Etherification of Diarylmethanols and 1-Phenylalkan-1-ols Over Platinum on Carbon

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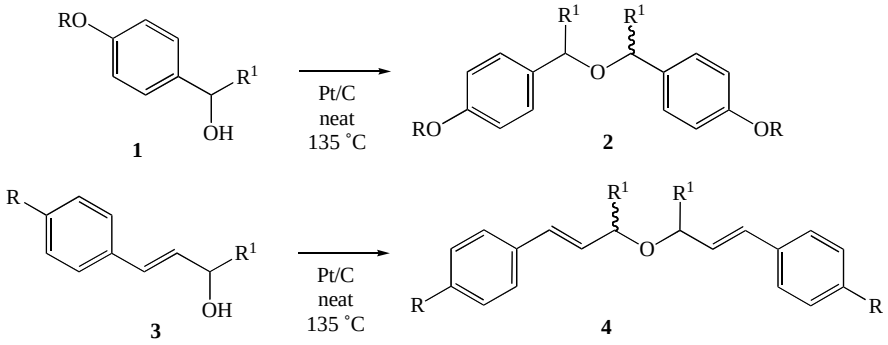
Abstract: In the presence of platinum on carbon (Pt/C), diarylmethanols and donor substituted 1-phenylalkan-1-ols undergo cross-etherification with primary and secondary alcohols.

Keywords: Etherification, platinum on carbon, diarylmethanols, 1-phenylalkan-1-ols.

INTRODUCTION

The preparation of ethers is a standard reaction in organic chemistry. Many of the syntheses of non-symmetric ethers utilise alcohols as only one of the components of the reaction, where the other reactant may be a halide [1], an alkene [2], a triflate [3], tosylate [4] or otherwise activated ester [5]. Although, there are many examples of *intramolecular* reactions of non-symmetric diols leading to non-symmetric ethers [6], only some preparative methods of intermolecular reactions of two alcohols to non-symmetric ethers are known [7]. Many of the latter approaches make use of the Lewis acidity of metal salts such as, zinc chloride [7e], magnesium perchlorate [7d] or zirconium tetrachloride [7g] or of halogens such as of iodine [7h,i]. Others utilise an *in situ* activation of one alcohol component under the conditions of a modified *Mitsunobu* condensation [7f]. Needless to say that it is necessary in these intermolecular reactions to suppress the homo-etherification in order that the cross-etherification between the two different alcohols becomes the main reaction.

In our quest for a cross-etherification process that does not necessitate added solvents, overly high temperatures or protracted purification procedures involving metal salt wastes, the author turned to the use of solid supported metals as catalysts in etherifications. Recently, the author reported that 4-alkoxyphenylalkyl carbinols (**1**) and cinnamyl alkyl carbinols (**3**) dimerise in the presence of Pt/C at 135 ºC to form ethers **2** and **4**, respectively (Scheme **1**) [8]. Also, (*E*)-1,3-diarylprop-2-en-1-ols were found to react with a number of primary and secondary alcohols [8b]. In the following, further studies on the cross-etherification of (*E*)-1,3-diarylprop-2-en-1-ols are discussed. Furthermore, it is shown that this Pt/C catalysed cross-etherification can also be applied to the reaction of diarylmethanols as well as to the donor-substituted phenylalkanols with primary and secondary alcohols.



Scheme 1. Etherification of 4-alkoxyphenyl alkyl carbinols and cinnamyl alkyl carbinols in the presence of Pt/C [8].

RESULTS AND DISCUSSION

When diarylmethanols **5** are heated at 130 ºC over a long period of time, a slow dimerisation to the corresponding bis(diarylmethyl) ethers **6** occurs, a process which does not complete even after 24h. When a catalytic amount of Pt/C is added to neat diarylmethanols **5** at 130 ºC, a rapid etherification ensues which gives products **6** in high yields (Scheme **2**). The same reaction can be found for 1,3-bis(4-biphenyl)-prop-2-en-1-ol (**7**), albeit already at lower temperatures (100 ºC).

An addition of 10 eq. of a second alcohol, either of a primary or a secondary alcohol, completely suppresses the

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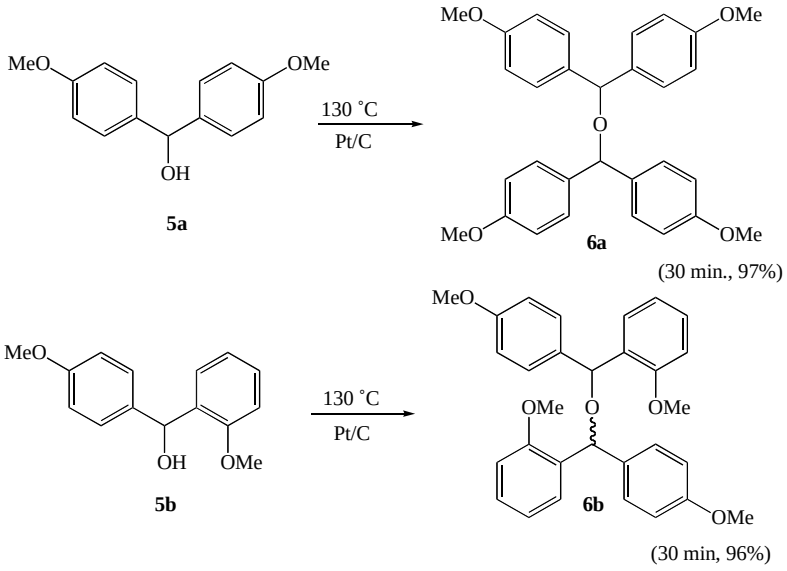
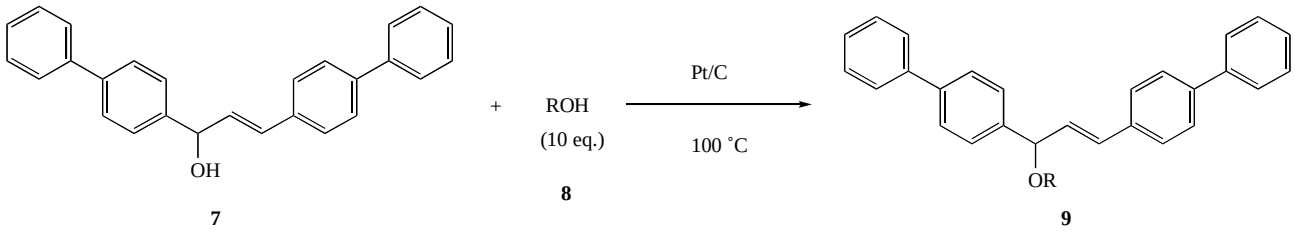
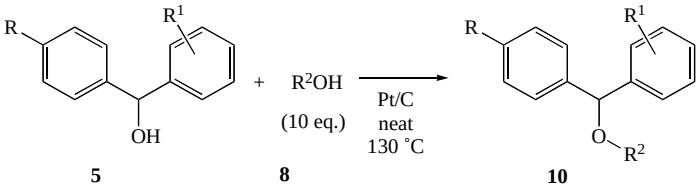


Scheme 2. Formation of ether dimers from dimethoxyphenyl carbinols over Pt/C.



Scheme 3. Etherification of 1,3-bis(4-biphenyl)-prop-2-en-1-ol over Pt/C.



Scheme 4.

Table 1. Etherification of 1,3-bis(4-biphenyl)-prop-2-en-1-ol over Pt/C

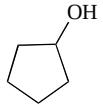
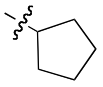
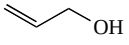
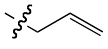
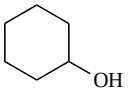
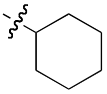
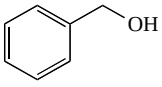
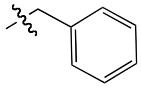
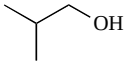
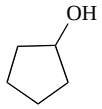
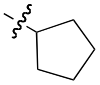
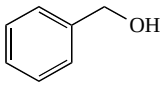
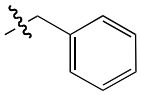
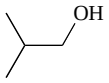
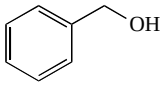
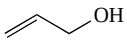
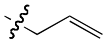
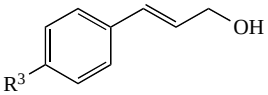
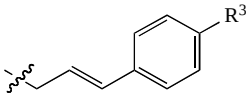
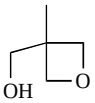
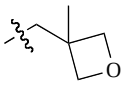
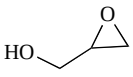
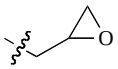
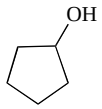
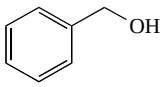
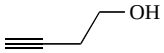
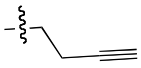
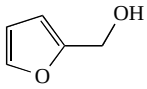
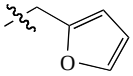
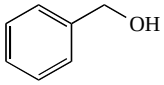
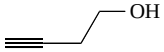
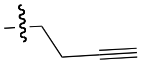
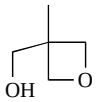
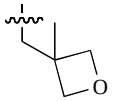
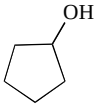
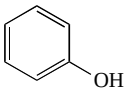
Alcohol	Product (R =)	Alcohol	Product (R =)
 **8a**	 (90 min., 85%) **9a**	 **8d**	 (30 min., 73%) **9d**
$CH_3(CH_2)_5CH_2OH$ **8b**	C_7H_{15} (30 min., 93%) **9b**	 **8e**	 (2h, 86%) **9e**
 **8c**	 (30 min., 90%) **9c**	 **8f**	$CH_2CH(CH_3)_2$ (1h, 94%) **9f**

Table 2. Etherification of Diarylcarbinols Over Pt/C

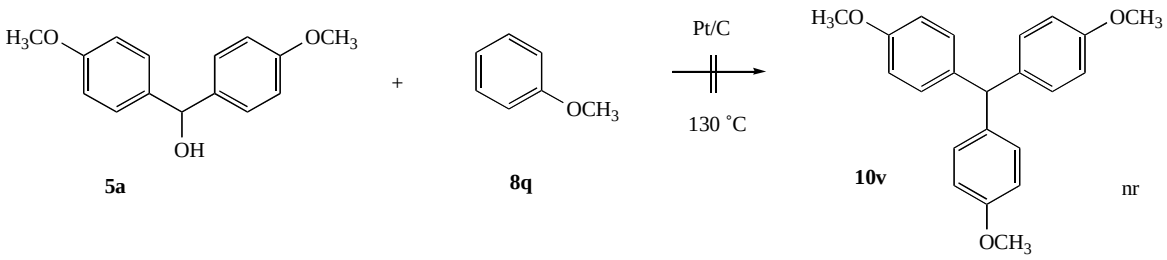
Substrate	Reactant	Product
5c: R = CH_3, R^1 = 4-CH_3	 **8a**	**10a:** R = CH_3, R^1 = 4-CH_3, (1h, 78%) R^2 = 
5c: R = CH_3, R^1 = 4-CH_3	 **8c**	**10b:** R = CH_3, R^1 = 4-CH_3, (1h, 82%) R^2 = 
5c: R = CH_3, R^1 = 4-CH_3	 **8f**	**10c:** R = CH_3, R^1 = 4-CH_3, R^2 = $CH_2CH(CH_3)_2$ (30 min., 85%)
5a: R = OCH_3, R^1 = 4-OCH_3	$CH_3(CH_2)_5CH_2OH$ **8b**	**10d:** R = OCH_3, R^1 = 4-OCH_3, R^2 = C_7H_{15} (30 min., 94%)
5a: R = OCH_3, R^1 = 4-OCH_3	 **8c**	**10e:** R = OCH_3, R^1 = 4-OCH_3, R^2 = CH_2Ph (30 min., 91%)
5a: R = OCH_3, R^1 = 4-OCH_3	 **8d**	**10f:** R = OCH_3, R^1 = 4-OCH_3, R^2 = (30 min., 89%) 
5a: R = OCH_3, R^1 = 4-OCH_3	 **8g**: R^3 = H **8h**: R^3 = OCH_3	**10g,h:** R = OCH_3, R^1 = 4-OCH_3, R^2 = **10g**: R^3 = H: (15 min., 88%) **10h**: R^3 = OCH_3: (30 min., 74%) 
5a: R = OCH_3, R^1 = 4-OCH_3	 **8i**	**10i:** R = OCH_3, R^1 = 4-OCH_3, R^2 = (30 min., 87%) 
5a: R = OCH_3, R^1 = 4-OCH_3	 **8j**	**10j:** R = OCH_3, R^1 = 4-OCH_3, R^2 = (30 min., 85%) 
5d: R = OCH_3, R^1 = H	$CH_3(CH_2)_5CH_2OH$ **8b**	**10k:** R = OCH_3, R^1 = H, R^2 = C_7H_{15} (30 min., 75%)
5d: R = OCH_3, R^1 = H	 **8a**	**10l:** R = OCH_3, R^1 = H, R^2 = (3h, 86%) 
5d: R = OCH_3, R^1 = H	 **8c**	**10m:** R = OCH_3, R^1 = H, R^2 = CH_2Ph (1h, 90%)

(Table 2). Contd…..

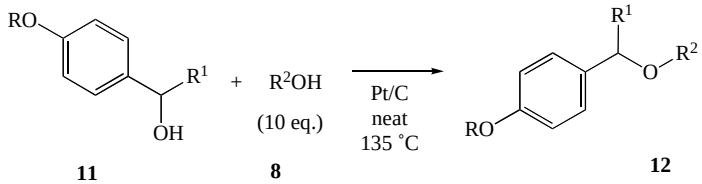
Substrate	Reactant	Product
5d: R = OCH_3, R^1 = H	 **8j**	**10n:** R = OCH_3, R^1 = H, R^2 =  (1h, 89%)
5d: R = OCH_3, R^1 = H	 **8k**	**10o:** R = OCH_3, R^1 = H, R^2 =  (1h, 86%)
5b: R = OCH_3, R^1 = 2-OCH_3	 **8c**	**10p:** R = OCH_3, R^1 = 2-OCH_3, R^2 = CH_2Ph (30 min., 90%)
5b: R = OCH_3, R^1 = 2-OCH_3	$CH_3(CH_2)_3CH_2OH$ **8l**	**10q:** R = OCH_3, R^1 = 2-OCH_3, R^2 = C_5H_{11} (30 min., 94%)
5b: R = OCH_3, R^1 = 2-OCH_3	 **8k**	**10r:** R = OCH_3, R^1 = 2-OCH_3, R^2 =  (30 min., 91%)
5b: R = OCH_3, R^1 = 2-OCH_3	 **8i**	**10s:** R = OCH_3, R^1 = 2-OCH_3, R^2 =  (30 min., 89%)
5b: R = OCH_3, R^1 = 2-OCH_3	 **8a**	**10t:** R = OCH_3, R^1 = OCH_3, R^2 =  (30 min., 81%)
5d: R = H, R^1 =H	$CH_3(CH_2)_5CH_2OH$ **8b**	**10u**: R = H, R^1 = H, R^2 = C_7H_{15} (3h, 25%)
5d: R = H, R^1 =H	 **8p**	**10v:** R = H, R^1 =H, R^2 = C_6H_5 = Ph (nr)

homo-etherification and solely products stemming from the cross-etherification can be found (Schemes **3** and **4**, Tables **1** and **2**). Again, reactions with diarylmethanols **5** are the best carried out at 130 ºC (Table **2**), while reaction with 1,3-bis(4-biphenyl)-prop-2-en-1-ol (**7**) necessitates lower temperatures (100 ºC) (Table **1**).

Reaction proceeds best with electron donor substituted **5**, such as with methyl or methoxy substituted **5**. Reaction with the parent compound **5e** proceeds more slowly. The scope of primary and secondary alcohols is large. Pt driven isomerisation of olefinic moieties does not take place. Thus, allyl alcohol (**8d**) reacts well to give the desired product **9d**. Interestingly, also acid sensitive alcohols such as oxiran-2-



Scheme 5.



11 **8** **12**

Scheme 6.

ylmethanol (**8j**) can be reacted with ease. Also, 4-methoxycinnamyl alcohol (**8h**), which itself would be expected to dimerise under the conditions, can be used successfully as reactant. Indeed, over Pt/C, 4-methoxycinnamyl alcohol (**8h**) itself undergoes slow dimerisation to bis(4-methoxycinnamyl)ether [9], which is obtained as a separable by-product. Nevertheless, in the reaction of bis(4-methoxyphenyl)methanol (**5a**) with 4-methoxycinnamyl alcohol (**8h**), the desired product **10h** can be isolated in acceptable yield. When cinnamyl alcohol (**8g**) is used and the reaction time shortened to 15 min (at 130 ºC), only the desired cross-etherification product **10g** is formed. On the other hand, phenols do not react well. Thus, when phenol (10 eq.) itself is reacted under the conditions with (4-methoxyphenyl)-phenylcarbinol (**5d**) at 130 ºC, no cross-etherification can be observed. Also, the trapping of a potential diphenylmethyl cation intermediate with electron rich arenes in a Friedel-Craft type reaction does not occur. Thus, the reaction of **5a** in the presence of 10eq. of anisole does not yield any tris(*p*-anisyl)methane (Scheme **5**), but provides exclusively ether **6a**.

Donor substituted 1-phenylalkan-1-ols **11** can be used successfully as substrates in the cross-etherication reaction, catalysed by Pt/C (Scheme **6** and Table **3**). In this case, steric congestion in the substrate such as in the case of **11c** leads to some loss of reactivity, indicating that the reactions proceed at or near to the surface of the catalyst support (see below). Here, much longer reaction times are needed (6h *vs.* normally 30 min. at 130 ºC).

The actual mechanism of the reaction has not been ascertained. Reports have appeared of the activation of oxygen containing substrates with salts of the platinum metal group, with Pt(II) complexes [10] and Pd(II) salts [11], in homogeneous reactions. There is some indication in the present case that the etherification proceeds on or near the surface of the catalyst. Thus, sterically more exacting alcohols do not react. *tert*-Butanol leads to only a small amount of product and phenylglycidol does not react, while glycidol itself reacts with ease. It can be believed that Pt (0), immobilised on the carbon support [12], functions as a weak Lewis acid catalyst. It is known that alcohols absorb on platinum [Pt(111)] with an oxygen lone-pair interaction with platinum of about 42 kJ mol^{-1} [13]. However, as the above experiments have been carried out at elevated temperatures and in the presence of oxygen, it can be known that, the catalytically active species is not Pt(0), but rather Pt in a higher oxidation state such as Pt(II). Thus, for other reactions, the authors have found that Pt, derived from $Pt(PPh_3)_4$, supports transformations in diphenyl ether at elevated temperatures in the presence of oxygen. These reactions are not supported by Pt(0) in deaerated diphenyl ether at the same temperatures, indicating that Pt(0) in aerated diphenyl ether can be oxidised to a higher oxidation state [14]. In the case of the presence of Pt(II), a reaction mechanism as detailed by M. M. Abu-Omar *et al.* and J. Muzart *et al.* [11] for Pd(II) can be taken into account. This involves a Pd-assisted S_N reaction of the alcohol, where with the catalytic system L_2PdCl_2/AgOTf an S_N1 reaction with intermediate cation formation from the diarylcarbinol substrate has been formulated [11c].

While the results reported here are from the experiments carried out with a 5 wt% Pt on carbon as supplied by Wako Co., also other commercially available samples of Pt on carbon were tested in the reactions. Of those tested, the only Pt on carbon that failed to produce any enhancement of reactivity was found to be Pt/C Degussa type F101 RA/W (wet). It could also be observed that the etherification of **5a**, a slow reaction in the absence of a catalyst, can be accelerated significantly even in the presence of platinum powder (200 mesh). The reaction of **5a** with **8c** to **10e** could also be carried out successfully with platinum powder, albeit with longer reaction times (8h, 130 ºC, 80%).

In conclusion, it could be shown that in the presence of platinum on carbon (Pt/C), diarylmethanols **5** and donor substituted 1-phenylalkan-1-ols **11** undergo cross-etherification with primary and secondary alcohols. Acid-labile alcohols such as oxiran-2-ylmethanol (**8j**) and (3-methyloxetan-3-yl) methanol (**8i**) are viable substrates for the reaction.

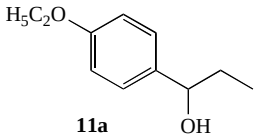
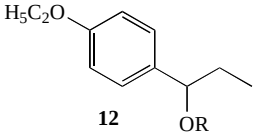
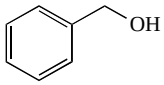
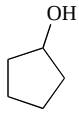
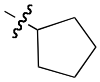
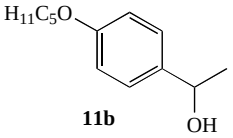
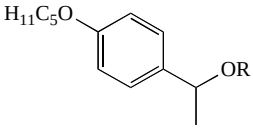
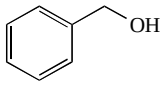
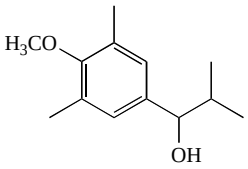
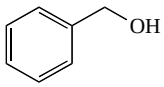
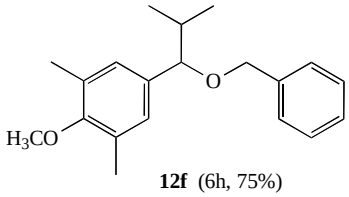
EXPERIMENTAL

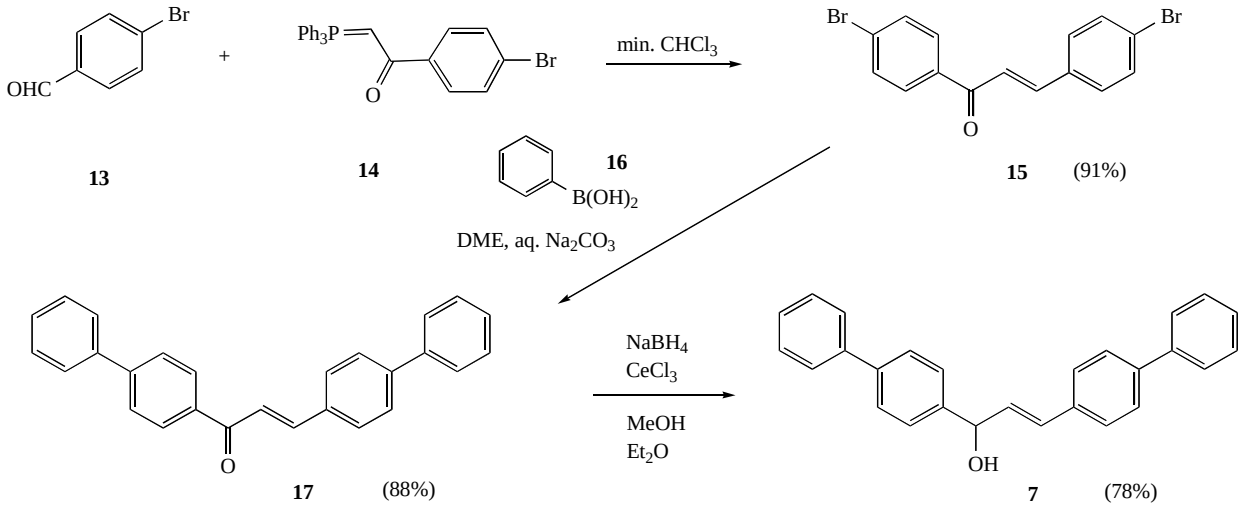
General

Melting points were measured on a Yanaco microscopic hotstage and are uncorrected. Infra-red spectra were measured with JASCO IR-700 and Nippon Denshi JIR-AQ2OM machines. ^{1}H- and ^{13}C-NMR spectra were recorded with a JEOL EX-270 spectrometer. The chemical shifts were relative to TMS (solvent $CDCl_3$, unless otherwise noted). Mass spectra were measured with a JMS-01-SG-2 spectrometer (EI, 70 eV). Column chromatography was carried out on Wakogel 300.

All diarylmethanols were prepared from the corresponding benzophenones by reduction with $NaBH_4$ in MeOH. 4,4'-Dimethoxybenophenone (TCI), 2,4'-dimethoxybenzophenone (TCI), and 4-methoxybenzophenone (4-benzoylanisole, TCI) were acquired commercially. 4-Methoxycinnamyl alcohol ($NaBH_4$, $CeCl_3$, MeOH) was obtained from 4-methoxycinnamaldehyde (TCI). 1-(4-Ethoxyphenyl)propan-1-ol and (1-4-octyloxyphenyl)ethanol were prepared by *O*-alkylation of phenol (EtI, KOH, DMSO

Table 3. Etherification of 1-Phenyl-alkan-1-ols Over Pt/C

Substrate	Reactant	Product (Reaction Time/Yield)
 **11a**	$CH_3(CH_2)_6CH_2OH$ **8m**	 **12**
11a	 **8c**	**12a:** R = C_8H_7 (30 min., 88%) **12b:** R = CH_2Ph (30 min., 86%)
11a	 **8a**	**12c:** R =  (1h, 84%)
 **11b**	$CH_3(CH_2)_6CH_2OH$ **8m**	 **12d:** R = C_8H_{17} (30 min., 87%)
11b	 **8c**	**12e:** R = CH^2Ph (30 min., 89%)
 **11c**	 **8c**	 **12f** (6h, 75%)



Scheme 7. Preparation of 1,3-bis(biphenyl)prop-2-en-1-one (**17**) and 1,3-bis(4-biphenyl)-prop-2-en-1-ol (**7**).

or $CH_3(CH_2)_6CH_2OH$, cetylammonium bromide, 20w% aq. NaOH), subsequent Friedel-Crafts acylation (acetyl chloride, propionyl chloride or isobutyroyl chloride, $AlCl_3$, 1,2-dichloroethane) and reduction with $NaBH_4$ in MeOH or in MeOH/ether. 1,3-Bis(4-biphenyl)-prop-2-en-1-ol (**7**) [see also ref. 15] was prepared by Luche reduction ($CeCl_3$,

$NaBH_4$, MeOH) [16] from 1,3-bis(4-biphenyl)-prop-2-en-1-one (**17**), which itself was synthesised from commercially available 4-bromobenzaldehyde (**13**) in two steps, namely by Wittig Olefination using the stabilised phosphorane **14** [17] to **15** followed by Suzuki-Miyaura cross coupling reaction with phenylboronic acid (**16**) (Scheme **7**).

Platinum on activated carbon (5 wt%) from Wako Pure Industries (Lot 167-13911) and platinum powder (200 mesh) from Kishida Chemicals were acquired commercially.

Procedure A

DL- and meso bis(2-anisyl-4-anisylmethyl)ether (**6b**). - A mixture of **5b** (550 mg, 2.25 mmol) and Pt/C (50 mg, 5 wt%, $12.7 \cdot 10^{-3}$ mmol Pt, 1.0 mol%) was kept at 130 ºC for 30 min. Thereafter, the cooled mixture was subjected to a filtration over silica gel (hexane/ether/$CHCl_3$ 2:1:1) to give **9b** (528 mg, 96%, *DL*/*meso* 55:45) as a colourless oil; IR (neat; A/B) ν 3000, 2950, 2834, 1611, 1516, 1462, 1439, 1339, 1246, 1169, 1113, 1040, 862, 815, 758 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 3.63 (s, 3H, OCH_3, A), 3.64 (s, 3H, OCH_3, B), 3.75 (s, 3H, OCH_3, A), 3.76 (s, 3H, OCH_3, B), 5.72 (s, 1H, A), 5.73 (s, 1H, B), 6.78 (m, 1H, A, B), 6.79 (d, 2H, ^3J 8.6 Hz, A, B); 6.96 (m, 1H, A, B), 7.24 (d, 2H, ^3J 8.6 Hz, A, B), 7.66 (m, 1H, A, B); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 55.16 (OCH_3, A, B), 55.27 (OCH_3, A, B), 73.51 (A), 73.70 (B), 110.46 (A), 110.50 (B), 113.28 (2C, A, B), 120.46 (A, B), 127.17 (B), 127.22 (A), 127.89 (A), 127.97 (B), 128.63 (B, 2C), 128.70 (A, 2C), 131.40 (A), 131.69 (B), 134.76 (B), 135.00 (A), 156.49 (A), 156.72 (B), 158.48 (B), 158.52 (A); MS (EI, 70 eV) *m/z* (%) = 470 (M^+, 2.4), 362 (18), 227 (100), 135 (33), 121 (89). HRMS Found: 470.2094. Calcd. for $C_{30}H_{30}O_5$: 470.2093.

Bis(bis[4-anisyl]methyl) ether (**6a**), prepared according to procedure A: colourless oil; IR (neat) ν 2928, 1612, 1508, 1456, 1240, 1171, 1030, 826, 751, 701 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 3.78 (s, 6H, 2 OCH_3), 5.28 (s, 2H), 6.84 (d, 2H, ^3J 8.9 Hz), 7.24 (d, 2H, ^3J 8.9 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 55.26 (4C), 78.91 (2C), 113.72 (8C), 128.45 (2C), 134.82 (4C), 158.84 (4C). MS (EI, 70 eV) *m/z* (%) = 470 (M^+, 2.0), 362 (5.0), 243 (23), 227 (100). HRMS Found: 470.2095. Calcd. for $C_{30}H_{30}O_5$: 470.2093.

1,3-Bis(4-biphenyl)-prop-2-en-1-ol (**7**). - At 0 ºC, $NaBH_4$ was added slowly to a mixture of **17** (3.8 g, 10.5 mmol) and anhydrous cerium trichloride (2.7 g, 11.1 mmol) in methanol (20 mL) and ether (10 mL); colourless solid. After the mixture was stirred for 15 min., it was concentrated *in vacuo* at ambient temperature. Thereafter, water (40 mL) was added and the ensuing mixture extracted with chloroform (2 X 30 mL). To the aqueous phase was added ammonium chloride was added and the mixture was extracted with chloroform (25 mL). The combined organic phase was dried over anhydrous $MgSO_4$, concentrated *in vacuo*, and the residue was subjected to column chromatography on silica gel (hexane/ether/$CHCl_3$ 1:1:1) to give **7** (3.0 g, 78%); mp. 142 ºC; IR (KBr) ν 3450 (bs, OH), 1486, 1406, 1074, 1004, 963, 824, 759, 694 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 2.14 (bs, 1H, OH), 5.45 (d, 1H, ^3J 5.9 Hz), 6.45 (dd, 1H, ^3J 15.4 Hz, ^3J 5.9 Hz), 6.76 (d, 1H, ^3J 15.4Hz); 7.24 - 7.62 (m, 18H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 74.93, 126.79 (2C), 126.91 (2C), 127.06 (2C), 127.10 (2C), 127.25 (2C), 127.33, 127.40 (2C), 128.77 (6C), 130.20, 131.48, 135.52, 140.57, 140.59, 140.78, 141.75; MS (EI, 70 eV) *m/z* (%) = 362 (M^+, 22), 344 (100), 181 (42). HRMS Found: 362.1672. Calcd. for $C_{27}H_{22}O$: 362.1671. Calcd. for $C_{27}H_{22}O$: C, 89.47; H, 6.12%. Found: C, 89.20; H, 6.10%.

Procedure B

1,3-Bis(4-biphenyl)-prop-2-en-1-yl heptyl ether (**9b**). - A mixture of **7** (300 mg, 0.83 mmol), *n*-heptanol (960 mg, 8.3 mmol, 10 eq.) and Pt/C (20 mg, 5 wt%, $5.1 \cdot 10^{-3}$ mmol Pt, 0.6 mol%) is kept at 100 ºC for 30 min. Thereafter, the cooled mixture is subjected to a filtration over silica gel (hexane/ether/$CHCl_3$ 5:1:1) to give **9b** (355 mg, 93%) as a colourless oil; IR (neat) ν 3028, 2928, 2854, 1601, 1510, 1486, 1459. 1450, 1406, 1378, 1298, 1032, 1006, 968, 839, 762, 697 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 0.88 (t, 3H, ^3J 6.5 Hz), 1.28 (m, 8H), 1.67 (m, 2H), 3.51 (m, 2H, $OC\underline{H}_2$), 4.96 (d, 1H, ^3J 7.0 Hz), 6.37 (dd, 1H, ^3J 16.7 Hz, ^3J 7.0 Hz), 6.67 (d, 1H, ^3J 16.7 Hz), 7.30 - 7.67 (m, 18H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 14.08, 22.62, 26.22, 29.17, 29.95, 31.85, 68.92, 81.41, 126.92 (2C), 127.03 (2C), 127.11 (2C), 127.20 (2C), 127.26 (5C), 127.28, 128.74 (2C), 128.76 (2C), 130.69, 130.78, 135.73, 140.43, 140,54, 140.67 (2C), 140.96; MS (EI, 70 eV) *m/z* (%) = 460 (M^+, 13), 346 (39), 281 (78), 198 (58), 181 (100), 149 (75). HRMS Found: 460.2762. Calcd. for $C_{34}H_{36}O$: 460.2766. Calcd. for $C_{34}H_{36}O \cdot 0.3H_2O$: C, 87.62; H, 7.91%. Found: C, 87.70; H, 7.83%.

9a - 9f were Prepared According to Procedure B

1,3-Bis(4-biphenyl)-prop-2-en-1-yl cyclopentyl ether (**9a**); colourless oil; IR (neat) ν 3024, 2952, 1601, 1514, 1485, 1450, 1404, 1174, 1092, 970, 838, 758, 696 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 1.75 - 1.79 (m, 8H), 4.11 (m, 1H), 5.05 (d, 1H, ^3J 7.0 Hz), 6.37 (dd, 1H, ^3J 16.5 Hz, ^3J 7.0 Hz), 6.67 (d, 1H, ^3J 16.5 Hz), 7.29 - 7.61 (m, 18H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 23.59, 23.65, 32.53, 32.59, 78.88, 80.09, 126.91 (2C), 127.02 (2C), 127.10 (2C), 127.19 (5C), 127.27, 127.32 (2C), 128.73 (2C), 128.76 (2C), 130.46, 131.16, 135.80, 140.37, 140.41, 140.69, 140.99, 141.01; MS (EI, 70 eV) *m/z* (%) = 430 (M^+, 12) 362 (52), 345 (100), 209 (25), 181 (94), 167 (78), 154 (48), 136 (30). HRMS Found: 430.2289. Calcd. for $C_{32}H_{30}O$: 430.2297.

1,3-Bis(4-biphenyl)-prop-2-en-1-yl benzyl ether (**9c**); colourless solid; mp. 126 ºC; ^1H NMR (270 MHz, $CDCl_3$) δ 4.63 (s, 2H, $OC\underline{H}_2Ph$), 5.08 (d, 1H, ^3J 7.0 Hz), 6.41 (dd, 1H, ^3J 15.9 Hz, ^3J 7.0 Hz), 6.70 (d, 1H, ^3J 15.9 Hz), 7.30 - 7.63 (m, 23H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 70.21, 81.33, 126.93 (2C), 127.07 (2C), 127.12 (2C), 127.24 (2C), 127.29, 127.35 (2C), 127.44 (2C), 127.59, 127.77 (2C), 128.43 (2C), 128.77 (5C), 130.27, 131.20, 135.63, 138.41, 140.17, 140.55, 140.67, 140.72, 140.91, 147.71; MS (FAB, 3-nitrobenzyl alcohol) m/z (%) = 452 (M^+, 0.7), 345 (4.1). HRMS Found: 452.2133. Calcd. for $C_{34}H_{28}O$: 452.2140. Calcd. for $C_{34}H_{28}O \cdot 0.1H_2O$: C, 89.87; H, 6.25%. Found: C, 89.70; H, 6.19%.

1,3-Bis(4-biphenyl)-prop-2-en-1-yl allyl ether (**9d**); colourless oil; ^1H NMR (270 MHz, $CDCl_3$) δ 4.09 (m, 2H), 5.05 (d, 1H, ^3J 6.8 Hz), 5.22 (m, 1H), 5.34 (m, 1H), 6.00 (m,

1H), 6.38 (dd, 1H, ^3J 15.9 Hz, ^3J 7.0 Hz), 6.69 (d, 1H, ^3J 15.9 Hz), 7.29 - 7.63 (m, 18H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 69.35, 81.54, 117.03, 126.93 (2C), 127.05 (2C), 127.11 (2C), 127.22 (2C), 127.27, 127.32 (4C), 127.35 (2C), 128.76 (4C), 130.27, 131.06, 134.83, 135.62, 140.19, 140.53, 140.66, 140.91; IR (neat) ν 3026, 2924, 2852, 1643, 1600, 1511, 1486, 1450, 1407, 1299, 1178, 1074, 1008, 969, 838, 761, 696 cm^{-1}; MS (FAB, 3-nitrobenzyl alcohol) *m/z* (%) = 402 (M^+, 5.5), 345 (57), 181 (100). HRMS Found: 402.1986. Calcd. for $C_{30}H_{26}O$: 402.1984

1,3-Bis(4-biphenyl)-prop-2-en-1yl cyclohexyl ether (**9e**); colourless oil; IR (neat) ν 3026, 2928, 2854, 1650, 1486, 1072, 969, 836, 761, 696 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.20 - 1.50 (m, 4H), 1.76 (m, 4H), 1.96 (m, 2H), 3.46 (m, 1H), 5.16 (d, 1H, ^3J 6.8 Hz), 6.37 (dd, 1H, ^3J 16.2 Hz, ^3J 6.8 Hz), 6.65 (d, 1H, ^3J 16.2 Hz), 7.27 - 7.60 (m, 18H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 24.21 (2C), 25.85, 32.56 (2C), 74.87, 78.91, 126.89 (2C), 127.01 (2C), 127.07 (2C), 127.16, 127.18 (4C), 127.26 (3C), 128.71 (2C), 128.74 (2C), 130.25, 131.37, 135.81, 140.34 (2C), 140.67, 140.96, 141.22; MS (FAB, 3-nitrobenzyl alcohol) *m/z* (%) = 444 (M^+, 8.4), 362 (46), 345 (100). HRMS Found: 444.2455. Calcd. for $C_{33}H_{32}O$: 444.2453.

1,3-Bis(4-biphenyl)-prop-2-en-1yl isopropyl ether (**9f**); colourless oil; IR (neat) ν3028, 2956, 2868, 1600, 1486, 1404, 1076, 1007, 968, 838, 761, 696 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 0.97 (d, 6H, ^3J 6.5 Hz, 2 CH_3), 1.97 (ddh, ^3J 6.8 Hz, ^3J 6.8 Hz, ^3J 6.5 Hz), 3.23 (dd, 1H, ^2J 9.5 Hz, ^3J 6.8 Hz), 3.34 (dd, 1H, ^2J 9.5 Hz, ^3J 6.8 Hz), 4.95 (d, ^3J 6.8 Hz), 6.36 (dd, 1H, ^3J 15.1 Hz, ^3J 6.8 Hz), 6.67 (d, 1H, ^3J 15.1 Hz), 7.30 - 7.61 (m, 18H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 19.56 (2C), 31.58, 75.67, 82.44, 126.93 (2C), 127.03 (2C), 127.11 (2C), 127.22 (8C), 127.29, 128.74 (2C), 128.76 (2C), 130.60, 130.87, 140.43, 140.47, 140.69, 140.74, 140.96; MS (FAB, 3-nitrobenzyl alcohol) *m/z* (%) = 418 (M^+, 22), 361 (20), 345 (53), 239 (37), 181 (100), 167 (51). HRMS Found: 418.2298. Calcd. for $C_{31}H_{30}O$: 418.2297.

10a - 10u and 12a - 12f were Prepared According to Procedure B, but the Reactions were Carried Out at 130 ºC

Bis(4-tolyl)methyl cyclopentyl ether (**10a**); colourless oil; IR (neat) ν 2924, 1515, 1453, 1260, 1087, 804, 699 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.47 (m, 2H), 1.73 (m, 6H), 2.31 (s, 6H, 2 CH_3), 3.96 (m, 1H), 5.35 (s, 1H), 7.10 (d, 4H, ^3J 8.1 Hz), 7.20 (d, 4H, ^3J 8.1 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 21.10 (2C), 23.60 (2C), 32.41 (2C), 78.87, 80.74, 127.09 (4C), 128.94 (4C), 136.71 (2C), 140.16 (2C); MS (EI, 70 eV) *m/z* (%) = 280 (M^+, 41), 105 (100), 180 (32), 165 (48), 119 (25). HRMS Found: 280.1832. Calcd. for $C_{20}H_{24}O$: 280.1827.

Bis(4-tolyl)methyl benzyl ether (**10b**); colourless oil; IR (neat) ν 3020, 2922, 2856, 1511, 1451, 1300, 1177, 1068, 813, 764, 733, 636 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 2.32 (s, 6H, 2 CH_3), 4.52 (s, 2H), 5.38 (s, 1H), 7.12 (d, 4H, ^3J 8.1 Hz), 7.25 (d, 4H, ^3J 8.1 Hz), 7.26 - 7.37 (m, 5H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 21.32 (2C), 70.53, 82.38, 127.23 (4C), 127.64, 127.88 (2C), 128.52 (2C), 129.26 (4C), 137.21 (2C), 138.81, 139.61 (2C); MS (EI, 70 eV) *m/z* (%) = 302 (M^+, 1.9), 211 (100), 195 (97), 181 (25), 165 (33), 119 (51), 91 (85). HRMS Found: 302.1665. Calcd. for $C_{22}H_{22}O$: 302.1671.

Bis(4-tolyl)methyl isobutyl ether (**10c**); colourless oil; IR (neat) ν 2929, 1512, 1466, 1177, 1088, 1020, 965, 814, 764, 697 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 0.93 (d, 6H, ^3J 6.5 Hz, 2 CH_3), 1.91 (dsept, 1H, ^3J 6.8 Hz, ^3J 6.5 Hz), 2.31 (s, 6H, 2 CH_3), 3.19 (d, 2H, ^3J 6.8 Hz), 5.25 (s, 1H), 7.10 (d, 4H, ^3J 7.8 Hz), 7.23 (d, 4H, ^3J 7.8 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 19.52 (2C), 21.08 (2C), 28.71, 75.83, 83.28, 126.83 (4C), 128.94 (4C), 136.73 (2C), 139.99 (2C); MS (EI, 70 eV) *m/z* (%) = 268 (M^+, 68), 195 (100), 165 (44), 119 (47). HRMS Found: 268.1828. Calcd. for $C_{19}H_{24}O$: 268.1827.

Bis(4-anisyl)methyl heptyl ether (**10d**); colourless oil; IR (neat) ν 2966, 2930, 2854, 1610, 1585, 1511, 1463, 1340, 1302, 1250, 1171, 1093, 1076, 827, 757 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 0.88 (m, 3H), 1.27 (m, 8H), 1.61 (m, 2H), 3.40 (t, 2H, ^3J 6.5 Hz), 3.78 (s, 6H, 2 OCH_3), 5.25 (s, 1H), 6.84 (d, 4H, ^3J 8.6 Hz), 7.23 (d, 4H, ^3J 8.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 14.80, 23.34, 26.95, 29.86, 30.62, 32.55, 55.94 (2C, OCH_3), 69.74, 83.37, 114.39 (4C), 128.85 (4C), 135.86 (2C), 159.51 (2C); MS (EI, 70 eV) m/z (%) = 342 (M^+, 51), 227 (100). HRMS Found: 342.2190. Calcd. for $C_{22}H_{30}O_3$: 342.2195. Calcd. for $C_{22}H_{30}O_3 \cdot H_2O$: C, 73.15; H, 8.99. Found: C, 73.35; H, 8.42%.

Bis(4-anisyl)methyl benzyl ether (**10e**); colourless oil; IR (neat) ν2924, 1624, 1505, 1455, 1245, 1172, 1031, 825, 736, 695 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 3.78 (s, 6H, 2 OCH_3), 4.51 (s, 2H), 5.36 (s, 1H), 6.85 (d, 4H, ^3J 8.6 Hz), 7.28 (d, 4H, ^3J 8.6 Hz), ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 55.24 (2C, 2 OCH_3), 70.25, 81.55, 113.75 (4C), 127.45, 127.70, 128.33 (4C), 134.58 (2C), 138.58, 158.91 (2C); MS (EI, 70 eV) *m/z* (%) = 334 (M^+, 14), 243 (31), 227 (100). HRMS Found: 334.1569. Calcd. for $C_{22}H_{22}O_3$: 334.1569. Calcd. for $C_{22}H_{22}O_3 \cdot 0.1H_2O$: C, 78.46; H, 6.69. Found: C, 78.41; H, 6.61%.

Bis(4-anisyl)methyl allyl ether (**10f**); colourless oil; IR (neat) ν2932, 1611, 1507, 1455, 1244, 1169, 1034, 926, 815, 772 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 3.78 (s, 6H, 2 OCH_3), 3.97 (m, 2H), 5.15 - 5.32 (m, 2H), 5.34 (s, 1H), 5.93 (m, 1H), 6.85 (d, 4H, ^3J 8.6 Hz), 7.25 (d, 4H, ^3J 8.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 55.23 (2C, 2 OCH_3), 69.46, 81.74, 113.73 (4C), 116.71, 128.20 (4C), 134.67 (2C), 134.97, 158.87 (2C); MS EI, 70 eV) *m/z* (%) = 284 (M^+, 25), 227 (100), 135 (38). HRMS Found: 284.1413. Calcd. for $C_{18}H_{20}O_3$: 284.1412.

Bis(4-anisyl)methyl cinnamyl ether (**10g**); colourless oil; ^{1}H NMR (270 MHz, $CDCl_3$) δ 3.78 (s, 6H, 2 OCH_3), 4.15 (d, 2H, ^3J 5.7 Hz), 5.40 (s, 1H), 6.33 (dt, 1H, ^3J 15.9 Hz, ^3J 5.7 Hz), 6.59 (d, 1H, ^3J 15.9 Hz), 6.86 (d, 4H, ^3J 8.9 Hz), 7.22 - 7.39 (m, 9H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 55.25, 66.13, 81.75, 113.76 (4C), 126.38, 126.46 (2C), 127.57, 128.25 (4C), 128.52 (2C), 132.14, 134.59 (2C), 136.83, 158.89 (2C); MS (EI, 70 eV) *m/z* (%) 360 (M^+, 3.6), 242 (49), 224 (100). HRMS Found: 360.1725. Calcd. for $C_{24}H_{24}O_3$: 360.1725.

Bis(4-anisyl)methyl 4-methoxycinnamyl ether (**10h**); colourless oil; ^{1}H NMR (270 MHz, $CDCl_3$) δ 3.78 (s, 6H, 2 OCH_3), 3.80 (s, 3H, OCH_3), 4.12 (dd, 2H, ^3J 6.2 Hz, ^4J 1.4

Hz), 5.40 (s, 1H), 6.19 (dt, 1H, ^3J 14.9 Hz, ^3J 6.2 Hz), 6.51 (dd, 1H, ^3J 14.9 Hz, ^4J 1.4 Hz), 6.84 (d, 2H, ^3J 8.6 Hz), 6.86 (d, 4H, ^3J 8.6 Hz), 7.27 (d, 4H, ^3J 8.6 Hz), 7.31 (d, 2H, ^3J 8.6 Hz); δ_C (67.8 MHz, $CDCl_3$) 55.24 (2C), 55,26, 69.32, 81.62, 113.75 (4C), 113.95 (2C), 124.11, 127.68 (2C), 128.26 (4C), 129.63, 131.88, 134.68 (2C), 158.88 (2C), 159.29; MS (FAB, 3-nitrobenzyl alcohol) *m/z* (%) 391 (MH^+, 1.6). HRMS Found: 391.1904. Calcd. for $C_{25}H_{27}O_4$: 391.1909, and bis(4-methoxycinnamyl) ether; colourless solid;[9] ^{1}H NMR (270 MHz, $CDCl_3$) δ 3.81 (s, 6H, 2 OCH_3), 4.17 (dd, 4H, ^3J 5.4 Hz, ^4J 1.4 Hz), 6.20 (dt, 2H, ^3J 15.7 Hz, ^3J 5.4 Hz), 6.57 (dd, 2H, ^3J 15.7 Hz, ^4J 1.4 Hz), 6.85 (d, 4H, ^3J 8.6 Hz), 7.32 (d, 4H, ^3J 8.6 Hz); δ_C (67.8 MHz, $CDCl_3$) 55.27 (2C, 2 OCH_3), 70.83 (2C), 113.97 (4C), 123.82 (2C), 127.68 (4C), 129.53 (2C), 132.28 (2C), 159.30 (2C); MS (EI, 70 eV) *m/z* (%) 310 (M^+, 14), 148 (100). HRMS Found: 310.1566. Calcd. for $C_{20}H_{22}O_3$: 310.1569.

Bis(4-anisyl)methyl (3-methyloxetan-3-yl)methyl ether (**10i**); colourless oil; IR (neat) ν 2988, 2951, 2870, 1610, 1586, 1506, 1459, 1342, 1300, 1084, 1036, 978, 855, 755 $cm^{-1;}$ ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.27 (s, 3H, CH_3), 3.47 (s, 2H), 3.79 (s, 6H, 2 OCH_3), 4.35 (d, 2H, ^2J 5.9 Hz), 4.53 (d, 2H, ^2J 5.9 Hz), 5.28 (s, 1H), 6.85 (d, 4H, ^3J 8.6 Hz), 7.23 (d, 4H, ^3J 8.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 21.11, 39.51, 54.77 (2C), 73.47, 79.80 (2C), 82.56, 113.25 (4C), 127.65 (4C), 134.23 (2C), 158.43 (2C); MS (EI, 70 eV) *m/z* (%) = 328 (M^+, 26), 243 (29), 227 (100), 135 (19). HRMS Found: 328.1677. Calcd. for $C_{20}H_{24}O_4$: 328.1675.

Bis(4-anisyl)methyl oxiran-2-ylmethyl ether (**10j**); colourless oil; IR (neat) ν 2930, 1614, 1509, 1461, 1245, 1172, 1088, 917, 880, 829, 755 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 2.59 (dd, 1H, ^2J 5.1 Hz, ^3J Hz 2.7 Hz), 2.77 (dd, 1H, ^2J 5.1 Hz, ^3J 4,3 Hz), 3.19 (m, 1H), 3.43 (dd, 1H, ^2J 11.3 Hz, ^3J 5.7 Hz), 3.68 (dd, 1H, ^2J 11.3 Hz, ^3J 3.2 Hz), 3.78 (s, 6H, 2 OCH_3), 5.37 (s, 1H), 6.85 (d, 4H, ^3J 8.6 Hz), 7.25 (d, 4H, ^3J 8.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 44.44, 50.97, 55.24 (2C), 69.33, 80.05, 113.77 (4C), 128.16 (2C), 128.28 (2C), 134.21, 134.32, 158.96, 158.99; MS (EI, 70 eV) *m/z* (%) = 300 (M^+, 42), 242 (20), 227 (100), 135 (30). HRMS Found: 300.1364. Calcd. for $C_{18}H_{20}O_4$: 300.1362.

(4-Anisyl)-phenylmethyl heptyl ether (**10k**); colourless oil; IR (neat) ν 3060, 3028, 2932, 1612, 1582, 1512, 1453, 1337, 1301, 1246, 1171, 1096, 1036, 829, 782, 725, 698 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 0.87 (m, 3H), 1.27 (m, 8H), 1.63 (m, 2H), 3.42 (t, 2H, ^3J 6.5 Hz), 3.78 (s, 3H, OCH_3), 5.29 (s, 1H), 6.84 (d, 2H, ^3J 8.6 Hz), 7.23 - 7.32 (m, 7H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 14.10, 22,69, 26.25, 29.16, 29.92, 31.85, 55.25, 69.16, 83.14, 113.73 (2C), 126.87 (2C), 127.18, 128.28 (4C), 134.98, 142.92, 158.94; MS (EI, 70 eV) *m/z* (%) = 312 (M^+, 43), 197 (100). HRMS Found: 312.2090. Calcd. for $C_{21}H_{28}O_2$: 312.2089.

(4-Anisyl)-phenylmethyl cyclopentyl ether (**10l**); colourless oil; IR (neat) ν 2952, 1611, 1509, 1460, 1302, 1246, 1171, 1034, 810, 732, 698 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.49 (m, 2H), 1.73 (m, 6H), 3.77 (s, 3H, OCH_3), 3.96 (m, 1H), 5.37 (s, 1H), 6.84 (d, 2H, ^3J 8.6 Hz), 7.20 - 7.31 (m, 7H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 23.56 (2C), 32.35, 32.45, 55.21, 78.92, 80.62, 113.66, 127.07 (4C), 128.20 (2C), 128.44 (2C), 135.12, 143.16, 158.80; MS (EI, 70 eV) m/z (%) = 282 (M^+, 21), 197 (100). HRMS Found: 282.1619. Calcd. for $C_{19}H_{22}O_2$: 282.1620.

(4-Anisyl)-phenylmethyl benzyl ether (**10m**); colourless oil; IR (neat) ν 3062, 3028, 2932, 2858, 1611, 1585, 1511, 1459, 1302, 1246, 1172, 1093, 1035, 826, 736, 699 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 3.78 (s, 3H, OCH_3), 4.53 (s, 2H), 5.40 (s, 1H), 6.86 (d, 2H, ^3J 8.9 Hz), 7.25 - 7.39 (m, 7H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 55.24, 70.36, 82.00, 113.79 (2C), 127.00 (2C), 127.32, 127.48, 127.69 (2C), 128.34 (4C), 128.47 (2C), 134.32, 138.49, 142.41, 159.00; MS (EI, 70 eV) *m/z* (%) = 304 (M^+, 17), 213 (59), 197 (100). HRMS Found: 304.1464. Calcd. for $C_{21}H_{20}O_2$: 304.1463. Calcd. for $C_{21}H_{20}O_2 \cdot 0.2H_2O$: C, 81.89; H, 6.67. Found: C, 81.91; H, 6.61%.

(4-Anisyl)-phenylmethyl but-3-ynyl ether (**10n**); colourless oil; IR (neat) ν 3290, 3060, 3028, 3002, 2928, 2866, 2834, 1612, 1585, 1513, 1454, 1248, 1173, 1082, 1034, 809, 740, 699, 634 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.96 (t, 1H, ^4J 2.7 Hz), 2.52 (dt, 2H, ^3J 7.0 Hz, ^4J 2.7 Hz), 3.58 (t, 2H, ^3J 7.0 Hz), 3.78 (s, 3H, OCH_3), 5.37 (s, 1H), 6.85 (d, 2H, ^3J 8.6 Hz), 7.24 - 7.33 (m, 7H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 19.99, 55.24, 66.96, 69.23, 81.40, 83.31, 113.79 (2C), 126.88 (2C), 127.39, 128.34 (4C), 134.17, 142.23, 159.03; MS (EI, 70 eV) *m/z* (%) = 266 (M^+, 29), 238 (15), 197 (100), 189 (50). HRMS Found: 266.1307. Calcd. for $C_{18}H_{18}O_2$: 266.1307. Calcd. for $C_{18}H_{18}O_2 \cdot 0.4H_2O$: C, 79.03; H, 6.93. Found: C, 79.00; H, 6.74%.

(4-Anisyl)-phenylmethyl fur-2-yl ether (**10o**); pale yellow oil; IR (neat) ν 3112, 3028, 2930, 2836, 1611, 1510, 1459, 1246, 1171, 1038, 919, 812, 738, 698 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 3.78 (s, 3H, OCH_3), 4.46 (s, 2H), 6.28 (bd, 1H, ^3J 3.0 Hz), 6.33 (dd, 1H, ^3J 3.0 Hz, ^3J 1.9 Hz), 6.85 (d, 2H, ^3J 8.6 Hz), 7.24 - 7.33 (m, 7H), 7.41 (dd, 1H, ^3J 1.9 Hz, ^4J 1.0 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 55.24, 62.45, 81.71, 109.30, 110.19, 113.80 (2C), 127.08 (2C), 127.37, 128.34 (2C), 128.57 (2C), 133.93, 142.02, 142.71, 151.92, 159.04; MS (EI, 70 eV) *m/z* (%) = 294 (M^+, 16), 213 (49), 197 (100). HRMS Found: 294.1256. Calcd. for $C_{19}H_{18}O_3$: 294.1256.

(2-Anisyl)-(4-anisyl)methyl benzyl ether (**10p**); colourless oil; IR (neat) ν 3062, 3030, 3002, 2935, 2834, 1610, 1513, 1459, 1243, 1172, 1052, 826, 756, 698 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 3.76 (s, 3H, OCH_3), 3.77 (s, 3H, OCH_3), 4.49 (d, 1H, ^2J 11.9 Hz), 4.54 (d, 1H, ^2J 11.9 Hz), 5.83 (s, 1H), 6.82 (m, 1H), 6.83 (d, 2H, ^3J 8.9 Hz), 6.98 (m, 1H), 7.19 - 7.37 (m, 8H), 7.57 (m, 1H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 55.20, 55.37, 70.56, 76.01, 110.54, 113.51 (2C), 120.77, 126.95, 127.31, 127.65 (2C), 128.21 (3C), 128.43 (2C), 130.92, 134.38, 138.80, 156.63, 158.69; MS (EI, 70 eV) *m/z* (%) = 334 (M^+, 45), 243 (69), 227 (90), 135 (100), 121 (87), 91 (77). HRMS Found: 334.1572. Calcd. for $C_{22}H_{22}O_3$: 334.1569.

(2-Anisyl)-(4-anisyl)methyl pentyl ether (**10q**); colourless oil; IR (neat) ν 2958, 1612, 1511, 1491, 1459, 1244, 1179, 1091, 1032, 818, 754 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 0.88 (t, 3H, ^3J 7.3 Hz, CH_3), 1.32 (m, 4H), 1.63 (m, 2H), 3.43 (t, 2H, ^3J 6.7 Hz), 3.76 (s, 3H, OCH_3), 3.79 (s, 3H, OCH_3), 5.71 (s, 1H), 6.81 (d, 2H, ^3J 8.9 Hz), 6.82 (m, 1H), 6.96 (m, 1H), 7.19 (m, 1H), 7.28 (d, 2H, ^3J 8.9 Hz),

7.49 (dd, 1H, ^3J 7.6 Hz, ^4J 1.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 14.03, 22.54, 28.44, 29.65, 55.19, 55.40, 69.16, 110.48, 113.46 (2C), 120.72, 126.79, 128.02, 128.27 (2C), 131.40, 134.88, 156.45, 158.59. MS (EI, 70 eV) *m/z* (%) = 314 (M^+, 59), 243 (50), 227 (100), 135 (37), 121 (58). HRMS Found: 314.1883. Calcd. for $C_{20}H_{26}O_3$: 314.1882. Calcd. for $C_{20}H_{26}O_3 \cdot 0.1H_2O$: C, 75.96; H, 8.36. Found: C, 75.62; H, 8.30%.

(2-Anisyl)-(4-anisyl)methyl but-1-ynyl ether (**10r**); colourless oil; IR (neat) ν3296, 2928, 1611, 1509, 1491, 1460, 1246, 1178, 1090, 825, 755 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.95 (m, 1H), 2.51 (m, 2H), 3.58 (m, 2H), 3.76 (s, 3H, OCH_3), 3.78 (s, 3H, OCH_3), 5.78 (s, 1H), 6.81 (d, 2H, ^3J 8.9 Hz), 6.83 (m, 1H), 6.96 (m, 1H), 7.22 (m, 1H), 7.29 (d, 2H, ^3J 8.9 Hz), 7.49 (d, 1H, ^3J 7.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 19.97, 55.20, 55.40, 67.04, 69.13, 76.69, 81.48, 110.50, 113.53 (2C), 120.74, 126.86, 128.29, 128.32 (2C), 130.68, 134.17, 156.54, 158.75; MS (EI, 70 eV) m/z (%) = 296 (M^+, 55), 243 (42), 227 (86), 189 (29), 135 (50), 121 (100). HRMS Found: 296.1415. Calcd. for $C_{19}H_{20}O_3$: 296.1412.

(2-Anisyl)-(4-anisyl)methyl (3-methyloxetan-3-yl)methyl ether (**10s**); colourless oil; IR (neat) ν 2928, 2864, 1613, 1514, 1487, 1468, 1244, 1170, 1086, 1032, 981, 833, 756 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 1.35 (s, 3H, CH_3), 3.48 (d, 1H, ^2J 8.6 Hz), 3.51 (d, 1H, ^2J 8.6 Hz), 3.76 (s, 3H, OCH_3), 3.79 (s, 3H, OCH_3), 4.34 (d, 2H, ^2J 5.4 Hz), 4.54 (d, 2H ^2J 5.4 Hz), 5.73 (s, 1H), 6.82 (d, 2H, ^3J 8.9 Hz), 6.83 (m, 1H), 6.96 (m, 1H), 7.26 (m, 1H), 7.28 (d, 2H, ^3J 8.9 Hz), 7.46 (dd, 1H, ^3J 7.6 Hz, ^4J 1.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 21.56, 31.57, 39.56, 55.17, 55.36, 74.11, 80.27 (2C), 80.32, 110.44, 113.48 (2C), 120.72, 126.74, 128.25 (3C), 131.04, 134.37, 156.48, 158.68; MS (EI, 70 eV) *m/z* (%) = 328 (M^+, 37), 243 (100), 227 (61), 221 (26), 121 (78). HRMS Found: 328.1678. Calcd. for $C_{20}H_{24}O_4$: 328.1675.

(2-Anisyl)-(4-anisyl)methyl cyclopentyl ether (**10t**); colourless oil; IR (neat) ν 2936, 2836, 1611, 1510, 1489, 1458, 1339, 1246, 1171, 1112, 1030, 986, 813, 754 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.78 (s, 1H), 1.72 (m, 6H), 1.47 (m, 2H), 3.75 (s, 3H, OCH_3), 3.77 (s, 3H, OCH_3), 3.93 (m, 1H), 6.80 (d, 2H, ^3J 8.9 Hz), 6.81 (m, 1H), 6.95 (m, 1H), 7.21 (m, 1H), 7.24 (d, 2H, ^3J 8.9 Hz), 7.46 (dd, 1H, ^3J 7.6 Hz, ^4J 1.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 23.58 (2C), 32.38, 32.50, 55.17, 55.36, 74.14, 79.05, 110.39, 113.42 (2C), 120.65, 127.36, 127.99, 128.40 (2C), 131.60, 135.15, 156.63, 158.15; MS (EI, 70 eV) *m/z* (%) = 312 (M^+, 33), 243 (12), 227 (100), 121 (50). HRMS Found: 312.1728. Calcd. for $C_{20}H_{24}O_3$: 312.1725.

Diphenylmethyl heptyl ether (**10u**) [18]; colourless oil; IR (neat) ν 3062, 3026, 2924, 2856, 1600, 1492, 1460, 1378, 1303, 1185, 1107, 797, 700, 653, 618 cm^{-1}; MS (EI, 70 eV) *m/z* (%) = 282 (M^+, 7.5), 243 (31), 213 (42), 167 (100). HRMS Found: 282.1981. Calcd. for $C_{20}H_{26}O$: 282.1984.

4-Ethoxy-1-(1-octyloxypropyl)benzene (**12a**); colourless oil; IR (neat) ν 2930, 2852, 1611, 1510, 1459, 1244, 1173, 1097, 1050, 963, 924, 828 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 0.87 (t, 3H, ^3J 7.3 Hz, CH_3), 0.93 (t, 3H, ^3J 6.8 Hz), 1.42 (t, 3H, ^3J 7.0 Hz), 1.54 - 1.87 (m, 14H), 3.40 (t, 2H, ^3J 6.5 Hz), 4.02 (q, 2H, ^3J 7.0 Hz), 4.16 (dd, 1H, ^3J 6.5 Hz, ^3J 6.5 Hz), 6.86 (d, 2H, ^3J 8.6 Hz), 7.23 (d, 2H, ^3J 8.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 10.37, 14.07, 14.88, 22.64, 26.22, 29.26, 29.42, 29.91, 31.11, 31.83, 63.37, 68.66, 83.31, 114.13 (2C), 127.81 (2C), 134.98, 158.21; MS (EI, 70 eV) *m/z* (%) = 292 (M^+, 4.2), 263 (100), 151 (81). HRMS Found: 292.2403. Calcd. for $C_{19}H_{32}O_2$: 292.2402.

4-Ethoxy-1-(1-benzyloxypropyl)benzene (**12b**); colourless oil; IR (neat) ν 3062, 3028, 2930, 2866, 1612, 1584, 1512, 1454, 1394, 1304, 1244, 1172, 1066, 921, 827, 732, 697 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 0.87 (t, 3H, ^3J 7.3 Hz, CH_3), 1.42 (t, 3H, ^3J 7.0 Hz, CH_3), 1.67 (m, 1H), 1.87 (m, 1H), 4.04 (q, 2H, ^3J 7.0 Hz), 4.16 (dd, 1H, ^3J 6.5 Hz, ^3J 6.5 Hz), 4.23 (d, 1H, ^2J 11.9 Hz), 4.43 (d, 1H, ^2J 11.9 Hz), 6.88 (d, 2H, ^3J 8.6 Hz), 7.20 - 7.32 (m, 5H), 7.23 (d, 2H, ^3J 8.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 10.38, 14.89, 31.06, 63.40, 70.10, 82.54, 114.28 (2C), 127.35, 127.73 (2C), 128.07 (2C), 128.27 (2C), 134.29, 138.88, 158.41; MS (EI, 70 eV) *m/z* (%) = 270 (M^+, 13), 241 (88), 213 (11), 91 (100). HRMS Found: 270.1618. Calcd. for $C_{18}H_{22}O_2$: 270.1620.

4-Ethoxy-1-(1-cyclopentyloxypropyl)benzene (**12c**); colourless oil; IR (neat) ν 2936, 1612, 1511, 1456, 1390, 1341, 1302, 1247, 1173, 1048, 965, 922, 831 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 0.87 (t, 3H, ^3J 7.3 Hz, CH_3), 1.26 - 1.70 (m, 8H), 1.41 (t, 3H, ^3J 7.0 Hz, CH_3), 3.76 (m, 1H), 4.02 (q, 2H, ^3J 7.0 Hz), 4.16 (m, 1H), 6.85 (d, 2H, ^3J 8.6 Hz), 7.19 (d, 2H, ^3J 8.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 10.59, 14.91, 23.42 (2C), 31.54, 31.59, 33.14, 63.37, 78.39, 80.91, 114.10 (2C), 127.83 (2C), 135.58, 158.17; MS (EI, 70 eV) *m/z* (%) = 248 (M^+, 3.8), 219 (29), 163 (38), 151 (100), 107 (19). HRMS Found: 248.1777. Calcd. for $C_{16}H_{24}O_2$: 248.1776.

4-Pentoxy-1-(1-octyloxyethyl)benzene (**12d**); colourless oil; IR (neat) ν 2928, 2856, 1615, 1512, 1469, 1246, 1173, 1102, 832 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 0.87 (t, 3H, ^3J 6.8 Hz), 0.93 (t, 3H, ^3J 6.8 Hz), 1.25 - 1.57 (m, 16H), 1.41 (d, 3H, ^3J 6.5 Hz, CH_3), 1.78 (m, 2H), 3.25 (t, 2H, ^3J 6.8 Hz), 3.94 (t, 2H, ^3J 6.8 Hz), 4.32 (q, 1H, ^3J 6.5 Hz), 6.86 (d, 2H, ^3J 8.6 Hz), 7.21 (d, 2H, ^3J 8.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 14.00, 14.07, 22.46, 22.64, 24.11, 26.19, 28.24, 29.03, 29.25, 29.42, 29.95, 31.82, 67.98, 68.53, 77.38, 114.28 (2C), 127.27 (2C), 136.12, 158.45; MS (EI, 70 eV) *m/z* (%) = 320 (M^+, 18), 305 (100), 193 (45), 121 (28). HRMS Found: 320.2714. Calcd. for $C_{21}H_{36}O_2$: 320.2715.

4-Pentoxy-1-(1-benzyloxyethyl)benzene (**12e**); colourless oil; IR (neat) ν 2926, 2858, 1611, 1510, 1244, 1173, 1094, 909, 831, 734, 696 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 0.93 (t, 3H, ^3J 7.3 Hz, CH_3), 1.34 - 1.48 (m, 4H), 1.46 (d, 3H, ^3J 6.5 Hz, CH_3), 1.78 (m, 2H), 3.96 (t, 2H, ^3J 6.8 Hz), 4.26 (d, 1H, ^2J 11.9 Hz), 4.42 (d, 1H, ^2J 11.9 Hz), 4.44 (t, 1H, ^3J 6.5 Hz), 6.89 (d, 2H, ^3J 8.6 Hz), 7.24 - 7.32 (m, 5H), 7.26 (d, 2H, ^3J 8.6 Hz); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 14.95, 23.41, 25.03, 29.18, 29.96, 68.95, 70.97, 77.65, 115.36 (2C), 128.33, 128.47 (2C), 128.64 (2C), 129.25 (2C), 136.41, 139.72, 159.59; MS (EI, 70 eV) *m/z* (%) = 298 (M^+, 62), 283 (40), 255 (19), 207 (29), 191 (48), 134 (17), 121 (64), 91 (100). HRMS Found: 298.1936. Calcd. for $C_{20}H_{26}O_2$: 298.1933.

3,5-Dimethyl-4-methoxy-1-(1-benzyloxy-2-methylpropyl) benzene (**12f**); colourless oil; IR (neat) ν 3032, 2939, 1605, 1510, 1491, 1476, 1460, 1223, 1096, 1020, 881, 848, 735,

698 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 0.72 (d, 3H, ^3J 6.7 Hz, CH_3), 1.03 (d, 3H, ^3J 6.7 Hz, CH_3), 1.91 (m, 1H), 2.28 (s, 6H, 2 CH_3), 3.73 (s, 3H, OCH_3), 3.82 (d, 1H, ^3J 7.6 Hz), 4.19 (d, 1H, ^2J 12.1 Hz), 4.43 (d, 1H, ^2J 12.1 Hz), 6.91 (s, 2H), 7.24 - 7.35 (m, 5H); ^{13}C NMR (67.8 MHz, $CDCl_3$) δ 16.21 (2C), 19.23, 34.84 (2C), 59.64, 70.44, 86.98, 127.24, 127.63 (2C), 127.90 (2C), 128.19 (2C), 130.21 (2C), 136.45, 139.09, 156.19; MS (EI, 70 eV) *m/z* (%) = 298 (M^+, 2.2), 255 (42), 163 (17), 91 (100). HRMS Found: 298.1936. Calcd. for $C_{20}H_{26}O_2$: 298.1933.

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