CHAPTER II

SYNTHESIS OF HOMOALLYLIC ALCOHOLS USING TETRABUTYL AMMONIUM BI-BENZOATE AS CATALYST
2.1. Introduction

The allylation reaction represents one of the most useful methods for the preparation of homoallylic alcohols.\textsuperscript{1,2} Homoallylic alcohols are useful intermediate for the construction of complex molecules\textsuperscript{3,4} and can be easily converted to many important building blocks of natural products.\textsuperscript{4-7} They have been prepared by a several methods, in particular by allylation of carbonyl compounds with allytrialkyl and allyltrialkyl stannane.\textsuperscript{5-8} Metal Lewis acids or transition metal complexes were most commonly used to catalyzes this type reaction.\textsuperscript{9-22} In recent years allyl boranes\textsuperscript{23}, and allylsilanes were also used as efficient reagent to achieve homoallylic product by activation of the carbonyl group with the aid of Lewis acid.\textsuperscript{24} However Lewis acid containing boron,\textsuperscript{9} aluminium, titanium\textsuperscript{12} and tin\textsuperscript{25} are extremely moisture sensitive and reactions are carried out under anhydrous conditions. In some reactions, Lewis acid promoters have also to be used.\textsuperscript{26} Moreover, in some cases, even a small amount of water appears distracting as it reacts instantly with the reagent compared to other substrate.\textsuperscript{21,27} Although several Lewis acid or Bronsted acid catalyzed allylation reaction in mixture of organic and aqueous media
with allyltributyltin have been reported. Some of the catalysts are expensive,
\textsuperscript{13,28,29} some are strongly corrosive\textsuperscript{30,31} and some are toxic.\textsuperscript{32,33}

In the recent past, several reports utilizing various metal complexes as catalyst have been published in the literature for this type of reaction.\textsuperscript{34-43}

### 2.2. Review of Literature

Over last decades, there are various catalysts have been addressed to allylation reaction. In recent years, several Lewis acids or Bronsted acid catalyzed allylation reaction. However, methods using Lewis acid must be carried out under strictly anhydrous condition. Zhao et al. reported a new method for homoallylic alcohol transformation using a polymer-supported sulfonamide of N-glycine as catalyst\textsuperscript{44} by the treatment of aldehyde with allyltributyl tin. Though the reaction produced good yield, but the method required long reaction time. Moreover, the catalyst is not available commercially and requires a multi-step procedure for its preparation.

Another group led by Kobayashi\textsuperscript{46} developed polymer-supported formamides (Fig. 2.1) organocatalysts for the allylation of aldehydes with allyl trichloro silane.

![PS-Formamide 1](attachment:ps-formamide.png)

![PS-Formamide 1](attachment:ps-formamide.png)

**Fig. 2.1**
Massa et al.\textsuperscript{47} reported the allylation of aldehydes with allyltrichlorosilane in the presence of sulfoxides. However, the reaction condition is low temperature and longer reaction time also afforded moderate to good yield.

\[
\begin{align*}
\text{R}^1 = \text{Ph} & \quad \text{R}^2 = \text{p-tolyl} \\
\end{align*}
\]

\textbf{Scheme. 2.0}

He Tian and his coworkers\textsuperscript{45} reported the allylation of aldehydes and imines carried out by treatment of allyltributyl stannane in the presence of ZrOCl\textsubscript{2}·8H\textsubscript{2}O. Recently there was a report of carrying out alkylation reactions in ionic liquids\textsuperscript{48} and polyethylene glycol (PEG)\textsuperscript{49}, especially the imidazolium ones with PF\textsubscript{6} and BF\textsubscript{4} counter ions, have been shown to have serious drawbacks.\textsuperscript{50,51} The high cost and disposability of these solvents also limit their usefulness. Ruma Rao and his co-workers\textsuperscript{52} reported allylation in aqueous medium catalyzed by β-cyclodextrin in the presence of HCl without any metal catalyst to afford the corresponding homo allylic alcohols in good yields.

Andrews et al.\textsuperscript{53} reported the alkylation of various carbonyl compounds to homoallylic alcohols under ultrasonic irradiation and solvent free conditions in the presence of metallic Sn and excess allyl bromides. Another group led by Phukan\textsuperscript{54} developed cuprous iodide, an efficient catalyst for alkylation in DMF to afford the homoallylic alcohols in good yields. Recently Phukan et
al.\textsuperscript{55} developed reusable catalyst titanium exchanged ZSM-5 for allylation reaction of aldehyde with allyltributyl stannane in toluene.

Although rhenium complex\textsuperscript{56} or scandium triflate are very effective as air-stable and water-tolerant catalysts for allylation reaction, these catalysts are expensive and many cases method required only at higher temperature. Several Pd-complexes\textsuperscript{57-59} and silver triflate\textsuperscript{13,60-63} have been reported by many researchers. However, Palladium and silver compounds are expensive and moisture sensitivity.

Shi and his co-workers\textsuperscript{64} reported bis(NHC)-Pd(II) (Fig. 2.3) complexes were effective in the allylation of aldehydes with allyltributanetin to give the products in good yields in most cases at room temperature. This method required a relatively long reaction time to achieve good yields.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{bis_nhc_pd_ii_complexes.png}
\caption{Bis(NHC)-Pd(II) complexes 1-3}
\end{figure}

Cerium (III) chloride (CeCl$_3$.7H$_2$O) in addition with NaI has been used as a stoichiometric promoter for the allylation of aldehydes with
allyltributylstannane. The drawback of this method is the use of a stoichiometric amount of the promoter to effect the reaction.

Although there are number of catalysts available in the literature for the synthesis of homoallylic alcohols, several of them have limitations. So, it is important to develop new catalyst for the synthesis of these compounds.

Tetrabutyl ammonium bi benzoate (TBABB) is a typical nucleophilic catalyst used for polymerization reaction, particularly for group transfer polymerization (GTP). Sivaram, developed a method for Mukaiyama aldol reaction using TBABB as the nucleophilic catalyst. Structure of the catalyst as determined by Sivaram et al. using single crystal X-ray crystallography is shown in figure 2.4.

![Structure of TBABB](image)

Fig. 2.4. Structure of TBABB
2.3. Present Work

Objective

During past few years, there are a variety of reactions systems have been developed for the allylation reactions. Many of these methods have disadvantages such as toxic solvents, high temperature, longer reaction times, use of expensive and moisture sensitive catalysts. However, there are very few reports in the literature for homoallylation reaction using organocatalyst. Catalytic application of tetrabutylammonium bi-benzoate (TBABB) is very limited in the literature. The objective of the present investigation is to study the catalytic efficiency of TBABB for homoallylic alcohol synthesis by reacting aldehydes with allyl tributyl stannane. (Scheme 2.1).

\[
\text{RCHO} + \text{SnBu}_3\text{C} = \text{OH} \\
\text{TBABB} \quad \text{THF, rt}
\]

Scheme 2.1
2.4 Results and Discussion

The catalyst, TBABB was synthesized by using a procedure reported in the literature.\textsuperscript{69} Initially, a systematic study on the synthesis of homoallylic alcohols was carried out using 4-chlorobenzaldehyde as substrate. In this reaction, allyl tri(n-butyl) tin (1 mmol) was added to a mixture of 4-chlorobenzaldehyde (1 mmol) and tetrabutyl ammonium bi-benzoate (10 mol\%) in dichloromethane at room temperature (\textit{Table 2.1}). Reaction at room temperature in DCM failed to yield the desired product after 24 h of the reaction. A comparative study was also carried out for evaluation of the catalyst using different solvents such as tetrahydrofuran, dimethylformamide. The reaction took relatively longer time to occur in dimethylformamide. Tetrahydrofuran gave the best results. The reaction was further examined in presence of different amount of catalyst (10\%, 15\%, 20\%). The yield generally increased with increasing concentration of the catalyst. However, further increase of the molar concentration of the catalyst did not significantly increase the yield of the product.

\textbf{Table 2.1. Synthesis of homoallylic alcohols from 4-chlorobenzaldehyde under various conditions.}\textsuperscript{a}

<table>
<thead>
<tr>
<th>Entry</th>
<th>TBABB (mol%)</th>
<th>Solvent</th>
<th>Temp.</th>
<th>Time (h)</th>
<th>Yield\textsuperscript{b} (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>10</td>
<td>DCM</td>
<td>RT</td>
<td>24</td>
<td>0</td>
</tr>
<tr>
<td>2.</td>
<td>10</td>
<td>DMF</td>
<td>RT</td>
<td>12</td>
<td>55</td>
</tr>
<tr>
<td>3.</td>
<td>10</td>
<td>THF</td>
<td>RT</td>
<td>8</td>
<td>52</td>
</tr>
<tr>
<td>4.</td>
<td>15</td>
<td>THF</td>
<td>RT</td>
<td>5</td>
<td>62</td>
</tr>
<tr>
<td>5.</td>
<td>20</td>
<td>THF</td>
<td>RT</td>
<td>5</td>
<td>64</td>
</tr>
<tr>
<td>6.</td>
<td>15</td>
<td>THF</td>
<td>Reflux</td>
<td>5</td>
<td>59</td>
</tr>
</tbody>
</table>

\textsuperscript{a} Reaction conditions: 4-Chlorobenzaldehyde (1 mmol), allyltributyltin (1 mmol), solvent (1 ml), 25\textdegree C. \textsuperscript{b} Isolated yield after chromatographic purification.
After optimizing the reaction conditions we extended the procedure using different aldehydes, the results are summarized in Table 2.2.

Table 2.2. Synthesis of homoallylation alcohols using the TBABB/THF system

<table>
<thead>
<tr>
<th>Entry</th>
<th>Aldehyde</th>
<th>Time (h)</th>
<th>Product</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td><img src="image" alt="Benzenaldehyde" /></td>
<td>6</td>
<td><img src="image" alt="Benzyl allylic alcohol" /></td>
<td>60</td>
</tr>
<tr>
<td>2.</td>
<td><img src="image" alt="Chlorobenzaldehyde" /></td>
<td>5</td>
<td><img src="image" alt="Chlorinated benzyl allylic alcohol" /></td>
<td>62</td>
</tr>
<tr>
<td>3.</td>
<td><img src="image" alt="Nitrobenzaldehyde" /></td>
<td>7</td>
<td><img src="image" alt="Nitrobenzyl allylic alcohol" /></td>
<td>59</td>
</tr>
<tr>
<td>4.</td>
<td><img src="image" alt="Nitrobenzaldehyde" /></td>
<td>9</td>
<td><img src="image" alt="Nitrobenzyl allylic alcohol" /></td>
<td>45</td>
</tr>
<tr>
<td>5.</td>
<td><img src="image" alt="Methoxybenzaldehyde" /></td>
<td>10</td>
<td><img src="image" alt="Methoxybenzyl allylic alcohol" /></td>
<td>53</td>
</tr>
<tr>
<td>6.</td>
<td><img src="image" alt="Methylbenzaldehyde" /></td>
<td>9</td>
<td><img src="image" alt="Methylbenzyl allylic alcohol" /></td>
<td>55</td>
</tr>
</tbody>
</table>
Reactions were carried out using 1 mmol of aldehyde, 1 mmol of allyltributyl stannane and 15 mol% of TBABB in a 1 mL of THF. The reaction mixture was stirred using a magnetic stirring bar at room temperature for the appropriate time as indicated by TLC. After completion of the reaction followed by work up the crude product was purified by column
chromatography. The products were characterized by comparing the IR and NMR spectroscopic data with those reported in the literature.

Aromatic aldehyde containing nitro (-NO_2) group and methoxy groups (-OMe) gave lower yield (Entry 3 and 5), while higher isolated yield was obtained for the reaction of aldehyde having chloro (-Cl) group. The catalyst was quite effective in the case of sterically congested reactions (Entry 4 and 10). Under similar conditions naphthaldehyde also passed by allylation to the homoallylic alcohol in 61% (Entry 8). For aliphatic aldehyde, octanal (Entry 12), the yield of the allylated product was markedly decreased.

Products were characterized using NMR and IR spectroscopic analysis. In general the IR spectrum shows a broad band at around 3400 cm\(^{-1}\) which indicate the presence of OH functionality. A band at around 1600 cm\(^{-1}\) appears due to aromatic CH stretching vibrations. In \(^1\)H NMR spectrum, the multiplet in the chemical shift range 5.058-5.10 is due to the terminal CH\(_2\) of the double bond. The multiplet nature probably due to the low resolution of the spectra(300MHz) NMR instrument. CH proton of the double bond comes at about 5.8 ppm as a multiplet. The CH protons attached to the OH group shows a multiplet at the chemical shift range 4.58-4.62. The OH proton signal sometime appears at a variable chemical shift range around 3 ppm as abroad signal. The multiplet in the range around 2.39-2.54 ppm is due to the allylic CH\(_2\) group. The aromatic proton comes between 7-8 ppm. In \(^{13}\)C NMR spectrum, the olefinic CH carbon appears at about 128 ppm and the olefinic CH\(_2\) carbon at about 113 ppm. CH carbon attached to the OH group appears at about 67 ppm and the other CH\(_2\) carbon appears at about 38 ppm. The aromatic carbon comes between 120-140 ppm. Some representative NMR spectra are shown in figure 2.4-2.9.
Fig. 2.4: $^1$H NMR Spectra of 1-(4-Chlorophenyl) but-3-en-1-ol (Entry 2)

Fig. 2.5: $^{13}$C NMR Spectra of 1-(4-Chlorophenyl) but-3-en-1-ol (Entry 2)
Fig. 2.6: $^1$H NMR Spectra of 1- (4-fluorophenyl) but-3-en-l-ol (Entry 9).

Fig. 2.7: $^{13}$C NMR Spectra of 1- (4-fluorophenyl) but-3-en-l-ol (Entry 9)
Fig. 2.8: $^1$H NMR Spectra of 1-(2-bromophenyl) but-3-en-1-ol (Entry 11)

Fig. 2.9: $^{13}$C NMR Spectra of 1-(2-bromophenyl) but-3-en-1-ol (Entry 11)
2.5. Conclusion

Tetrabutyl ammonium bi-benzoate has been applied for the allylation of aldehydes with allyltributyl stannane to prepare homoallylic alcohols in good yields. In comparison to existing methodologies using Lewis acid and transition metal catalysts, this method produces lower yield of corresponding homoallylic alcohols.

2.6. Experimental Section

Synthesis of tetrabutyl ammonium bi-benzoate

Benzoic acid (2 g, 16 mmol) in 15.9 mL 10% aqueous tetra-n-butyl ammonium hydroxide at 27 °C. After 15 min stirring, the solution is extracted with 15 mL of dichloromethane. The dichloromethane solution is dried over anhydrous Na₂SO₄, filtered and stripped free of solvent. Residual solid was dissolved in 15 mL dry ether and allowed to stand for 12h. Long needles of bioxy anions appears.

Typical procedure for the TBABB catalysed allylation reaction.

To a mixture of aldehydes (1 mmol), allyltributyltin (1 mmol) in 1 mL THF in a 25 mL round bottom flask, TBABB (0.15 mmol) was added. The resulting mixture was stirred at room temperature for a specific period. The progress of the reaction was monitored by TLC. After completion of the reaction, diethyl ether (10 mL) was added and the solution was then filtered.
The filtrate was dried over anhydrous sodium sulphate (Na₂SO₄) followed by evaporation of solvent using a rotary evaporator under reduced pressure and concentrated to dryness gave the dried product. The crude product was purified by column chromatography over silica gel (230-400 mesh) using petroleum ether-ethylacetate mixture as eluent.

Experimental Data

Entry 1: 1-phenylbut-3-en-1-ol

\[
\begin{align*}
\text{IR (KBr, cm}^{-1}) : & \quad 3383, 3076, 3030, 2978, 2906, 1642, 1494, 1455, 1435, 1048, 916, 700 \\
\text{\textsuperscript{1}H NMR (300 MHz, CDCl\textsubscript{3}) :} & \quad \delta 212 - 2.47 (m, 2H), 4.52-4.63 (m, 1H), 5.15 - 5.27 (m, 2H), 5.71 - 5.87 (m, 1H), 7.12 - 7.23 (m, 2H), 7.22 - 7.42 (m, 2H). \\
\text{\textsuperscript{13}C NMR (75 MHz, CDCl\textsubscript{3}) :} & \quad \delta 41.3, 70.4, 117.2, 127.1, 128.3, 128.6, 131.8, 134.3, 141.1.
\end{align*}
\]
Entry 2: 1-(4-chlorophenyl) but-3-en-1-ol

\[
\begin{align*}
\text{IR(KBr, cm}^{-1}\text{):} & \quad 3390, 3076, 2925, 1679, 1644, 1597, 1054, 916 \\
\delta^1 \text{H NMR (300 MHz, CDCl}_3\text{):} & \quad \delta 2.40 - 2.54 (m, 2H), 4.71 - 4.75 (m, 1H), 5.14 - 5.19 (m, 2H), 5.72 - 5.86 (m, 1H), 7.26 - 7.42 (m, 4H) \\
\delta^{13} \text{C NMR (75 MHz, CDCl}_3\text{):} & \quad \delta 43.5, 72.5, 118.3, 127.1, 128.1, 128.2, 132.8, 133.8, 142.2.
\end{align*}
\]

Entry 3: 1 - (4-nitrophenyl) but-3-en-1-ol

\[
\begin{align*}
\text{IR(KBr, cm}^{-1}\text{):} & \quad 3416, 3078, 2911, 1641, 1604, 1591, 1347, 1055 \\
\delta^1 \text{H NMR (300 MHz, CDCl}_3\text{):} & \quad \delta 2.40 - 2.56 (m, 2H), 4.84 - 4.89 (m, 1H), 5.16 - 5.30 (m, 2H), 5.74 - 5.83 (m, 1H), 7.53 (d, J=8.7Hz,2H), 8.20 (d, J=8.7Hz,2H). \\
\delta^{13} \text{C NMR (75 MHz, CDCl}_3\text{):} & \quad \delta 42.4, 68.3, 120.3, 125.3, 128.5, 128.9, 131.1, 134.8, 140.3
\end{align*}
\]

58
Entry 4: 1-(2-nitrophenyl) but-3-en-1-ol

\[
\begin{align*}
\text{IR(KBr, cm}^{-1}\text{):} & \quad 3419, 3083, 2016, 1648, 1355, 1048 \\
^1\text{H NMR (300 MHz, CDCl}_3\text{):} & \quad \delta 1.62 (br, 1H) 2.37 - 2.47 (m, 2H), 5.19 - 5.34 (m, 3H), 5.83 - 5.97 (m, 1H), 7.41 - 7.46 (m, 1H), 7.63 - 7.68 (m, 1H), 7.85 (dd, J=7.8Hz, 2H) \\
^{13}\text{C NMR (75 MHz, CDCl}_3\text{):} & \quad \delta 43.7, 69.2, 120.1, 125.3, 128.9, 129.1, 134.3, 134.8, 140.1
\end{align*}
\]

Entry 5: 1 - (4-methoxy phenyl) but-3-en-1-ol

\[
\begin{align*}
\text{IR(KBr, cm}^{-1}\text{):} & \quad 3410, 3072, 3008, 2938, 2832, 1643, 1615, 1514, 1243 \\
^1\text{H NMR (300 MHz, CDCl}_3\text{):} & \quad \delta 2.40 - 2.48 (m, 2H), 3.71 (s, 3H), 4.54 - 4.58 (m, 1H), 5.03 - 5.09 (m, 2H), 5.68 - 5.77 (m, 1H), 6.82 (d, J=8.7Hz, 2H), 7.19 (d, J=8.7Hz, 2H) \\
^{13}\text{C NMR (75 MHz, CDCl}_3\text{):} & \quad \delta 38.4, 49.9, 67.9, 108.5, 112.4, 122.4, 112.4, 122.1, 129.6, 131.1, 153.6.
\end{align*}
\]
Entry 6: 1-(4-methyl phenyl) but-3-en-l-ol

\[ \text{IR}(\text{neat, cm}^{-1}): \]
\[ 3348, 3027, 2921, 2853, 1609, 1499, 1446, 1272, 1027 \]

\[ ^1\text{H NMR (300 MHz, CDCl}_3\text{):} \]
\[ \delta 2.40 (s, 3H), 2.49 - 2.53 (m, 2H), 2.83(br,1H), 4.63 - 4.69 (m, 1H), 5.13 - 5.19 (m, 2H), 5.71 - 5.86 (m, 1H), 7.18 - 7.27 (m, 4H) \]

\[ ^{13}\text{C NMR (75 MHz, CDCl}_3\text{):} \]
\[ \delta 20.99, 43.50, 73.12, 117.78, 125.73, 128.89, 134.57, 136.89, 140.87. \]

Entry 7: 1-(4-bromophenyl) but-3-en-l-ol

\[ \text{OH} \]
\[ \text{IR}(\text{KBr, cm}^{-1}): \]
\[ 3388, 3077, 2978, 2906, 1641, 1591, 1488, 1405, 1068, 1010, 919, 826 \]

\[ ^1\text{H NMR (300 MHz, CDCl}_3\text{):} \]
\[ \delta 2.37 - 2.58 (m, 2H), 3.17(br,1H), 4.55 - 4.59 (m, 1H), 5.06 - 5.11 (m, 2H), 5.63 - 5.77 (m, 1H), 7.13 (d,J=8.4Hz, 2H), 7.42 (d, J=8.4Hz, 2H). \]

\[ ^{13}\text{C NMR (75 MHz, CDCl}_3\text{):} \]
\[ \delta 43.4, 72.5, 118.4, 121.0, 127.5, 131.2, 133.8, 142.7. \]
Entry 8: 1-(naphthalene-2-yl) but-3-en-l-ol

\[
\text{H} \quad \text{OH}
\]

IR(KBr, cm\textsuperscript{-1}): 3384, 3072, 3033, 2975, 1638, 1490, 1451, 915, 700

\textsuperscript{1}H NMR (300 MHz, CDCl\textsubscript{3}): \delta 2.61 - 2.65 (m, 2H), 3.12 (br, 1H), 4.83 - 4.87 (m, 1H), 5.17 - 5.23 (m, 2H), 5.79 - 5.93 (m, 1H), 7.48 - 7.57 (m, 2H), 7.79 - 7.88 (m, 4H)

\textsuperscript{13}C NMR (75 MHz, CDCl\textsubscript{3}): \delta 43.5, 72.6, 114.8, 115.1, 118.1, 127.3, 127.4, 134.0, 139.5.

Entry 9: 1-(4-fluorophenyl) but-3-en-l-ol

\[
\text{F} \quad \text{OH}
\]

IR(KBr, cm\textsuperscript{-1}): 3340, 3035, 2922, 2753, 1625, 1475, 1425, 1283, 1030

\textsuperscript{1}H NMR (300 MHz, CDCl\textsubscript{3}): \delta 2.39 - 2.43 (m, 2H), 3.22 (br, 1H), 4.58 - 4.62 (m, 1H), 5.05 - 5.10 (m, 2H), 5.64 - 5.78 (m, 1H), 6.95 - 7.01 (m, 2H), 7.21 - 7.27 (m, 2H)

\textsuperscript{13}C NMR (75 MHz, CDCl\textsubscript{3}): \delta 43.5, 72.6, 114.8, 115.1, 118.1, 127.3, 127.4, 134.0, 139.5.
Entry 10: 1 – (2-Chlorophenyl) but-3-en-l-ol

\[
\text{IR(KBr,cm}^{-1}\text{): } 3380, 3085, 2930, 1635, 1445, 1045
\]

\[
\text{^1H NMR (300 MHz, CDCl}_3\text{): } \delta 1.26 (br, 1H), 2.26 - 2.43 (m, 2H), 5.11 - 5.22 (m, 2H), 5.80 - 5.94 (m, 1H), 7.18 - 7.34 (m, 3H), 7.57 (d, J=7.2Hz, 1H)
\]

\[
\text{^13C NMR (75 MHz, CDCl}_3\text{): } \delta 42.8, 70.4, 119.6, 127.8, 129.3, 130.2, 132.5, 135.1, 141.9.
\]

Entry 11: 1 – (2-bromophenyl) but-3-en-l-ol

\[
\text{IR(KBr,cm}^{-1}\text{): } 3383, 3065, 2975, 1654, 1590, 1475, 1065, 1025, 927
\]

\[
\text{^1H NMR (300 MHz, CDCl}_3\text{): } \delta 2.28 - 2.38 (m, 1H), 2.54-2.60(m,1H), 2.99 (br,1H), 5.0 - 5.18 (m, 3H), 5.81 - 5.92 (m, 1H), 7.07 - 7.13 (m, 1H), 7.28 - 7.33 (m, 1H), 7.48 - 7.52 (m, 2H).
\]

\[
\text{^13C NMR (75 MHz, CDCl}_3\text{): } \delta 41.8, 71.7, 118.2, 121.6, 127.2, 127.5, 128.6, 132.4, 134.1, 142.6.
\]
Entry 12: 1-Undecen-4-ol

\[
\begin{align*}
\text{IR(KBr, cm}^{-1}\text{):} & \quad 3358, 3077, 2956, 2927, 2856, 1641, 1466, 994, 912 \\
\text{\textsuperscript{1}H NMR (300 MHz, CDCl}_3\text{):} & \quad \delta \ 0.87 \ (t, J=7.1Hz, 3H), \ 1.28 - 1.46 \ (m, 12H), \ 1.56(s, 1H), \ 2.08 - 2.20 \ (m, 1H), \ 2.25-2.34 \ (m, 1H) \ 3.58-3.68 \ (m, 1H), \ 5.10 - 5.16 \ (m, 2H), \ 5.76 - 5.90 \ (m, 1H) \\
\text{\textsuperscript{13}C NMR (75 MHz, CDCl}_3\text{):} & \quad \delta \ 14.1, 22.6, 25.7, 29.3, 29.6, 31.8, 31.63, 36.8, 40.2, 70.7, 117.8, 134.1.
\end{align*}
\]
2.7. References


