Supplementary Material

Diels-Alder reactions with 1,1-Diethoxybut-3-yn-2-one and some 1,1-diethoxy-5-hydroxyalk-3-yn-2-ones and their acetates

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Flash-column chromatography gradient elution

Flash-column chromatography (FC) was performed using a Reveleris X” Flash System Silica Gel from Fluka Analytical (230–400 mesh) and eluting with a gradient of ethyl acetate in a mixture of hexanes for in total 16.5 min. The gradient applied can be summarized as follows:

<table>
<thead>
<tr>
<th>Time span per mixture (min)</th>
<th>Ethyl acetate (% v/v) in hexanes</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0 – 1.5</td>
<td>0</td>
</tr>
<tr>
<td>1.5 – 2.5</td>
<td>1</td>
</tr>
<tr>
<td>2.5 – 4.0</td>
<td>2</td>
</tr>
<tr>
<td>4.0 – 9.5</td>
<td>3</td>
</tr>
<tr>
<td>9.5 – 16.5</td>
<td>10</td>
</tr>
</tbody>
</table>
2,2-Diethoxy-1-(4-tolyl)ethanone (3a)

Figure 1. IR spectrum of 2,2-diethoxy-1-(4-tolyl)ethanone
Figure 2: $^1$H-NMR spectrum of 2,2-diethoxy-1-(4-tolyl)ethanone

Figure 3: $^{13}$C-NMR spectrum of 2,2-diethoxy-1-(4-tolyl)ethanone
2,2-Diethoxy-1-(3,4-dimethylphenyl)ethanone (3b)

![Chemical Structure](image)

Figure 4 IR spectrum of 2,2-diethoxy-1-(3,4-dimethylphenyl)ethanone
Figure 5 $^1$H-NMR spectrum of 2,2-diethoxy-1-(3,4-dimethylphenyl)ethanone

Figure 6 $^{13}$C-NMR spectrum of 2,2-diethoxy-1-(3,4-dimethylphenyl)ethanone
2,2-Diethoxy-1-(2,4-dimethylphenyl)ethanone (3c)

Figure 7 IR spectrum of 2,2-diethoxy-1-(2,4-dimethylphenyl)ethanone
Figure 8 $^1$H-NMR spectrum of 2,2-diethoxy-1-(2,4-dimethylphenyl)ethanone

Figure 9 $^{13}$C-NMR spectrum of 2,2-diethoxy-1-(2,4-dimethylphenyl)ethanone
(2-Diethoxyacetyl-4,5-dimethylphenyl)methyl acetate (7a)

\[
\begin{align*}
\text{AcO} & \quad - \quad \text{OEt} \\
\quad & \quad \text{OEt}
\end{align*}
\]

**Figure 10** IR spectrum of (2-diethoxyacetyl-4,5-dimethylphenyl)methyl acetate
Figure 11 $^1$H-NMR spectrum of (2-diethoxyacetyl-4,5-dimethylphenyl)methyl acetate

Figure 12 $^{13}$C-NMR of (2-diethoxyacetyl-4,5-dimethylphenyl)methyl acetate
1-(2-Diethoxyacetyl-4,5-dimethylphenyl)ethyl acetate (7b)

Figure 13 IR spectrum of 1-(2-diethoxyacetyl-4,5-dimethylphenyl)ethyl acetate
Figure 14 $^1$H-NMR spectrum of 1-(2-diethoxyacetyl-4,5-dimethylphenyl)ethyl acetate

Figure 15 $^{13}$C-NMR spectrum of 1-(2-diethoxyacetyl-4,5-dimethylphenyl)ethyl acetate
1-(2-Diethoxyacetyl-4,5-dimethylphenyl)octyl acetate (7c)

![Chemical Structure]

Figure 16 IR spectrum of 1-(2-diethoxyacetyl-4,5-dimethylphenyl)octyl acetate
Figure 17 $^1$H-NMR spectrum of 1-(2-diethoxyacetyl-4,5-dimethylphenyl)octyl acetate

Figure 18 $^{13}$C-NMR spectrum of 1-(2-diethoxyacetyl-4,5-dimethylphenyl)octyl acetate
(2-Diethoxyacetyl-4,5-dimethylphenyl)(phenyl)methyl acetate (7d)

Figure 19 IR spectrum of (2-diethoxyacetyl-4,5-dimethylphenyl)(phenyl)methyl acetate
Figure 20 $^1$H-spectrum of (2-diethoxyacetyl-4,5-dimethylphenyl)(phenyl)methyl acetate

Figure 21 $^{13}$C-spectrum of (2-diethoxyacetyl-4,5-dimethylphenyl)(phenyl)methyl acetate