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# Rare Earth Metal Trifluoromethanesulfonates Catalyzed Benzyl-Etherification

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**Rare earth metal trifluoromethanesulfonates [rare earth metal triflate, RE(OTf)$_3$] were found to be efficient catalyst for benzyl-etherification. In the presence of a catalytic amount of RE(OTf)$_3$, condensation of benzyl alcohols and aliphatic alcohols proceeded smoothly to afford the benzyl ethers. The condensation between benzyl alcohols and thiols also proceeded, and thio ethers were obtained in good yield. In these reactions, RE(OTf)$_3$ could be recovered easily after the reactions were completed and could be reused without loss of activity.**

**Key words** rare earth metal trifluoromethanesulfonate; benzyl-etherification; recyclable catalyst; benzyl alcohol; benzyl ether; Lewis acid

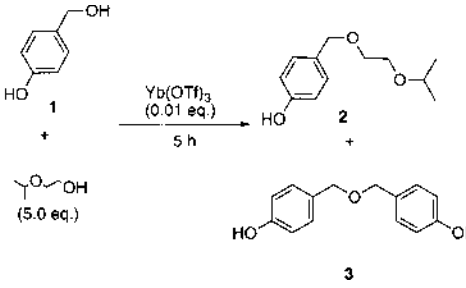
Etherification by direct condensation of alcohols is one of the most used method.[1)] This reaction is commonly achieved by use of small amounts of an organic or inorganic acid.[1)] On the other hand, Lewis acid-catalyzed etherification by direct condensation has also been reported.[2)] In most cases, these reactions required stoichiometric amounts of catalyst, because the Lewis acid was decomposed by water which was formed as a co-product in the condensation of alcohols. Kim *et al.* reported that etherification by direct condensation of alcohols proceeded by using a catalytic amount of zinc chloride as the Lewis acid; however, catalytic etherification was limited to activated benzyl alcohols.[2a)] Quite recently, Maruoka and co-workers reported that a small amount of MeAl(NTf$_2$)$_2$, as a strong Lewis acid, catalyzed etherification by direct condensation.[2d)]

It is known that rare earth metal trifluoromethanesulfonates [rare earth metal triflate, RE(OTf)$_3$] are stable in water and therefore do not decompose under aqueous work-up conditions, which are different from conventional Lewis acids. In this decade, there have been many reports that RE(OTf)$_3$ as a Lewis acid catalyst has been successfully applied to several synthetic reactions.[3)] Recently, we have developed RE(OTf)$_3$ catalyzed Friedel–Crafts acylation and alkylation; these reactions proceed smoothly in the presence of small amounts of RE(OTf)$_3$.[4)] It has also been demonstrated that the catalyst can be easily recovered by aqueous work-up from the reaction mixture and reused without decrease in its catalytic activity. From these reports, we expected that RE(OTf)$_3$ would work as a Lewis acid efficiently even in the presence of water as the co-product in condensation of alcohols. Very recently, Sharma *et al.* examined ytterbium (III) trifluoromethanesulfonates [ytterbium (III) triflate; Yb(OTf)$_3$] catalyzed benzyl-etherification by condensation of alcohols as described in preliminary report independently from us.[5)] Therefore, we would like to report the etherification of benzyl alcohols fully employing the characteristic features of RE(OTf)$_3$ efficiently.[6)]

## Results and Discussion

In the first place, several reaction conditions were examined in the reaction of 4-hydroxybenzyl alcohol (**1**) and 2-isopropoxyethanol with catalytic amounts of Yb(OTf)$_3$.[7)] The results are summarized in Table 1. Acetonitrile, dichloromethane, toluene, *N*,*N*-dimethylformamide (DMF), or 1,3-dimethyl-2-imidazolidinone (DMI) was used as a solvent. In each set of conditions, the reaction proceeded and 4-[(2-isopropoxyethoxy)methyl]phenol (**2**) and bis(4-hydroxybenzyl) ether (**3**) were obtained. It should be noted that only a 0.01 molar amount of Yb(OTf)$_3$ was required as catalyst in this reaction. The yields of **2** were increased in accordance with the amount of 2-isopropoxyethanol. When a 1.0 molar amount of 2-isopropoxyethanol was used, the yield of **2** was 34% (entry 1). On the other hand, when a 5.0 molar amount of 2-isopropoxyethanol was used, the yield of **2** was improved to 81% (entry 4). Reaction temperature also affected

Table 1. Yb(OTf)$_3$ Catalyzed Etherification of **1**



| Entry | Solvent | Temp./°C | Yield/%[a)] | |
|---|---|---|---|---|
| | | | **2** | **3** |
| 1[b)] | MeCN | 80 | 34 | 5 |
| 2[c)] | MeCN | 80 | 72 | 6 |
| 3 | MeCN | 50 | 68 | 20 |
| 4 | MeCN | 80 | 81 | 2 |
| 5 | CH$_2$Cl$_2$ | Reflux | 70 | 19 |
| 6 | Toluene | 50 | 68 | 3 |
| 7 | Toluene | 80 | 82 | 7 |
| 8 | Toluene | Reflux | 7 | Trace |
| 9 | DMF | 50 | 1 | Trace |
| 10 | DMF | 100 | 42 | 16 |
| 11 | DMI[d)] | 50 | 11 | 5 |
| 12 | DMI[d)] | 100 | 85 | 1 |

*a*) Yields were determined by HPLC using the internal standard method. *b*) 1.0 molar amount of 2-isopropoxyethanol was used. *c*) 3.0 molar amounts of 2-isopropoxyethanol was used. *d*) 1,3-Dimethyl-2-imidazolidinone.

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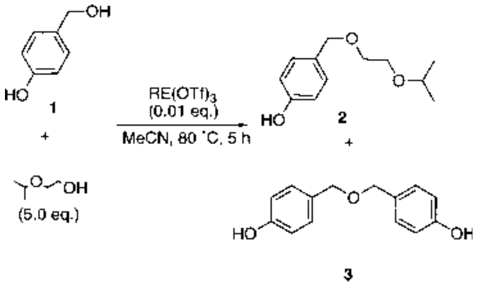
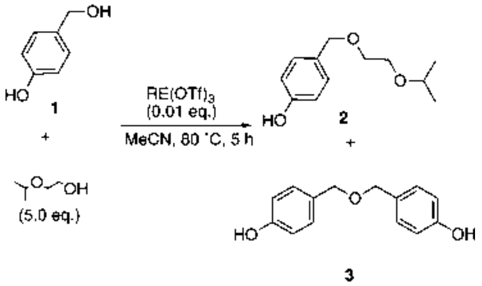
Table 2. Effect of Rare Earth Metal Triflate



| RE | Yield/%[a)] | | RE | Yield/%[a)] | |
|---|---|---|---|---|---|
| | **2** | **3** | | **2** | **3** |
| Sc | 61 | 9 | Gd | 78 | 4 |
| Y | 79 | 6 | Dy | 79 | 3 |
| La | 75 | 9 | Ho | 75 | 3 |
| Pr | 80 | 9 | Er | 80 | 6 |
| Nd | 77 | 12 | Tm | 65 | 12 |
| Sm | 74 | 5 | Yb | 81 | 2 |
| Eu | 85 | 8 | Lu | 76 | 9 |

*a*) Yields were determined by HPLC using the internal standard method.

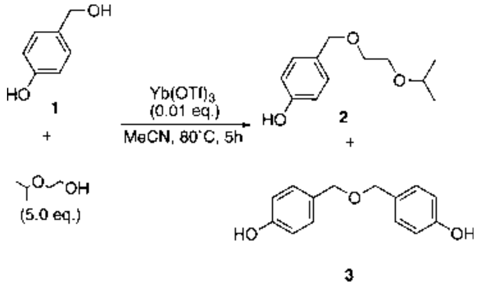
Table 3. Catalytic Activity Of Yb(OTf)$_3$ and Sc(OTf)$_3$



| Entry | Time | RE | Yield/%[a)] | |
|---|---|---|---|---|
| | | | **2** | **3** |
| 1 | 5 min | Yb | 18 | 8 |
| 2 | 5 min | Sc | 78 | 5 |
| 3 | 10 min | Yb | 37 | 15 |
| 4 | 10 min | Sc | 76 | Trace |
| 5 | 30 min | Yb | 65 | 16 |
| 6 | 1 h | Yb | 61 | 14 |
| 7 | 2 h | Yb | 79 | 8 |
| 8 | 5 h | Yb | 81 | 2 |
| 9 | 5 h | Sc | 61 | 9 |

*a*) Yields were determined by HPLC using the internal standard method.

Table 4. Reuse of Yb(OTf)$_3$ in Etherification in **1**



| No. of times used | Yield/%[a)] | | Recovery of Yb(OTf)$_3$/% |
|---|---|---|---|
| | **2** | **3** | |
| 1 | 85 | 4 | 72 |
| 2 | 84 | 4 | 70 |
| 3 | 81 | 5 | 70 |

*a*) Yields were determined by HPLC using the internal standard method.

the yield. For example, while the reaction of **1** and 2-isopropoxyethanol in toluene at 80 °C gave 82% of **2** (entry 7), the reaction under reflux condition gave only 7% (entry 8). The result of gel permeation chromatography (GPC) analysis of the reaction mixture in entry 9 showed that a polymerization product was formed. We presumed that **1** or **2** was polymerized at high temperature and the yield of **2** was decreased.

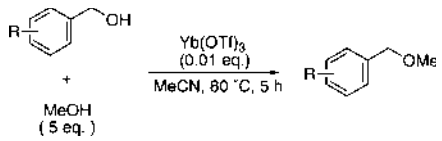
Other RE(OTf)$_3$ were then examined as a catalyst in the reaction of **1** with 2-isopropoxyethanol (Table 2). Each RE(OTf)$_3$ was prepared by the reaction of the corresponding rare earth metal oxide with trifluoromethanesulfonic acid in an aqueous solution.[7)] All the RE(OTf)$_3$ listed were effective catalysts in the reaction of **1** and 2-isopropoxyethanol; **2** was obtained in moderate yield.

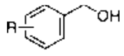
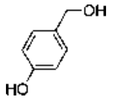
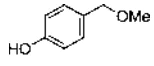
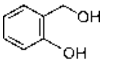
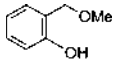
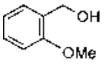
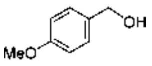
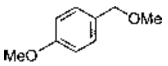
The difference of catalytic activity between Yb(OTf)$_3$ and scandium (III) trifluoromethanesulfonate [scandium (III) triflate; Sc(OTf)$_3$] is clearly shown in Table 3. This table shows that Sc(OTf)$_3$ is a more active catalyst than Yb(OTf)$_3$. For example, while the Sc(OTf)$_3$-catalyzed reaction in 5 min yielded 78% of **2** (entry 2), Yb(OTf)$_3$ catalyzed reaction gave only 18% (entry 1). On the other hand, the yields of the Yb(OTf)$_3$ catalyzed reaction increased in accordance with the extension of reaction time; the reaction in 5 h gave 81% of **2** (entry 8). However, the yields of the Sc(OTf)$_3$ catalyzed reaction decreased in accordance with the extension of reaction time; the reaction of 5 h gave only 61% of **2** (entry 9). The result of GPC analysis of the reaction mixture in entry 9 showed that a polymerization product was formed. These results showed that **2** was polymerized by Sc(OTf)$_3$ because of its strong Lewis acidity,[8)] thus the yield of **2** was decreased.

Reusability of the catalyst is one of the main advantages of using RE(OTf)$_3$. RE(OTf)$_3$ could be easily recovered from the reaction mixture by simple extraction and removing water, which could be reused without further purification (see Experimental). Efficiency of the recovery and catalytic activity of reused Yb(OTf)$_3$ were examined in the reaction of **1** with 2-isopropoxyethanol using Yb(OTf)$_3$ as a representative RE(OTf)$_3$. As shown in Table 4, the yields of etherification product **2** in the second and third uses were almost the same as those in the first use. However, the recovery of Yb(OTf)$_3$ was only 70%. We presumed that the catalyst was soluble in the organic layer. The organic layer contained ethyl acetate, 2-isopropoxyethanol, and water, because 2-isopropoxyethanol is highly soluble in ethyl acetate and water. Therefore, a part of the catalyst was dissolved in the organic layer, and the amount of the recovered catalyst decreased.

The reactivity of several kinds of benzyl alcohols was examined in RE(OTf)$_3$ catalyzed etherification. The results are shown in Table 5. Although the etherification of benzyl alcohol did not occur, introduction of a hydroxy or alkoxy group to the benzene ring, such as hydroxybenzyl alcohol or methoxybezyl alcohol, was effective for promoting the reaction. For example, **1** reacted with methanol smoothly to

Table 5. $Yb(OTf)_3$ Catalyzed Etherification of Benzylalcohols and Methanol



| Entry |  | Product | Yield/%[a] |
|---|---|---|---|
| 1 |  **1** |  **4** | 85 |
| 2 |  **5** |  **6** | 27 |
| 3 |  **7** | ND[b] | |
| 4 |  **8** |  **9** | 37 |

[a] Isolated yield. [b] Not detected.

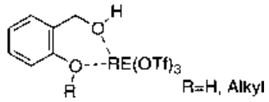
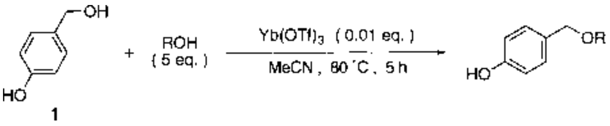
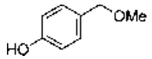
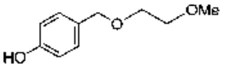
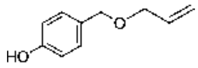
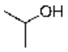
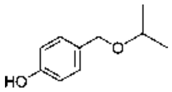
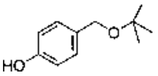
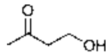
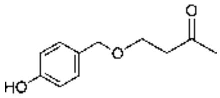


Chart 1. A Possible Stable Complex of *ortho*-Substituted Benzyl Alcohols

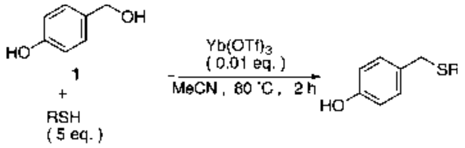
Table 6. $Yb(OTf)_3$ Catalyzed Etherification of **1** with Various Alcohols

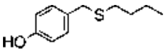
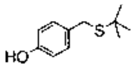


| Entry | ROH | Product | Yield/%[a] |
|---|---|---|---|
| 1 | MeOH |  **4** | 85 |
| 2 |  |  **10** | 65 |
| 3 |  |  **11** | 70 |
| 4 |  |  **12** | 62 |
| 5 | *tert*-BuOH |  **13** | 15 |
| 6 |  |  **14** | 64 |

[a] Isolated yield.

Table 7. $Yb(OTf)_3$ Catalyzed Reaction of **1** with Thiols



| Entry | RSH | Product | Yield (%)[a] |
|---|---|---|---|
| 1 | *n*-BuSH |  **15** | 93 |
| 2 | *tert*-BuSH |  **16** | 89 |

[a] Isolated yield.

give 4-(methoxymethyl)phenol (**4**) in good yield (entry 1). This table also shows that $Yb(OTf)_3$ catalyzed etherification is influenced strongly by the position of a hydroxy or alkoxy group on the benzene ring. For example, the etherification of 2-hydroxybenzyl alcohol (**5**) gave a low yield of 2-(methoxymethyl)phenol (entry 2). As for the reaction of methoxybenzyl alcohols, while 4-methoxybenzyl alcohol (**8**) reacted to give 1-methoxy-4-(methoxymethyl)benzene (**9**), 2-methoxybenzyl alcohol (**7**) did not react (entries 3, 4). From these results, we assume that **5** or **7** would be stabilized by bidentate chelation of $Yb(OTf)_3$ shown in Chart 1.

The results of the $Yb(OTf)_3$ catalyzed etherification with **1** and various aliphatic alcohols are summarized in Table 6. Although the etherification with methanol proceeded smoothly, the reaction with isopropyl alcohol or 1,1-dimethylethanol gave lower yield product (entries 1, 4, 5). For example, methanol gave 85% yield of 4-(methoxymethyl)phenol, but 1,1-dimethylethanol gave only 15% yield of 4-(*tert*-butoxymethyl)phenol (**13**). These results show that a sterically hindered aliphatic alcohol influences the yield of the product. Etherification using an allyl alcohol or a keto alcohol also proceeded and gave the corresponding product in moderate yield (entries 3, 6).

$RE(OTf)_3$ was also found to be an excellent catalyst in condensation between benzyl alcohols and thiols.[9] The results are summarized in Table 7. Butane-1-thiol or 1,1-dimethylethane-1-thiol was used as thiols. As might be expected, both reactions proceeded quite smoothly, and the corresponding thio ethers were obtained quantitatively.

## Conclusions

We have demonstrated the $RE(OTf)_3$ catalyzed etherification of hydroxybenzyl alcohols and aliphatic alcohols by direct condensation, which was found to be have specific features as follows.

1) The reaction proceeds smoothly using a catalytic amount of $RE(OTf)_3$.
2) $Sc(OTf)_3$ is the most active catalyst among the $RE(OTf)_3$.
3) $RE(OTf)_3$ is easily recovered and reused without decrease in its catalytic activity.

4) RE(OTf)$_3$ is also effective in thio etherification.

## Experimental

Melting points were measured with a Yanagimoto micro melting point apparatus. IR spectra were recorded on a JASCO FT/IR-350. $^1$H-NMR spectra [tetramethylsilane (TMS) as an internal standard] were recorded on a Varian VXR-500 spectrometer. Chemical shifts are given in ppm. Mass spectra were measured on a VG-70SE spectrometer. Elemental analyses were carried out using a Yanaco MT-5. For thin layer chromatography (TLC) analyses throughout this work, Merck precoated TLC plates (silica-gel 60 F$_{254}$) were used. HPLC analysis for the determination of yield was carried out on a SHIMADZU LC-6A and an UV detector of 254 nm. GPC analysis was carried out on a TOSOH HLC-8020. Silica-gel column chromatography was performed using Merck Kieselgel 60 (70—230 mesh).

All of the RE(OTf)$_3$ were prepared from the corresponding rare earth metal oxide (Soekawa Chemicals Co., Ltd.) and trifluoromethanesulfonic acid (Kanto Chemicals Co., Ltd.) in water according to the literature.[7] Reagents used in this work were commercially available.

**A Typical Experimental Procedure for the RE(OTf)$_3$ Catalyzed Benzyl-Etherification** Typical experimental procedure is described for the reaction of 4-hydroxybenzyl alcohol with 2-isopropoxyethanol; Yb(OTf)$_3$ was prepared from the ytterbium (III) oxide (Yb$_2$O$_3$) and trifluoromethanesulfonic acid.[7] A mixture of Yb(OTf)$_3$ (124 mg, 0.2 mmol), 2-isopropoxyethanol (11.6 ml, 100 mmol) and 4-hydroxybenzyl alcohol (2.48 g, 20 mmol) in acetonitrile (20 ml) was stirred at 80 °C for 5 h. After dilution with water, the mixture was extracted with ethyl acetate. After removal of the solvents, the crude products were purified by column chromatography on silica-gel using ether–dichloromethane (1 : 10) as eluent to afford 4-[(2-isopropoxyethoxy)methyl]phenol (**2**) (85% yield) and bis(4-hydroxybenzyl) ether (**3**) (4% yield). The aqueous layer was concentrated *in vacuo* to give a crystalline residue, which was finally heated at 180 °C for 48 h *in vacuo* to afford 89 mg (72%) of Yb(OTf)$_3$ as colorless crystals. The recovered Yb(OTf)$_3$ was reused in the next reaction.

4-[(2-Isopropoxyethoxy)methyl]phenol (**2**): Yellow oil. IR (KBr) 3300, 1620, 1520, 1220, 1080 cm$^{-1}$; $^1$H-NMR (CDCl$_3$) $\delta$: 1.18 (6H, d, $J$=6.0 Hz), 3.58—3.67 (5H, m), 4.47 (2H, s), 5.70—5.75 (1H, br s), 6.74 (2H, d, $J$=8.5 Hz), 7.16 (2H, d, $J$=8.5 Hz). High resolution FAB-MS. Found: $m/z$ 210.1255 [M]$^+$. Calcd for $C_{12}H_{18}O_3$: 210.1270.

Bis(4-hydroxybenzyl) Ether (**3**)[10]: mp 84—85 °C (ether–$n$-hexane) (lit. 63—65 °C); IR (KBr) 3200, 1620, 1520, 1250, 1060 cm$^{-1}$; $^1$H-NMR (CD$_3$OD) $\delta$: 4.39 (4H, s), 4.87 (2H, s), 6.75 (4H, d, $J$=8.5 Hz), 7.15 (4H, d, $J$=8.5 Hz), FAB-MS (positive ion mode) $m/z$: 230 [M]$^+$. *Anal.* Calcd for $C_{14}H_{14}O_3 \cdot 1/2H_2O$: C, 70.27; H, 6.31%. Found: C, 70.39; H, 6.15%.

4-(Methoxymethyl)phenol (**4**)[10]: mp 82—83 °C (ether–$n$-hexane) (lit. 81—83 °C); IR (KBr) 3240, 1620, 1520, 1070 cm$^{-1}$; $^1$H-NMR (CDCl$_3$) $\delta$: 3.37 (3H, s), 4.39 (2H, s), 5.37 (1H, s), 6.77 (2H, d, $J$=8.5 Hz), 7.16 (2H, d, $J$=8.5 Hz). FAB-MS (positive ion mode) $m/z$: 138 [M]$^+$. *Anal.* Calcd for $C_8H_{10}O_2$: C, 69.54; H, 7.29%. Found: C, 69.62; H, 7.35%.

2-(Methoxymethyl)phenol (**6**)[11]: Colorless oil. IR (CHCl$_3$) 3400, 3000, 2920, 1600, 1490, 1240, 1080 cm$^{-1}$; $^1$H-NMR (CDCl$_3$) $\delta$: 3.45 (3H, s), 4.67 (2H, s), 6.85 (1H, dt, $J$=7.5, 1.5 Hz), 6.90 (1H, dd, $J$=7.5, 1.5 Hz), 7.02 (1H, dd, $J$=7.5, 1.5 Hz), 7.22 (1H, dt, $J$=7.5, 1.5 Hz), 7.45 (1H, br s). FAB-MS (positive ion mode) $m/z$: 138 [M]$^+$.

1-Methoxy-4-(methoxymethyl)benzene (**9**)[12]: Colorless oil. IR (CHCl$_3$) 3000, 2920, 2840, 1620, 1520, 1240, 1080 cm$^{-1}$; $^1$H-NMR (CDCl$_3$) $\delta$: 3.35 (3H, s), 3.80 (3H, s), 4.39 (2H, s), 6.88 (2H, d, $J$=8.5 Hz), 7.26 (2H, d, $J$=8.5 Hz). *Anal.* Calcd for $C_9H_{12}O_2$: C, 71.02; H, 7.94%. Found: C, 71.03; H, 7.85%.

4-(2-Methoxyethoxymethyl)phenol (**10**)[13]: Yellow oil. IR (KBr) 3300, 1610, 1510, 1240, 1080 cm$^{-1}$; $^1$H-NMR (CDCl$_3$) $\delta$: 3.39 (3H, s), 3.60 (4H, s), 4.47 (2H, s), 5.74 (1H, br s), 6.74 (2H, d, $J$=8.5 Hz), 7.16 (2H, d, $J$=8.5 Hz). FAB-MS (positive ion mode) $m/z$: 182 [M]$^+$. *Anal.* Calcd for $C_{10}H_{14}O_3 \cdot 1/5H_2O$: C, 64.63; H, 7.81%. Found: C, 64.80; H, 7.64%.

(4-Allyloxymethyl)phenol (**11**): Colorless oil. IR (KBr) 3300, 1610, 1510, 1240, 1080 cm$^{-1}$; $^1$H-NMR (CDCl$_3$) $\delta$: 4.02 (2H, ddd, $J$=5.5, 2.0, 1.0 Hz), 4.45 (2H, s), 5.21 (1H, ddd, $J$=10.0, 3.0, 1.0 Hz), 5.30 (1H, ddd, $J$=17.0, 3.0, 2.0 Hz), 5.59 (1H, br s), 5.95 (1H, ddt, $J$=17.0, 10.0, 5.5 Hz), 6.76 (2H, d, $J$=8.5 Hz), 7.16 (2H, d, $J$=8.5 Hz). FAB-MS (positive ion mode) $m/z$: 164 [M]$^+$. *Anal.* Calcd for $C_{10}H_{12}O_2 \cdot 1/10H_2O$: C, 72.35; H, 7.40%. Found: C, 72.14; H, 7.37%.

(4-Isopropoxymethyl)phenol (**12**): mp 61.5—62.5 °C (ether–$n$-hexane); IR (KBr) 3200, 1620, 1520, 1040 cm$^{-1}$; $^1$H-NMR (CDCl$_3$) $\delta$: 1.12 (6H, d, $J$=6.0 Hz), 3.69 (1H, sept, $J$=6.0 Hz), 4.43 (2H, s), 5.23 (1H, s), 6.75 (2H, d, $J$=8.5 Hz), 7.16 (2H, d, $J$=8.5 Hz). FAB-MS (positive ion mode) $m/z$: 166 [M]$^+$. *Anal.* Calcd for $C_{10}H_{14}O_2$: C, 72.26; H, 8.48%. Found: C, 72.40; H, 8.50%.

4-(*tert*-Butoxymethyl)phenol (**13**): mp 62—65 °C (ether–$n$-hexane); IR (KBr) 3280, 1620, 1520, 1250, 1040 cm$^{-1}$; $^1$H-NMR (CDCl$_3$) $\delta$: 1.29 (9H, s), 4.36 (2H, s), 5.12 (1H, br s), 6.72 (2H, d, $J$=8.5 Hz), 7.16 (2H, d, $J$=8.5 Hz). FAB-MS (positive ion mode) $m/z$: 180 [M]$^+$. High resolution FAB-MS. Found: $m/z$ 180.1186 [M]$^+$. Calcd for $C_{11}H_{16}O_2$: 180.1150.

4-Hydroxybenzyloxy-2-butanone (**14**): Colorless oil. IR (neat) 3320, 2880, 1700, 1620, 1520, 1230 cm$^{-1}$; $^1$H-NMR (CDCl$_3$) $\delta$: 2.18 (3H, s), 2.71 (2H, t, $J$=6.5 Hz), 3.71 (2H, t, $J$=6.5 Hz), 4.42 (2H, s), 5.06 (1H, br s), 6.76 (2H, d, $J$=8.5 Hz), 7.17 (2H, d, $J$=8.5 Hz). FAB-MS (positive ion mode) $m/z$: 194 [M]$^+$.

4-(Butylthiomethyl)phenol (**15**): Yellow oil. IR (KBr) 3360, 1620, 1520 cm$^{-1}$; $^1$H-NMR (CDCl$_3$) $\delta$: 0.88 (3H, t, $J$=15.0 Hz), 1.37 (2H, sext, $J$=7.5 Hz), 1.54 (2H, quint, $J$=7.5 Hz), 2.41 (2H, t, $J$=15.0 Hz), 3.65 (2H, s), 4.86 (1H, br s), 6.77 (2H, d, $J$=8.5 Hz), 7.16 (2H, d, $J$=8.5 Hz). FAB-MS (positive ion mode) $m/z$: 197 [M+1]$^+$. High resolution FAB-MS. Found: $m/z$ 197.1003 [M+1]$^+$. Calcd for $C_{11}H_{16}OS$: 197.1000.

4-(*tert*-Butylthiomethyl)phenol (**16**): mp 94.5—95.5 °C (ether–$n$-hexane); IR (KBr) 3200, 1520 cm$^{-1}$; $^1$H-NMR (CDCl$_3$) $\delta$: 1.34 (9H, s), 3.71 (2H, s), 4.76 (1H, br s), 6.75 (2H, d, $J$=8.5 Hz), 7.2 (2H, d, $J$=8.5 Hz). FAB-MS (positive ion mode) $m/z$: 197 [M+1]$^+$. High resolution FAB-MS. Found: $m/z$ 197.1003 [M+1]$^+$. Calcd for $C_{11}H_{16}OS$: 197.1000.

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