3H, s, OMe), 3.91—3.99 (1H, m, OCH), 5.14 (1H, d, J=1 Hz, OCH), 6.89 (2H, d, J=8 Hz, ArH), 7.42 (2H, d, J=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 21.7 (q), 22.7 (q), 55.3 (q), 68.2 (d), 69.5 (d), 74.4 (d), 82.9 (s), 113.9 (d×2), 128.6 (d×2), 131.3 (s), 159.6 (s); MS m/z: 204 (M^+); high resolution mass Calcd for $C_{13}H_{16}O_2$: 204.1150, Found m/z 204.1080.

1-(1-Propargyloxy-2-propyn-1-yl)benzene (7bb)[46,47]

1-Allyloxy-3-phenyl-2-propyn-1-ylbenzene (7ca) A yellow oil, IR (KBr, cm^{-1}) ν: 3066, 3032, 2923, 2858, 2225, 1716, 1601, 1493, 1450, 1277, 1180, 1061, 926, 756, 694, 532; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 4.16—4.19 (1H, m, OCH_2), 4.24—4.26 (1H, m, OCH_2), 5.23 (1H, dd, J=1, 10 Hz, olefinic H), 5.35 (1H, dd, J=1, 14 Hz, olefinic H), 5.44 (1H, s, OCH), 5.96—6.02 (1H, m, olefinic H), 7.29—7.31 (3H, m, ArH), 7.32—7.33 (1H, m, ArH), 7.34—7.39 (2H, m, ArH), 7.47—7.48 (2H, m, ArH), 7.59 (2H, d, J=7 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 69.2 (t), 71.1 (d), 86.9 (s), 87.5 (s), 117.7 (t), 122.5 (s), 127.5 (d×2), 128.2 (d×2), 128.4 (d), 128.5 (d×3), 131.8 (d×2), 134.3 (d), 138.7 (s); MS m/z: 248 (M^+), 191 (M^+-allyloxy); high resolution mass Calcd for $C_{18}H_{16}O$: 248.1201, Found m/z 248.1161.

1-(3-Phenyl-1-propargyloxyprop-2-yn-1-yl)benzene (7cb)[46,47] A yellow oil, IR (KBr, cm^{-1}) ν: 3294, 3062, 3033, 2903, 2854, 2360, 2224, 2117, 1640, 1599, 1574, 1491, 1454, 1443, 1330, 1311, 1193, 1177, 1062, 1028, 984, 938, 918, 900, 844, 758, 726, 692, 640, 591, 556, 528; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 2.48 (1H, t, J=2 Hz, acetylenic H), 4.31 (1H, dd, J=2, 14 Hz, OCH_2), 4.44 (1H, dd, J=2, 14 Hz, OCH_2), 5.64 (1H, s, OCH), 7.29—7.31 (4H, m, ArH), 7.34—7.40 (2H, m, ArH), 7.47—7.48 (2H, m, ArH), 7.59 (2H, d, J=7 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 55.4 (t), 70.5 (d), 75.0 (d), 79.3 (s), 85.9 (s), 88.0 (s), 122.3 (s), 127.7 (d×2), 128.2 (d×2), 128.5 (d×2), 128.6 (d×2), 131.8 (d×2), 137.9 (s); MS m/z: 245 (M^+-1), 191 (M^+-OCH_2CCH). *Anal.* Calcd for $C_{18}H_{14}O$: C, 87.78; H, 5.73. Found: C, 87.56; H, 5.88.

1-Cyclopentyloxy-3-phenylprop-2-yn-1-ylbenzene (7cc) White powders, mp 90—91 °C, IR (KBr, cm^{-1}) ν: 3062, 3032, 2958, 2871, 2224, 1715, 1599, 1491, 1451, 1369, 1306, 1278, 1175, 1053, 1004, 990, 915, 757, 723, 692, 640, 527; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 1.51—1.56 (2H, m, CH_2), 1.72—1.87 (6H, m, CH_2), 4.35—4.37 (1H, m, OCH), 5.38 (1H, s, OCH), 7.29—7.32 (4H, m, ArH), 7.36—7.38 (2H, m, ArH), 7.46—7.48 (2H, m, ArH), 7.56—7.58 (2H, m, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 23.6 (t×2), 32.0 (t), 32.9 (t), 70.1 (d), 79.7 (d), 86.7 (s), 88.0 (s), 122.8 (s), 127.4 (d×2), 128.1 (d), 128.2 (d×2), 128.3 (d), 128.4 (d×2), 131.8 (d×2), 139.5 (s); MS m/z: 276 (M^+); high resolution mass Calcd for $C_{20}H_{20}O$: 276.1514, Found m/z 276.1501.

1-Cyclohexyloxy-3-phenylprop-2-yn-1-ylbenzene (7 cd) A yellow oil, IR (KBr, cm^{-1}) ν: 3062, 3031, 2931, 2856, 2220, 1949, 1716, 1599, 1491, 1451, 1375, 1328, 1303, 1278, 1189, 1156, 1132, 1068, 1028, 980, 945, 914, 847, 756, 723, 693, 652, 610, 565, 528; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 1.26—1.60 (6H, m, CH_2), 1.75—1.78 (2H, m, CH_2), 1.99—2.01 (2H, m,

CH_2), 3.71—3.76 (1H, m, OCH), 5.49 (1H, s, OCH), 7.28—7.30 (4H, m, ArH), 7.35—7.38 (2H, m, ArH), 7.45—7.47 (2H, m, ArH), 7.58—7.60 (2H, m, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 24.1 (t), 24.2 (t), 25.8 (t), 31.8 (t), 33.0 (t), 69.0 (d), 75.8 (d), 86.5 (s), 88.2 (s), 122.8 (s), 127.3 (d×2), 128.1 (d), 128.2 (d×2), 128.3 (d), 128.4 (d×2), 131.7 (d×2), 139.7 (s); MS *m/z*: 290 (M^+), 207 ($M^+-C_6H_5$), 191 ($M^+-C_6H_5O$); high resolution mass Calcd for $C_{21}H_{22}O$: 290.1670, Found *m/z* 290.1711.

1-Phenylpropargyloxy-2-heptyn-1-ylbenzene (7da) A yellow oil, IR (KBr, cm^{-1}) *v*: 3064, 3033, 2957, 2932, 2871, 2861, 2360, 2235, 1599, 1491, 1454, 1380, 1356, 1303, 1132, 1058, 1029, 914, 757, 694; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.92 (3H, t, *J*=7 Hz, Me), 1.41—1.47 (2H, m, CH_2), 1.51—1.57 (2H, m, CH_2), 2.30 (2H, dt, *J*=2, 7 Hz, CH_2), 4.45 (1H, d, *J*=16 Hz, OCH_2), 4.57 (1H, d, *J*=16 Hz, OCH_2), 5.47 (1H, s, OCH), 7.28—7.38 (6H, m, ArH), 7.45—7.50 (2H, m, ArH), 7.56 (2H, d, *J*=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 13.5 (q), 18.5 (t), 21.9 (t), 30.6 (t), 55.9 (t), 70.4 (d), 84.9 (s), 86.4 (s), 89.1 (s), 122.6 (s), 127.7 (d×2), 128.2 (d×2), 128.3 (d×2), 128.4 (d×2), 131.7 (d×2), 138.6 (s); MS *m/z*: 301 (M^+-1), 245 (M^+-Bu). *Anal.* Calcd for $C_{22}H_{22}O$: C, 87.38; H, 7.33. Found: C, 87.03; H, 7.41.

1-(Benzyloxy)-2-heptyn-1-ylbenzene (7db) A yellow oil, IR (KBr, cm^{-1}) *v*: 3063, 3032, 2957, 2932, 2862, 2274, 2224, 1715, 1604, 1495, 1454, 1381, 1328, 1303, 1272, 1203, 1176, 1132, 1085, 1061, 1028, 1001, 940, 914, 735, 697, 641, 602, 548; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.91 (3H, t, *J*=7 Hz, Me), 1.40—1.46 (2H, m, CH_2), 1.51—1.56 (2H, m, CH_2), 2.28 (2H, t, *J*=7 Hz, CH_2), 4.62 (1H, d, *J*=11 Hz, OCH_2), 4.68 (1H, d, *J*=11 Hz, OCH_2), 5.20 (1H, br s, OCH), 7.24-7.37 (8H, m, ArH), 7.51 (2H, d, *J*=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 13.5 (q), 18.5 (t), 21.9 (t), 30.7 (t), 69.0 (t), 70.6 (d), 77.2 (s), 88.6 (s), 127.4 (d×2), 127.6 (d), 128.0 (d×2), 128.1 (d), 128.3 (d×2), 137.9 (s), 139.2 (s); MS *m/z* 278 (M^+), 221 (M^+-Bu); high resolution mass Calcd for $C_{20}H_{22}O$: 278.1670, Found *m/z* 278.1676.

1-Methoxy-4-[1-(4-decanesulfanyl)-3-(phenylsulfanyl)-2-propyn-1-yl]-benzene (8aa) A brown oil, IR (KBr, cm^{-1}) *v*: 3061, 2925, 2853, 2187, 1609, 1583, 1509, 1478, 1464, 1441, 1377, 1303, 1249, 1110, 1034, 900, 834, 738, 687, 564, 528; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.88 (3H, t, *J*=7 Hz, Me), 1.24 (14H, br s, CH_2), 1.57—1.61 (2H, m, CH_2), 2.56—2.60 (1H, m, CH_2), 2.71—2.76 (1H, m, CH_2), 3.79 (3H, s, OMe), 4.96 (1H, s, CHS), 6.87 (2H, d, *J*=8 Hz, ArH), 7.19—7.21 (1H, m, ArH), 7.30—7.33 (2H, m, ArH), 7.42—7.44 (4H, m, ArH); ^{13}C-NMR (600 MHz, $CDCl_3$) δ: 14.08 (q), 22.64 (t), 28.94 (t), 29.08 (t), 29.17 (t), 29.28 (t), 29.47 (t), 29.52 (t), 31.86 (t×2), 39.47 (d), 55.27 (q), 71.31 (s), 96.93 (s), 113.95 (d×2), 126.13 (d×2), 126.42 (d), 128.94 (d×2), 129.18 (d×2), 130.09 (s), 132.96 (s), 159.15 (s); MS *m/z*: 426 (M^+). *Anal.* Calcd for $C_{26}H_{34}OS_2$: C, 73.19; H, 8.03. Found: C, 72.93; H, 7.97.

1-Methoxy-4-[1-(2-naphthylsulfanyl)-3-(phenylsulfanyl)-2-propyn-1-yl]benzene (8ab) Brown powers, mp 85—87 °C, IR (KBr, cm^{-1}) *v*: 3452, 3054, 2929, 2853, 1711, 1608, 1583, 1509, 1478, 1441, 1304, 1250, 1174, 1069, 1032, 834, 814, 740, 689, 472; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 3.72 (3H, s, OMe), 5.34 (1H, s, CH), 6.81 (2H, d, *J*=8 Hz, ArH), 7.01—7.06 (3H, m, ArH), 7.15 (2H, d, *J*=8 Hz, ArH), 7.36—7.48 (5H, m, ArH), 7.67—7.76 (3H, m, ArH), 7.93 (1H, br s, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 44.2 (d), 55.2 (q), 72.6 (s), 96.7 (s), 133.9 (d×2) 126.2 (d×2), 126.3 (d), 126.4 (d), 126.5 (d), 127.6 (d), 127.7 (d), 128.2 (d), 129.0 (d×2), 129.2 (d×2), 130.5 (d), 131.1 (s), 132.4 (s), 132.7 (s), 132.9 (d), 133.5 (s), 134.2 (s), 159.3 (s); MS *m/z*: 412 (M^+), 303 (M^+-SPh), 253 ($M^+-C_{10}H_7S$). *Anal.* Calcd for $C_{26}H_{20}OS_2$: C, 75.69; H, 4.89. Found: C, 75.42; H, 4.93.

4-[3-(Phenylsulfanyl)-1-(2-thienylsulfanyl)prop-2-ynyl]-1-methoxybenzene (8ac) A brown oil, IR (KBr, cm^{-1}) *v*: 2835, 2186, 1608, 1583, 1509, 1478, 1441, 1400, 1304, 1249, 1217, 1173, 1112, 1086, 1033, 833, 740, 708, 688, 467; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 3.79 (3H, s, OMe), 5.10 (1H, s, CH), 6.84 (2H, d, *J*=7 Hz, ArH), 6.95 (1H, dd, *J*=3, 5 Hz, ArH), 7.05—7.06 (1H, dd, *J*=1, 3 Hz, ArH), 7.21 (1H, t, *J*=7 Hz, ArH), 7.29—7.32 (4H, m, ArH), 7.35—7.39 (3H, m, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 47.2 (d), 55.3 (q), 73.5 (s), 96.3 (s), 113.9 (d×2), 126.3 (d×2), 126.5 (d), 127.5 (d), 129.1 (s), 129.2 (d×2), 129.2 (d×2), 131.0 (s), 131.5 (d), 132.6 (s), 136.7 (d), 159.4 (s); MS *m/z*: 368 (M^+), 291 (M^+-Ph), 253 ($M^+-C_4H_3S_2$); high resolution mass Calcd for $C_{20}H_{16}OS_3$: 368.0364, Found *m/z* 368.0352.

1-Methoxy-4-[1-(4-chlorophenylsulfanyl)-3-(phenylsulfanyl)-2-propyn-1-yl]benzene (8ad) White powders, mp 67—68 °C, IR (KBr, cm^{-1}) *v*: 2955, 2928, 2836, 2186, 1734, 1608, 1583, 1509, 1475, 1441, 1388, 1325, 1304, 1250, 1174, 1111, 1093, 1033, 822, 739, 688, 662; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 3.79 (3H, s, OMe), 5.20 (1H, s, CH), 6.84 (2H, d, *J*=7 Hz, ArH), 7.20—7.23 (3H, m, ArH), 7.26—7.29 (4H, m, ArH), 7.32 (4H, d, *J*=7 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 44.4 (d), 55.3 (q), 73.1 (s), 96.2 (s), 113.9 (d×2), 126.3 (d×2), 126.5 (d), 128.9 (d×2), 129.1 (d), 129.2 (d×3), 131.7 (s), 132.4 (s), 134.7 (s), 135.5 (d×2), 159.4 (s); MS *m/z*: 396 (M^+), 287 (M^+-SPh), 253 ($M^+-C_6H_4Cl$). *Anal.* Calcd for $C_{22}H_{17}ClOS_2$: C, 66.57; H, 4.32. Found: C, 66.47; H, 4.32.

1-Methoxy-4-[1-(cyclohexylsulfanyl)-3-(phenylsulfanyl)-2-propyn-1-yl]benzene (8ae) A yellow oil, IR (KBr, cm^{-1}) *v*: 2928, 2851, 2359, 2185, 1608, 1583, 1509, 1478, 1461, 1441, 1325, 1303, 1248, 1203, 1173, 1110, 1068, 1034, 999, 887, 834, 739, 688, 564, 529; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 1.21—1.46 (5H, m, CH_2), 1.56—1.59 (1H, m, CH_2), 1.72—1.75 (2H, m, CH_2), 1.90—1.92 (1H, m, CH_2), 2.04—2.05 (1H, m, CH_2), 2.93—2.97 (1H, m, CH_2), 3.78 (3H, s, OMe), 5.03 (1H, s, CHO), 6.86 (2H, d, *J*=8 Hz, ArH), 7.19—7.22 (1H, m, ArH), 7.30—7.33 (2H, m, ArH), 7.42—7.44 (4H, m, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 25.75 (t), 25.80 (t), 25.97 (t), 32.95 (t), 33.61 (t), 38.09 (d), 44.24 (d), 55.26 (q), 71.10 (s), 97.26 (s), 113.98 (d×2), 126.14 (d×2), 126.39 (d), 128.94 (d×2), 129.16 (d×2), 130.10 (s), 132.97 (s), 159.10 (s); MS *m/z*: 368 (M^+). *Anal.* Calcd for $C_{22}H_{24}OS_2$: C, 71.70; H, 6.56. Found: C, 71.64; H, 6.57.

2-[1-(1-Decylsulfanyl)-3-(phenylsulfanyl)prop-2-ynyl]thiophene (8ba) A brown oil, IR (KBr, cm^{-1}) *v*: 3429, 3066, 2924, 2854, 2360, 2183, 1712, 1674, 1581, 1469, 1362, 1273, 1227, 1173, 1080, 1026, 845, 737, 698, 490; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.88 (3H, t, *J*=7 Hz, Me), 1.23—1.34 (14H, br s, CH_2), 1.55—1.63 (2H, m, CH_2), 2.60—2.65 (1H, m, CH_2), 2.74—2.78 (1H, m, CH_2), 5.23 (1H, s, CH), 6.91—6.92 (1H, m, ArH), 7.13 (1H, d, *J*=1 Hz, ArH), 7.19—7.23 (3H, m, ArH), 7.31 (1H, t, *J*=8 Hz, ArH), 7.43 (2H, d, *J*=8 Hz, ArH); ^{13}C-NMR (125 MHz, $CDCl_3$) δ: 14.1 (q), 22.6 (t), 28.9 (t), 29.0 (t), 29.1 (t), 29.3 (t), 29.4 (t), 29.5 (t), 31.6 (t), 31.8 (t), 35.4 (d), 71.4 (s), 96.0 (s), 125.5 (d), 126.1 (d), 126.2 (d×2), 126.5 (d×2), 129.2 (d×2), 132.6 (s), 142.3 (s); MS *m/z*: 402 (M^+), 293 (M^+-SPh). *Anal.* Calcd for $C_{23}H_{30}S_2$: C, 68.60; H, 7.51. Found: C, 68.29; H, 7.43.

2-[1-(*p*-Chlorophenyl)-3-(phenylsulfanyl)prop-2-ynyl]thiophene (8bb) A brown oil, IR (KBr, cm^{-1}) *v*: 3073, 3060, 2923, 2852, 2182, 1657, 1581, 1573, 1475, 1441, 1389, 1265, 1230, 1173, 1093, 1024, 1013, 852, 822, 739, 703, 688, 500; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 5.45 (1H, s, CH), 6.88 (1H, dd, *J*=3, 6 Hz, ArH), 6.69 (1H, d, *J*=3 Hz, ArH), 7.20—7.24 (4H, m, ArH), 7.24—7.33 (4H, m, ArH), 7.33—7.35 (2H, m, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 40.3 (d), 73.3 (s), 95.2 (s), 125.9 (d), 126.4 (d×2), 126.6 (d), 126.7 (d), 126.8 (d), 128.9 (d×2), 129.2 (d×2), 130.9 (s), 132.1 (s), 135.2 (s), 135.9 (d×2), 140.9 (s); MS *m/z*: 372 (M^+), 263 (M^+-SPh), 229 ($M^+-C_6H_4SCl$). *Anal.* Calcd for $C_{19}H_{13}S_3Cl$: C, 61.19; H, 3.51. Found: C, 60.85; H, 3.61.

1-[1-(2-Naphthylsulfanyl)-3-(phenylsulfanyl)prop-2-ynyl]naphthalene (8ca) Yellow crystals, mp 100—105 °C, IR (KBr cm^{-1}) *v*: 3445, 3054, 1625, 1582, 1510, 1500, 1478, 1441, 1395, 1132, 1072, 1023, 944, 857, 814, 800, 777, 740, 687, 476; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 6.01 (1H, s, CH), 7.04—7.08 (3H, m, ArH), 7.20 (2H, d, *J*=8 Hz, ArH), 7.32 (1H, t, *J*=8 Hz, ArH), 7.41—7.51 (4H, m, ArH), 7.57 (1H, t, *J*=8 Hz, ArH), 7.63—7.70 (3H, m, ArH), 7.77 (2H, d, *J*=8 Hz, ArH), 7.86 (1H, d, *J*=8 Hz, ArH), 7.94 (1H, s, ArH), 8.32 (1H, d, *J*=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 42.2 (d), 73.1 (s), 96.7 (s), 123.4 (d), 125.1 (d), 125.9 (d), 126.1 (dx3), 126.3 (d), 126.4 (d×2), 126.5 (d), 126.8 (d), 127.6 (d), 127.8 (d), 128.2 (d), 129.0 (d), 129.0 (d×2), 129.1 (s), 130.7 (d), 131.0 (s), 132.3 (s), 132.4 (s), 132.8 (s), 133.2 (d), 133.5 (s), 134.0 (s); MS *m/z*: 432 (M^+), 323 (M^+-SPh), 273 ($M^+-C_{10}H_7S$). *Anal.* Calcd for $C_{29}H_{20}S_2$: C, 80.52; H, 4.66. Found: C, 80.43; H, 4.64.

5-[1-(2-Naphthylsulfanyl)-3-(phenylsulfanyl)prop-2-ynyl]-1,3-benzodioxole (8ga) Pale orange crystals, mp 105—106 °C, IR (KBr, cm^{-1}) *v*: 3651, 3054, 2894, 2777, 1582, 1501, 1487, 1442, 1364, 1249, 1132, 1102, 1039, 941, 927, 858, 814, 781, 740, 688, 477; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 5.29 (1H, s, CH), 5.29 (2H, s, OCH_2O), 6.70 (1H, d, *J*=8 Hz, ArH), 6.88 (1H, dt, *J*=1, 8 Hz, ArH), 7.02—7.09 (4H, m, ArH), 7.15—7.16 (2H, m, ArH), 7.43—7.49 (3H, m, ArH), 7.50—7.73 (2H, m, ArH), 7.78 (1H, d, *J*=8 Hz, ArH), 7.95 (1H, br s, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 44.7 (d), 72.9 (s), 96.3 (s), 101,1 (t), 108.0 (d), 108.5 (d), 121.6 (d), 126.1 (d×2), 126.3 (d), 126.4 (d), 126.5 (d), 127.6 (d), 127.7 (d), 128.3 (d), 129.0 (d×2), 130.5 (d), 130.9 (s), 131.0 (s), 132.3 (s), 132.8 (s), 133.0 (d), 133.4 (s), 147.4 (s), 147.8 (s); MS *m/z*: 426 (M^+), 317 (M^+-SPh), 267 ($M^+-C_{10}H_7S$); high resolution mass Calcd for $C_{26}H_{18}O_2S_2$: 426.0748. Found: 426.0748.

5-[1-(1-Decylsulfanyl)-3-(phenylsulfanyl)prop-2-ynyl]-1,3-benzodioxole (8gb) A yellow oil, IR (KBr, cm^{-1}) *v*: 3060, 2922, 2852, 2190, 1859, 1729, 1610, 1583, 1503, 1488, 1442, 1365, 1247, 1181, 1156, 1123, 1101, 1040, 941, 862, 815, 783, 738, 687; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.88 (3H, t, *J*=8 Hz, Me), 1.24—1.36 (14H, br s, CH_2), 1.56—1.64 (2H, m,

CH_2), 2.56—2.61 (1H, m, CH_2), 2.71—2.75 (1H, m, CH_2), 4.91 (1H, s, CH), 5.95 (2H, s, CH_2), 6.76 (1H, d, J=8 Hz, ArH), 6.96 (1H, dd, J=2, 8 Hz, ArH), 7.03 (1H, d, J=2 Hz, ArH), 7.20—7.23 (1H, m, ArH), 7.31—7.34 (2H, m, ArH), 7.42 (2H, d, J=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 14.1 (q), 22.6 (t), 28.9 (t), 29.0 (t), 29.1 (t), 29.2 (t), 29.4 (t), 29.5 (t), 31.8 (t×2), 39.8 (d), 71.6 (s), 96.6 (s), 101.1 (t), 107.9 (d), 108.3 (d), 121.1 (d), 126.1 (d×2), 126.4 (d), 129.1 (d×2), 131.9 (s), 132.8 (s), 147.2 (s), 147.8 (s); MS *m*/*z*: 440 (M^+); high resolution mass Calcd for $C_{26}H_{32}O_2S_2$: 440.1843, Found *m*/*z* 440.1783.

5-[1-(*p*-Chlorophenyl)-3-(phenylsulfanyl)prop-2-ynyl]-1,3-benzodioxole (8gc) Orange crystals, mp 58 °C, IR (KBr, cm^{-1}) ν: 3434, 3060, 2893, 2777, 1609, 1581, 1502, 1487, 1476, 1442, 1389, 1365, 1249, 1181, 1093, 1039, 939, 928, 861, 816, 781, 740, 688; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 5.14 (1H, s, CH), 5.93 (2H, s, CH_2), 6.70 (1H, d, J=8 Hz, ArH), 6.82 (1H, d, J=8 Hz, ArH), 6.96 (1H, br s, ArH), 7.19—7.28 (7H, m, ArH), 7.33 (2H, d, J=7 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 44.9 (d), 73.4 (s), 95.8 (s), 101.3 (t), 108.0 (d), 108.4 (d), 121.6 (d), 126.3 (d×2), 126.6 (d), 128.9 (d×2), 129.2 (d×2), 130.8 (s), 131.6 (s), 132.3 (s), 134.8 (s), 135.5 (d×2), 147.5 (s), 147.8 (s); MS *m*/*z*: 410 (M^+), 301 (M^+-SPh), 267 ($M^+-C_6H_4SCl$). *Anal.* Calcd for $C_{22}H_{15}O_2S_2Cl$: C, 64.30; H, 3.68. Found: C, 64.69; H, 3.81.

1-[1′-(Decylsulfanyl)-3′-phenylprop-2-ynyl]benzene (9ca) A pale yellow oil, IR (KBr, cm^{-1}) ν: 3081, 3061, 3030, 2953, 2925, 2853, 1599, 1491, 1454, 1377, 1298, 1277, 1070, 1029, 913, 825, 756, 692, 618; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.88 (3H, t, J=7 Hz, Me), 1.24—1.38 (14H, br s, CH_2), 1.57—1.68 (2H, m, CH_2), 2.60—2.64 (1H, m, CH_2), 2.77—2.81 (1H, m, CH_2), 4.99 (1H, s, CH), 7.27—7.35 (6H, m, ArH), 7.47—7.49 (2H, m, ArH), 7.57 (2H, d, J=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 14.1 (q), 22.7 (t), 29.0 (t), 29.1 (t), 29.2 (t), 29.3 (t), 29.5 (t×2), 31.8 (t), 31.9 (t), 39.3 (d), 85.7 (s), 87.4 (s), 123.0 (s), 127.6 (d), 127.9 (d×2), 128.2 (d×3), 128.5 (d×2), 131.7 (d×2), 138.6 (s); MS *m*/*z*: 364 (M^+), 223 ($M^+-C_{10}H_{21}$), 191 ($M^+-C_{10}H_{21}S$). *Anal.* Calcd for $C_{25}H_{32}S$: C, 82.36; H, 8.85. Found: C, 82.24; H, 8.81.

[1-(2-Naphthylsulfanyl)-3-phenylprop-2-ynyl]benzene (9cb) A pale yellow oil, IR (KBr, cm^{-1}) ν: 3512, 3055, 1598, 1490, 1454, 1132, 1028, 858, 814, 756, 694, 634, 529, 477, 448, 433, 422, 410; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 5.29 (1H, s, CH), 7.20—7.26 (6H, m, ArH), 7.32 (2H, s, ArH), 7.40 (2H, d, J=4 Hz, ArH), 7.45 (2H, d, J=7 Hz, ArH), 7.50—7.51 (1H, m, ArH), 7.68 (2H, br s, ArH), 7.73 (1H, d, J=5 Hz, ArH), 7.95 (1H, s, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 44.3 (d), 87.0 (s), 87.4 (s), 122.7 (s), 126.3 (d), 126.5 (d), 127.6 (d), 127.7 (d), 127.8 (d), 128.0 (d×3), 128.1 (d×2), 128.2 (d), 128.4 (d×2), 130.7 (s), 131.1 (d), 131.5 (d×2), 132.8 (s), 133.4 (s), 133.7 (d), 137.9 (s); MS *m*/*z*: 350 (M^+), 273 (M^+-Ph), 191 ($M^+-C_{10}H_7S$); high resolution mass Calcd for $C_{25}H_{18}S$: 350.1129, Found *m*/*z* 350.1138.

1-[1′-(*p*-Chlorophenylsulfanyl)-3′-phenylprop-2-ynyl]benzene (9cc) A pale yellow oil, IR (KBr, cm^{-1}) ν: 3060, 3031, 1713, 1598, 1573, 1490, 1475, 1454, 1442, 1388, 1093, 1013, 915, 828, 817, 757, 693, 666, 621, 530, 499; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 5.19 (1H, s, CH), 7.23 (2H, d, J=8 Hz, ArH), 7.27—7.33 (6H, m, ArH), 7.35—7.39 (4H, m, ArH), 7.41 (2H, d, J=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 44.3 (d), 86.9 (s), 87.2 (s), 122.7 (s), 127.9 (d), 128.0 (d×2), 128.3 (d×2), 128.4 (d), 128.5 (d×2), 128.8 (d×2), 131.5 (s), 131.6 (d×2), 134.9 (s), 136.0 (d×2), 137.7 (s); MS *m*/*z*: 334 (M^+), 191 ($M^+-C_6H_4SCl$). *Anal.* Calcd for $C_{21}H_{15}ClS$: C, 75.32; H, 4.52. Found: C, 75.11; H, 4.61.

1-(Decylsulfanyl)hept-2-ynylbenzene (9da) A yellow oil, IR (KBr, cm^{-1}) ν: 3428, 3029, 2956, 2926, 2854, 2359, 1694, 1600, 1494, 1465, 1455, 1378, 1260, 1073, 1030, 914, 758, 698, 616, 411; ^{1}H-NMR (600 MHz, $CDCl_3$) δ: 0.88 (3H, t, J=8 Hz, Me), 0.92 (3H, t, J=8 Hz, Me), 1.25 (14H, br s, CH_2), 1.42—1.47 (2H, m, CH_2), 1.52—1.67 (4H, m, CH_2), 2.29 (2H, dt, J=2, 8 Hz, CH_2), 2.51—2.56 (1H, m, CH_2), 2.67—2.71 (1H, m, CH_2), 4.75 (1H, s, CH), 7.22—7.25 (1H, m, ArH), 7.30—7.35 (2H, m, ArH), 7.48 (2H, d, J=8 Hz, ArH); ^{13}C-NMR (150 MHz, $CDCl_3$) δ: 13.5 (q), 14.0 (q), 18.6 (t), 21.9 (t), 22.6 (t), 28.9 (t), 29.0 (t), 29.1 (t), 29.2 (t), 29.4 (t), 29.5 (t), 30.8 (t), 31.6 (t), 31.8 (t), 39.0 (d), 77.8 (s), 86.2 (s), 127.3 (d), 127.7 (d×2), 128.3 (d×2), 139.2 (s); MS *m*/*z*: 344 (M^+), 171 ($M^+-C_{10}H_{21}S$); high resolution mass Calcd for $C_{23}H_{36}S$: 344.2537, Found *m*/*z* 344.2284.

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