

# Allylation of Diselenides, Aldehydes and Ketones with Ytterbium/Allyl Bromide System

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In the presence of methyl iodide, ytterbium can react with allyl bromide smoothly to form allylytterbium bromide, which further reacts with diselenides, aldehydes and ketones to afford allylselenides and homoallylic alcohols respectively in good yields under mild and neutral conditions.

**Keywords** Diselenides, aldehyde, ketone, allyl bromide, ytterbium, allylytterbium bromide

## Introduction

Rapid development of lanthanides, especially Samarium reagents, in organic synthesis has been recently achieved.<sup>1</sup> However, studies on application of ytterbium metal in organic synthesis were relatively few.<sup>2</sup> Nevertheless, there are indications that ytterbium reagents have the potential to serve as selective nucleophiles in C=X  $\pi$ -bond addition reactions, and eventually they will play a unique role among the other lanthanide reagents in organic synthesis.

Hou<sup>3</sup> and his group reported the reaction of ytterbium with diaryl ketones such as benzophenone produced the corresponding dianionic complexes that react easily with a variety of electrophiles like ketones, esters, epoxides, nitriles, carbon dioxide and acetylenes producing adducts. As Grignard-type reagent, phenyllytterbium iodide can react with carbon dioxide to form the carboxylic acid;<sup>4</sup> with benzophenone to give triphenylmethanol; with chlorodimethylsilane or chlorotrimethyl-

silane to provide trimethylphenylsilane, with isocyanobenzene to produce benzenilide;<sup>5,6</sup> with aldehydes and ketones to afford corresponding alcohols;<sup>7-9</sup> with carboxylic acid derivatives to yield ketones.<sup>10,11</sup> But there are no reports on the allylation of diselenides, aldehydes or ketones with organoytterbium reagent.

Here we wish to report the preparation of allylytterbium bromide (Scheme 1) and the synthesis of allylselenides and homoallylic alcohols by allylytterbium bromide (Scheme 2).

## Results and discussion

In our experimental work, it was found that ytterbium metal neither reacts directly with allyl bromide to form allylytterbium bromide as does samarium, nor can be activated by addition of potassium iodide, sodium iodide or iodine as can samarium. Surprisingly, in the presence of a catalytic amount of methyl iodide, ytterbium can react with allyl bromide easily to produce allylytterbium bromide (Scheme 1). It was also found that diselenides, aldehydes and ketones reacted with allylytterbium bromide more easily than with allylsamarium bromide. The results were summarized in Table 1 and Table 2, respectively.

Table 1 shows that dialkyl diselenides can react with allylytterbium bromide to give alkylselenides in moderate yields and diaryl diselenides give corresponding

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arylselenides in good yields. It is noteworthy that electrostatic effect in the aromatic ring affects the yields of allylselenides. If the substituted groups are electron withdrawing in the substrates, the yields are higher than the case of substrates with electron-donating groups. As shown in Table 1, although allyl ytterbium bromide is more reactive than allylsamarium bromide, it is not so stable as allylsamarium bromide.

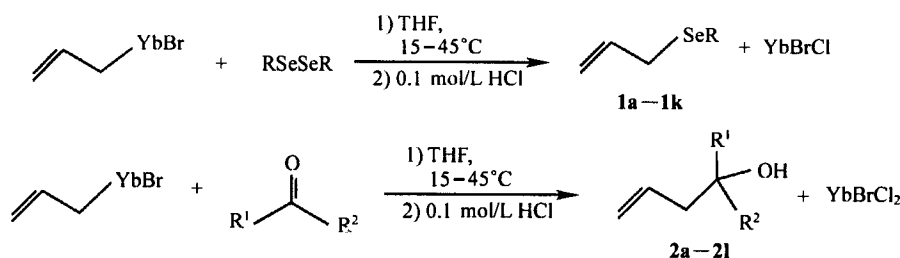
As shown in Table 2, the yields of the allylation for

ketones with allyl ytterbium bromide are higher than those for aldehydes. As to the temperature effect on the reaction efficiency, it was found that increasing reaction temperature could not provide better yields of homoallylic alcohols (Entries **2g**–**2l**). This phenomenon was probably caused by the lower stability of allyl ytterbium bromide with the increase of temperature and as a result, some side-reaction such as coupling reaction of allyl ytterbium bromide may occur at the same time.

### Scheme 1



### Scheme 2



## Experimental

### General

Tetrahydrofuran (THF) was freshly distilled from sodium-benzophenone and allyl bromide was also distilled before use. Infrared spectra were recorded on an IR-408 spectrometer in film with absorption in  $\text{cm}^{-1}$ .  $^1\text{H-NMR}$  spectra were recorded on a Bruker AC-80 spectrometer using  $\text{CDCl}_3$  as solutions, and tetramethylsilane as internal standard ( $\delta = 0$ ). Mass spectra were recorded on a HP 5989B MS spectrometer. Microanalysis was carried out on a Carlo-Erba 1106 instrument.

### General procedure for the preparation of allyl ytterbium bromide

0.173 g (1 mmol) of ytterbium powder and 1 drop of methyl-iodide were added to a three-necked flask at room temperature under a nitrogen atmosphere. The mixture was heated slightly to activate the ytterbium metal for about 15 min, and then was cooled to room temperature. Anhydrous THF (2 mL) was added with a syringe to give

a colored slurry (pale yellow). The slurry was cooled to  $0^\circ\text{C}$ , and 0.121 g (1 mmol) of freshly distilled allyl bromide in anhydrous THF (15 mL) was added with a syringe. The mixture was stirred at  $0^\circ\text{C}$  for 10–15 min. A brown solution of allyl ytterbium bromide was formed.

### General procedure for the synthesis of allylselenides

A solution of diselenides<sup>23,24</sup> (0.5 mmol) in THF (2 mL) was added to the brown solution of allyl ytterbium bromide prepared mentioned above by a syringe in one portion at noted temperature (Table 1) under nitrogen atmosphere. After being stirred for given time (monitored by TLC), the reaction was quenched with 5 mL of 0.1 mol/L HCl and extracted with ether ( $3 \times 20$  mL). The crude product was isolated according to usual procedure and purified by preparative thin layer chromatography with cyclohexane as eluent.

Allylsamarium bromide was prepared according to the published procedure.<sup>1</sup> The allylation of diselenides with allylsamarium bromide was carried out following the procedure.<sup>15</sup>

Table 1 Allylation of diselenides with allyl metal bromide

Entry	R	Metal	Time (h)	Temperature (°C)	Yield (%) <sup>a</sup>
1a	C <sub>6</sub> H <sub>5</sub>	Sm	1	25	83
		Yb	0.5	15	85
1b	<i>p</i> -ClC <sub>6</sub> H <sub>4</sub>	Sm	1	25	85
		Yb	0.5	15	87
1c	<i>o</i> -ClC <sub>6</sub> H <sub>4</sub>	Sm	1	25	86
		Yb	0.5	15	87
1d	<i>m</i> -ClC <sub>6</sub> H <sub>4</sub>	Sm	1	25	83
		Yb	0.5	15	84
1e	<i>p</i> -MeC <sub>6</sub> H <sub>4</sub>	Sm	1	25	80
		Yb	0.5	15	79
1f	<i>o</i> -MeC <sub>6</sub> H <sub>4</sub>	Sm	1	25	78
		Yb	0.5	15	78
1g	<i>m</i> -MeC <sub>6</sub> H <sub>4</sub>	Sm	1	25	78
		Yb	0.5	15	83
1h	2-Pyridyl	Sm	10 min	25	90
		Yb	2 min	15	92
		Sm	1	reflux	56
1i	C <sub>2</sub> H <sub>5</sub>	Yb	0.5	40—45	57
		Yb	0.5	reflux	38
		Sm	1	reflux	62
1j	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>3</sub>	Yb	0.5	40—45	62
		Yb	0.5	reflux	35
		Sm	1	reflux	58
1k	CH <sub>3</sub> CH <sub>2</sub> CH(CH <sub>3</sub> )	Yb	0.5	40—45	60
		Yb	0.5	reflux	33

<sup>a</sup> Isolated yields.

Table 2 Allylation of aldehydes and ketones with allylterbium bromide

Entry	R <sup>1</sup>	R <sup>2</sup>	Temperature (°C)	Time (h)	Yield (%) <sup>a</sup>
2a	C <sub>6</sub> H <sub>5</sub>	H	25	1	75
2b	<i>p</i> -CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	H	25	1	78
2c	<i>p</i> -CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	H	25	1	76
2d	<i>p</i> -ClC <sub>6</sub> H <sub>4</sub>	H	25	1	80
2e	<i>o</i> -ClC <sub>6</sub> H <sub>4</sub>	H	25	1	79
2f	<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	H	25	1	81
2g	C <sub>6</sub> H <sub>5</sub>	CH <sub>3</sub>	25	1	82
			15	1	88
2h	<i>p</i> -ClC <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	25	1	79
			15	1	83
2i	<i>p</i> -BrC <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	25	1	81
			15	1	86
2j	<i>p</i> -CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	25	1	80
			15	1	83
2k	<i>p</i> -CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	25	1	78
			15	1	82
2l	C <sub>6</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub>	25	1	84
			15	1	87

<sup>a</sup> yields of isolated products based on aldehydes or ketones. All products gave satisfactory <sup>1</sup>H NMR and IR spectral data.*General procedure for the synthesis of homoallylic alcohols*

Aldehydes or ketones (1 mmol) in THF (2 mL) were added to the brown solution of allylterbium bro-

midate. The mixture was stirred for 1 h at given temperatures shown in Table 2 under nitrogen, followed by hydrolysis with 0.1 mol/L HCl, extracted with ether (30 mL × 2). The combined organic extracts were washed

with brine (20 mL  $\times$  2), dried over anhydrous  $\text{MgSO}_4$ , concentrated *in vacuo* and purified by preparative TLC on silica gel (cyclohexane/ethyl acetate as eluent) to give monoallylic alcohols in good yields. All of the allylic alcohols prepared were identified by IR and  $^1\text{H}$ -NMR spectra.

**1a:**<sup>12-14</sup> oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 3.15—3.45 (d,  $J = 6.5$  Hz, 2H), 4.60—5.00 (m, 2H), 5.50—5.95 (m, 1H), 7.00—7.60 (m, 5H); IR (film)  $\nu$ : 3080, 2940, 1640, 1480, 1180, 990, 910, 730, 680  $\text{cm}^{-1}$ .

**1b:**<sup>12-14</sup> oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 3.20—3.55 (d,  $J = 6.5$  Hz, 2H), 4.60—5.05 (d,  $J = 5.2$  Hz, 2H), 5.60—6.00 (m, 1H), 6.95—7.60 (m, 4H); IR (film)  $\nu$ : 3110, 2950, 1650, 1480, 1180, 1100, 1000, 820, 690  $\text{cm}^{-1}$ .

**1c:** light yellow oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 3.40—3.50 (d,  $J = 6.5$  Hz, 2H), 4.85—5.20 (d,  $J = 5.2$  Hz, 2H), 5.50—6.00 (m, 1H), 6.90—7.40 (m, 4H); IR (film)  $\nu$ : 3050, 2930, 1635, 1580, 1456, 1435, 1250, 1200, 1130, 920, 750  $\text{cm}^{-1}$ ; MS  $m/z$  (%): 234 ( $\text{M}^+ + 2$ , 24), 232 ( $\text{M}^+$ , 71), 191 (24), 112 (6), 41 (100); Anal. calcd for  $\text{C}_6\text{H}_9\text{ClSe}$ : C 46.68, H 3.92; Found: C 46.56, H 3.80.

**1d:** red oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 3.50—3.57 (d,  $J = 6.5$  Hz, 2H), 4.99—5.05 (d,  $J = 5.2$  Hz, 2H), 5.91—5.98 (m, 1H), 7.18—7.49 (m, 4H); IR (film)  $\nu$ : 3100, 2950, 2400, 1590, 1470, 1100, 920, 780, 750, 680  $\text{cm}^{-1}$ ; MS  $m/z$  (%): 234 ( $\text{M}^+ + 2$ , 15), 232 ( $\text{M}^+$ , 43), 191 (19), 112 (8), 41 (100); Anal. calcd for  $\text{C}_9\text{H}_9\text{ClSe}$ : C 46.68, H 3.92; found: C 46.58, H 3.86.

**1e:**<sup>12-14</sup> oil;  $^1\text{H}$ NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 2.30 (s, 1H), 3.20—3.45 (d,  $J = 6.5$  Hz, 2H), 4.50—5.00 (d,  $J = 5.2$  Hz, 2H), 5.50—5.90 (m, 1H), 6.70—7.50 (m, 4H); IR (film)  $\nu$ : 3100, 2970, 2880, 1640, 1510, 1380, 990, 840  $\text{cm}^{-1}$ .

**1f:**<sup>15</sup> oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 2.33 (s, 3H), 3.18—3.43 (d,  $J = 6.5$  Hz, 2H), 4.60—5.10 (d,  $J = 5.2$  Hz, 2H), 5.60—6.00 (m, 1H), 6.70—7.60 (m, 4H); IR (film)  $\nu$ : 3080, 2960, 1645, 1510, 1380, 990, 900, 830  $\text{cm}^{-1}$ .

**1g:** red oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 2.40 (s, 3H), 3.30—3.40 (d,  $J = 6.5$  Hz, 2H), 4.70—5.00 (d,  $J = 5.2$  Hz, 2H), 5.50—6.00 (m, 1H), 6.90—7.20 (m, 4H); IR (film)  $\nu$ : 3370, 3030,

2920, 2360, 1580, 1470, 1280, 1210, 1160, 1060, 980, 910, 820, 770, 670  $\text{cm}^{-1}$ ; MS  $m/z$  (%): 212 ( $\text{M}^+$ , 21), 211 (3), 171 (13), 91 (100), 77 (6), 41 (20); Anal. calcd for  $\text{C}_{10}\text{H}_{12}\text{Se}$ : C 56.88; H 5.73; found: C 56.70; H 5.70.

**1h:** light yellow oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 3.7—3.80 (d,  $J = 6.5$  Hz, 2H), 4.80—5.20 (d,  $J = 5.2$  Hz, 2H), 5.60—6.20 (m, 1H), 6.85—8.40 (m, 4H); IR (film)  $\nu$ : 3050, 2940, 2375, 1630, 1575, 1450, 1415, 1280, 1110, 980, 910, 760, 700  $\text{cm}^{-1}$ ; MS  $m/z$  (%): 200 ( $\text{M}^+ + 2$ , 27), 199 ( $\text{M}^+ + 1$ , 19), 198 ( $\text{M}^+$ , 19), 197 (14), 196 (10), 184 (72), 118 (100), 119 (45), 78 (48); Anal. calcd for  $\text{C}_8\text{H}_9\text{NSe}$ : C 48.51; H 4.58; N, 7.07; found: C 48.65; H 4.40; N, 6.87.

**1i:**<sup>16</sup> oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 1.25—1.48 (t,  $J = 7.0$  Hz, 3H), 3.15—3.50 (m, 4H), 4.55—5.00 (d,  $J = 5.2$  Hz, 2H), 5.50—5.90 (m, 1H); IR (film)  $\nu$ : 2970, 2940, 2860, 1650, 1210, 1160, 1000, 910  $\text{cm}^{-1}$ .

**1j:**<sup>16</sup> oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\nu$ : 1.0—1.68 (m, 7H), 2.70—3.00 (m, 2H), 3.15—3.45 (d,  $J = 6.5$  Hz, 2H), 4.60—5.0 (d,  $J = 5.2$  Hz, 2H), 5.50—5.90 (m, 1H); IR (film)  $\nu$ : 2965, 2930, 2890, 1640, 1470, 1320, 990, 910  $\text{cm}^{-1}$ .

**1k:**<sup>15</sup> oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 1.00—1.72 (m, 11H), 2.90—3.50 (m, 4H), 4.60—5.95 (m, 3H); IR (film)  $\nu$ : 3000, 2900, 2870, 1640, 1190, 990, 890  $\text{cm}^{-1}$ .

**2a:**<sup>17</sup> oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 1.70 (s, 1H, OH), 2.4 (t,  $J = 6.5$  Hz, 2H,  $\text{CH}_2$ ), 4.55 (t,  $J = 5.2$  Hz, 1H, CH), 4.75—5.90 (m, 3H, CH =  $\text{CH}_2$ ), 7.10—7.30 (m, 5H, ArH); IR (film)  $\nu$ : 3350 (br, OH), 1640 (w, C = C), 1200, 950, 910 (m,  $\text{CH}_2 = \text{C}$ )  $\text{cm}^{-1}$ .

**2b:**<sup>18</sup> oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 1.75 (s, 1H, OH), 1.90 (s, 3H,  $\text{CH}_3$ ), 2.35 (t,  $J = 6.50$ , 2H,  $2\text{H}_2$ ), 4.50 (t,  $J = 5.2$  Hz, 1H, CH), 4.76—5.90 (m, 3H, CH =  $\text{CH}_2$ ), 6.95—7.20 (m, 4H, ArH); IR (film)  $\nu$ : 3380 (br, OH), 1650 (w, C = C), 1190, 990, 910 (m,  $\text{CH}_2 = \text{C}$ )  $\text{cm}^{-1}$ .

**2c:**<sup>18</sup> oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 80 MHz)  $\delta$ : 1.80 (s, 1H, OH), 2.30 (t,  $J = 6.5$  Hz, 2H,  $\text{CH}_2$ ), 3.75 (s, 3H,  $\text{OCH}_3$ ), 4.45 (t,  $J = 5.0$  Hz, 1H, CH), 4.80—6.00 (m, 3H, CH =  $\text{CH}_2$ ), 6.65—6.90 (m, 4H, ArH); IR (film)  $\nu$ : 3350 (s, OH), 1645 (w, C = C), 1200, 980, 910 (m,  $\text{CH}_2 = \text{C}$ )  $\text{cm}^{-1}$ .

**2d:**<sup>19</sup> oil; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 80 MHz) δ: 1.75 (s, 1H, OH), 2.27 (t, *J* = 6.5 Hz, 2H, CH<sub>2</sub>), 4.43 (t, *J* = 5.0 Hz, 1H, CH), 4.70—5.90 (m, 3H, CH = CH<sub>2</sub>), 7.00—7.20 (m, 4H, ArH); IR (film) ν: 3360 (s, OH), 1650 (w, C = C), 1210, 985, 915 (m, CH<sub>2</sub> = C) cm<sup>-1</sup>.

**2e:**<sup>20</sup> oil; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 80 MHz) δ: 1.75 (s, 1H, OH), 2.27 (t, *J* = 6.5 Hz, 2H, CH<sub>2</sub>), 4.53 (t, *J* = 5.0 Hz, 1H, CH), 4.80—6.00 (m, 3H, CH = CH<sub>2</sub>), 7.00—7.40 (m, 4H, ArH); IR (film) ν: 3365 (s, OH), 1650 (w, C = C), 1215, 990, 920 (m, CH<sub>2</sub> = C) cm<sup>-1</sup>.

**2f:**<sup>19</sup> oil; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 80 MHz) δ: 1.72 (s, 1H, OH), 2.35 (t, *J* = 6.5 Hz, 2H, CH<sub>2</sub>), 4.50 (t, *J* = 5.0 Hz, 1H, CH), 4.80—5.98 (m, 3H, CH = CH<sub>2</sub>), 7.00—7.45 (m, 4H, ArH); IR (film) ν: 3370 (s, OH), 1655 (w, C = C), 1215, 995, 925 (m, CH<sub>2</sub> = C) cm<sup>-1</sup>.

**2g:**<sup>21</sup> oil; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 80 MHz) δ: 1.30 (s, 3H, CH<sub>3</sub>), 1.60 (s, 1H, OH), 2.40 (d, *J* = 6.5 Hz, 2H, CH<sub>2</sub>), 4.75—5.70 (m, 3H, CH = CH<sub>2</sub>), 6.85—7.40 (m, 4H, ArH); IR (film) ν: 3400 (s, OH), 1645 (w, C = C), 910 (m, CH<sub>2</sub> = C) cm<sup>-1</sup>.

**2h:**<sup>19</sup> oil; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 80 MHz) δ: 1.40 (s, 3H, CH<sub>3</sub>), 1.90 (s, 1H, OH), 2.45 (d, *J* = 6.4 Hz, 2H, CH<sub>2</sub>), 4.80—5.80 (m, 3H, CH = CH<sub>2</sub>), 7.30—7.65 (m, 4H, ArH); IR (film) ν: 3390 (br, OH), 1660 (w, C = C), 920 (m, CH<sub>2</sub> = C) cm<sup>-1</sup>.

**2i:**<sup>22</sup> oil; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 80 MHz) δ: 1.50 (s, 3H, CH<sub>3</sub>), 2.00 (s, 1H, OH), 2.55 (d, *J* = 6.3 Hz, 2H, CH<sub>2</sub>), 5.0—5.80 (m, 3H, CH = CH<sub>2</sub>), 7.25—7.60 (m, 4H, ArH); IR (film) ν: 3400 (br, OH), 1665 (w, C = C), 920 (m, CH<sub>2</sub> = C) cm<sup>-1</sup>.

**2j:**<sup>22</sup> oil; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 80 MHz) δ: 1.35 (s, 3H, CH<sub>3</sub>), 2.05 (s, 1H, OH), 2.40 (d, *J* = 6.2 Hz, 2H, CH<sub>2</sub>), 3.65 (s, 3H, OCH<sub>3</sub>), 4.80—5.80 (m, 3H, CH = CH<sub>2</sub>), 6.70—6.90 (m, 4H, ArH); IR (film) ν: 3420 (br, OH), 1660 (w, C = C), 920 (m, CH<sub>2</sub> = C) cm<sup>-1</sup>.

**2k:**<sup>19</sup> oil; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 80 MHz) δ: 1.40 (s, 3H, CH<sub>3</sub>), 1.85 (s, 1H, OH), 2.55 (d, *J* = 6.5 Hz, 2H, CH<sub>2</sub>), 5.0—5.7 (m, 3H, CH = CH<sub>2</sub>), 6.80—7.20 (m, 4H, ArH); IR (film) ν: 3430 (br, OH), 1640 (w, C = C), 915 (m, CH<sub>2</sub> = C) cm<sup>-1</sup>.

**2l:**<sup>20</sup> oil; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 80 MHz) δ: 2.55 (s, 1H, OH), 3.25 (d, *J* = 6.8 Hz, 2H, CH<sub>2</sub>), 5.20—6.20 (m, 3H, CH = CH<sub>2</sub>), 7.00—7.80 (m, 10H, ArH); IR (film) ν: 3500 (br, OH), 1650 (w, C = C), 1200, 1000, 930 (m, CH<sub>2</sub> = C) cm<sup>-1</sup>.

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