SUPPORTING INFORMATION

Simple salts of abundant metals (Fe, Bi, and Ti) supported on Montmorillonite as efficient and recyclable catalysts for regioselective intramolecular and intermolecular hydroalkoxylation reactions of double bonds and tandem processes.

Irene Notar Francesco, a Bastien Cacciuttolo, b Oana Pascu, c Cyril Aymonier, c Mathieu Pucheault b and Sylvain Antoniotti* a

a Institut de Chimie de Nice, UMR 7272 CNRS - Université Nice Sophia Antipolis, Parc Valrose, 06108 Nice, France. E-mail : sylvain.antoniotti@unice.fr

b Institut des Sciences Moléculaires, UMR 5255 CNRS - Université de Bordeaux, 351 cours de la libération, 33405 Talence cedex, France.

c Institut de Chimie de la Matière Condensée de Bordeaux, UPR 9048 CNRS - Université de Bordeaux, 87, Avenue du Docteur Schweitzer, 33608 PESSAC cedex, France.

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Experimental Section

1. Materials and Methods.

\(^1\)H NMR and \(^{13}\)C NMR spectra were recorded on BRUCKER AC 200 (200 MHz). \(^1\)H NMR spectra are reported as follows: chemical shift in ppm (δ) relative to the chemical shift of CDCl\(_3\) at 7.26 ppm, integration, multiplicities (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and br = broadened), and coupling constants (Hz). \(^{13}\)C NMR spectra reported in ppm (δ) relative to CDCl\(_3\) at 77.16 ppm.

Column chromatography was carried out on silica gel (spherical, neutral, 63-200 μm, Geduran Si 60, Merck KGaA).

GC-TCD analysis were carried out using a Shimadzu QP2010 plus gas chromatograph, under the following operation conditions: vector gas, He; injector temperature, 250 °C; detector temperature, 210 °C at 60 mA; split ratio, 1/20; total flow, 22,5 mL/min; Phenomenex Zebon ZB5MS column, polydimethylsiloxane (10 m, inside diameter 0.10 mm, film thickness 0.10 μm); temperature program, 80-200 °C at 10 °C/min and 200 °C for 8 min.

GC/MS analysis were performed by using a Shimadzu QP2010 gas chromatograph (conditions: carrier gas, He; injector and detector temperatures, 250 °C; injected volume, 0.5 μL; split ratio, 1/100; (pressure, 180 kPa); SLB-5ms capillary column (thickness: 0.25 mm, length: 30 m, inside diameter: 0.25 mm); temperature program, 60-250 °C at 2 °C/min, and 250 °C, coupled to a mass selective detector. Mass spectra were obtained by electron ionisation at 70 eV, m/z 35-400, source temperature 250 °C; only the most abundant ions are given.

High resolution mass spectrometry (HRMS) was performed at ERINI platform (Grasse, FRANCE) using a Waters APGC coupled with a Waters Xevo G2 QTOF spectrometer.

Screening reactions were performed in a Carousel 12 Plus parallel synthesizer purchased from Radleys.

2. General procedure for cyclisation of unsaturated alcohols.

In a Schlenk tube, substrate (1 mmol), Fe-MMT (5 mol% of metal, 100 mg of material) and DMC (2 ml) are introduced and the tube closed with a PTFE cap. The mixture is stirred at the desired temperature and the reaction monitored by TLC or GC-TCD. After completion, the mixture is filtered through a cotton wool pad. The filter is rinsed with diethyl ether and the solution concentrated at reduced pressure affording the crude cyclic ether which is then purified by flash chromatography over silica gel (petroleum ether/Et\(_2\)O).
3. Catalyst characterization

Metal-doped montmorillonite (M-MMT):

- XRD analysis

![XRD spectra of Bi, Fe, and Cu-doped MMT and pristine MMT.](image)

**Figure S1.** XRD spectra of Bi, Fe, and Cu-doped MMT and pristine MMT.

**Table S1.** Data obtained from XRD analysis

<table>
<thead>
<tr>
<th>Sample</th>
<th>Obs max first peak</th>
<th>d(obsmx)</th>
<th>2θ</th>
<th>sinθ</th>
<th>d(calc)</th>
<th>FWHM</th>
</tr>
</thead>
<tbody>
<tr>
<td>MMT</td>
<td>7,2</td>
<td>12,2676</td>
<td>7,11</td>
<td>0,06200665</td>
<td>12,20981904</td>
<td>1,029</td>
</tr>
<tr>
<td>Fe@MMT</td>
<td>5,9</td>
<td>14,96759</td>
<td>5,826</td>
<td>0,05084569</td>
<td>14,88995517</td>
<td>0,93</td>
</tr>
<tr>
<td>Bi@MMT</td>
<td>6,371</td>
<td>13,86</td>
<td>6,365</td>
<td>0,05551655</td>
<td>13,63719566</td>
<td>1,738</td>
</tr>
<tr>
<td>Cu@MMT</td>
<td>6,18</td>
<td>14,29006</td>
<td>6,17</td>
<td>0,053817</td>
<td>14,0678596</td>
<td>1,214</td>
</tr>
</tbody>
</table>

The XRD spectrum of Bi-MMT clearly shows the presence of BiOCl within the matrix.
ICP-MS titration of metal content in the modified clays showed the following w/w metal contents: (Cu-MMT 2.04%, Fe-MMT 2.67%, Bi-MMT 4.87%, Ti-MMT 1.81%).

Metal nanoparticles (M NPs):

The supported NPs were analyzed by powder X-ray diffraction (XRD - PANalytical X’Pert Pro with Cu $\lambda$K$_{\alpha}$ radiation) for the composition determination and metal NPs crystallite size evaluation using the Scherer equation. The nanostructured catalysts were also observed by transmission electron microscopy (TEM- JEOL 2100).

Table S2. Preparation and XRD-characterisation of M-NPs

<table>
<thead>
<tr>
<th>Label</th>
<th>Precursor</th>
<th>Metal loading (wt%)</th>
<th>Surfactant (molar ratio Metal : surf of 1:2)</th>
<th>ScCO$_2$+EtOH (co-solvent)</th>
<th>Phase (XRD)</th>
<th>$D_{XRD}$ (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pt NPs/SiO$_2$</td>
<td>Pt(hfac)$_2$</td>
<td>-</td>
<td>-</td>
<td>Pt</td>
<td>Cu$_3$Pt, Cu</td>
<td>7</td>
</tr>
<tr>
<td>Pt-Cu NPs/SiO$_2$</td>
<td>Cu(hfac)$_2$</td>
<td></td>
<td>Hexadecylamine</td>
<td></td>
<td>Cu</td>
<td>5.7</td>
</tr>
<tr>
<td></td>
<td>Pt(hfac)$_2$</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cu NPs/SiO$_2$(1)</td>
<td>Cu(tmhd)$_2$</td>
<td></td>
<td>Hexadecylamine</td>
<td>Cu</td>
<td>Cu</td>
<td>25</td>
</tr>
<tr>
<td>Cu NPs/SiO$_2$(2)</td>
<td>Cu(hfac)$_2$</td>
<td></td>
<td>Hexadecylamine</td>
<td></td>
<td>Cu</td>
<td>21</td>
</tr>
<tr>
<td>Cu$_x$O NPs/SiO$_2$(1)</td>
<td>Cu(hfac)$_2$</td>
<td>5</td>
<td>-</td>
<td>CuO</td>
<td>Cu$_{2+1}$O</td>
<td>13</td>
</tr>
<tr>
<td>Cu$_x$O NPs/SiO$_2$(2)</td>
<td>Cu(hfac)$_2$</td>
<td></td>
<td>Oleic acid</td>
<td></td>
<td>SnO$_2$</td>
<td>27</td>
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<tr>
<td>SiO$_2$@Sn1b</td>
<td>Sn(hfac)$_2$</td>
<td></td>
<td>-</td>
<td>Sn</td>
<td>SnO$_2$</td>
<td>-</td>
</tr>
<tr>
<td>SiO$_2$@Sn2b</td>
<td>Sn(hfac)$_2$</td>
<td></td>
<td>Hexadecylamine</td>
<td>Sn</td>
<td>SnO$_2$</td>
<td>-</td>
</tr>
<tr>
<td>SiO$_2$@Sn3</td>
<td>Sn(hfac)$_2$</td>
<td></td>
<td>Sodium dodecyl sulphate</td>
<td>Sn</td>
<td>SnO$_2$</td>
<td>-</td>
</tr>
</tbody>
</table>

Figure S2. Data obtained from XPS analysis of Bi, Fe, and Cu-doped MMT.
4. Starting materials characterisation

Compounds 1a, 1i, 1l, and 1r are commercially available and were purchase from Sigma-Aldrich. Compound \((E)-1o\) was a kind gift from Firmenich CH (Dr Charles Fehr).

Scheme S1. Various synthetic routes used in this study to access alcohols 1c-1n.
5-Methyl-2,2-diphenyl-hex-4-en-1-ol.

1b was prepared in three steps from 1,1-diphenylacetic acid following published procedures.²

\[
\begin{align*}
\text{HO} & \quad \text{MeOH, rt} \quad \text{HCl} \quad \text{MeO} \quad \text{Br} \quad \text{MeO} \quad \text{LiAlH}_4 \\
\text{Ph} & \quad \text{Ph} \quad \text{Ph} & \quad \text{Ph} \quad \text{Ph} & \quad \text{Ph} \quad \text{Ph} & \quad \text{Ph} \quad \text{Ph}
\end{align*}
\]

R\text{f} = 0.43 (cyclohexane, EtOAc = 8/2)

\[^1\text{H} \text{NMR} \ (\text{CDCl}_3, \ 200 \text{ MHz}): \ \delta \ 7.30-7.05 \ (m, 10\text{H}), \ 4.76 \ (m, 1\text{H}), \ 4.04 \ (d, J_d=6.8 \text{ Hz}, 2\text{H}), \ 2.81 \ (d, J=7.2 \text{ Hz}, 2\text{H}), \ 1.52 \ (s, 3\text{H}), \ 1.46 \ (s, 3\text{H}).
\]

\[^{13}\text{C} \text{NMR} \ (\text{CDCl}_3, \ 50 \text{ MHz}): \ \delta \ 145.75, \ 134.60, \ 128.43, \ 128.19, \ 126.32, \ 119.95, \ 68.33, \ 53.36, \ 35.10, \ 26.08, \ 18.00.\]

MS (EI; 70 eV) 266(0.2) [M]\text{ }^+\text{, }197(55), \ 165(11), \ 115(10), \ 105(100), \ 91(95); \ 77(25), \ 41(17).\]

2-Methylundec-2-en-6-ol

R\text{f} = 0.25 (cyclohexane, EtOAc = 8/2)

\[^{1}\text{H} \text{NMR} \ (\text{CDCl}_3, \ 200 \text{ MHz}): \ \delta \ 5.13 \ (m, 1\text{H}), \ 3.60 \ (bs, 1\text{H}), \ 2.08 \ (m, 2\text{H}), \ 1.69 \ (s, 3\text{H}), \ 1.62 \ (s, 3\text{H}), \ 1.55-1.18 \ (m, 10\text{H}), \ 0.89 \ (t, J=6.26 \text{ Hz}, 3\text{H}).
\]

MS (EI; 70 eV) 184(2) [M]\text{ }^+, \ 127(11), \ 113(13), \ 110(25), \ 109(11); \ 99(12), \ 96(13), \ 95(99), \ 83(16), \ 82(79), \ 81(36), \ 71(18), \ 69(73), \ 57(26), \ 55(61), \ 41(100).\]
7-Methyl-1-phenyloct-6-en-3-ol

Rᵣ = 0.22 (cyclohexane, EtOAc = 8/2)

$^1$H NMR (CDCl₃, 200 MHz): $\delta$ 7.34-7.08 (m, 5H), 5.13 (m, 1H), 3.74-3.50 (m, 1H), 2.92-2.54 (m, 2H), 2.22-1.96 (m, 2H), 1.87-1.40 (m, 4H), 1.69 (s, 3H), 1.62 (s, 3H).

MS (EI; 70 eV) 218(0) [M]$^+$, 157(13), 144(12), 130(21), 109(14); 105(17), 96(34), 91(100), 82(18), 69(31), 55(17), 41(49).

6-Methyl-1-phenylhept-5-en-2-ol

Rᵣ = 0.24 (cyclohexane, EtOAc = 8/2)

$^1$H NMR (CDCl₃, 200 MHz): $\delta$ 7.27 (d, $J$=8.2 Hz, 2H), 7.18 (d, $J$=8.2 Hz, 2H), 5.16 (m, 1H), 4.67 (m, 1H), 2.37 (s, 3H), 2.19-1.66 (m, 4H), 1.72 (s, 3H), 1.62 (s, 3H).

MS (EI; 70 eV) 204(11) [M]$^+$, 186(18), 171(23), 161(25), 147(42); 143(61), 134(70), 128(14), 121(100), 119(51), 105(34), 93(96), 77(56), 65(25), 55(53), 41(71).

3-(2-methylprop-2-enyloxy)propan-1,2-diol prepared in two steps from solketal by methallylation and deprotection of the acetonide.

Rᵣ = 0.05 (cyclohexane, EtOAc = 7/3)

$^1$H NMR (CDCl₃, 200 MHz): $\delta$ 4.94 (m, 1H), 4.91 (m, 1H), 3.92 (s, 2H), 3.87 (m, 1H), 3.67 (m, 2H), 3.49 (d, $J$=2.0 Hz, 1H), 3.47 (d, $J$=4.0 Hz, 1H), 2.92 (bs, 1H), 2.52 (bs, 1H), 1.73 (s, 3H).

$^{13}$C NMR (CDCl₃, 50 MHz): 141.79, 112.81, 75.54, 71.66, 70.74, 64.27, 19.51.

MS (EI; 70 eV) 146(0) [M]$^+$, 72(71), 61(30), 57(46), 55(100), 45(12), 43(50).
2-Hydroxy-3-(2-methylprop-2-enyloxy)propyl acetate

Rf = 0.37 (cyclohexane, EtOAc = 7/3)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 4.94 (m, 1H), 4.91 (m, 1H), 4.17 (d, $J$=4.3 Hz, 1H), 4.14 (d, $J$=4.3 Hz, 1H), 4.09-3.96 (m, 1H), 3.92 (s, 2H), 3.45 (m, 2H), 2.52 (bs, 1H), 2.09 (s, 3H), 1.73 (s, 3H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 171.26, 141.80, 112.76, 75.50, 70.72, 68.94, 65.76, 20.96, 19.47.

MS (EI; 70 eV) 188(5) [M]$^+$, 117(26), 57(12), 55(42), 43(100).

1-(tert-Butyldimethylsilyloxy)-3-(2-methylprop-2-enyloxy)propan-2-ol

Rf = 0.59 (cyclohexane, EtOAc = 7/3)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 4.91 (m, 1H), 4.89 (m, 1H), 3.91 (s, 2H), 3.86-3.76 (m, 1H), 3.72-3.58 (m, 2H), 3.50-3.37 (m, 2H), 2.51 (d, $J$=5.0 Hz, 1H), 1.72 (s, 3H), 0.9 (s, 9H), 0.06 (s, 6H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 142.16, 112.43, 75.43, 70.83, 70.77, 64.18, 26.00, 19.53, 18.42, -5.29.

MS (EI; 70 eV) 260(0) [M]$^+$, 131(100), 117(28), 101(21), 89(10); 75(50), 55(85).

(Z)-5-Phenylpent-4-en-1-ol

Rf = 0.24 (cyclohexane, EtOAc = 1/1)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 7.50-7.10 (m, 5H), 6.46 (d, $J$=11.6 Hz, 1H), 5.67 (dt, $J_d$=11.6 Hz, $J_t$=7.3 Hz, 1H), 3.67 (t, $J$=6.6 Hz, 2H), 2.42 (m, 2H), 1.76 (m, 2H).

MS (EI; 70 eV) 162(19) [M]$^+$, 144(23), 129(100), 115(59), 104(12); 91(69), 77(16), 65(14) 51(14), 41(4).
(E)-5-Phenylpenta-4-en-1-ol\(^5\)

\[ R_f = 0.24 \text{ (cyclohexane, EtOAc = 1/1)} \]

\(^1\)H NMR (CDCl\(_3\), 200 MHz): \( \delta \) 7.50-7.10 (m, 5H), 6.43 (d, \( J = 15.8 \) Hz, 1H), 6.23 (dt, \( J_d = 15.8 \) Hz, \( J_t = 6.6 \) Hz, 1H), 3.71 (t, \( J = 6.4 \) Hz, 2H), 2.32 (dt, \( J_d = 6.6 \) Hz, \( J_t = 6.6 \) Hz, 2H), 1.76 (tt, \( J_d = 6.4 \) Hz, \( J_t = 6.6 \) Hz, 2H).

MS (EI; 70 eV) 162(4) [M]+, 144(14), 129(65), 115(72), 104(16); 91(100), 77(35), 65(31) 51(42), 41(12).

\[(E,Z)-3,3\text{-Dimethylhex-4-en-1-ol}\]

\[ R_f = 0.58 \text{ (cyclohexane, EtOAc = 1/1)} \]

\(^1\)H NMR (CDCl\(_3\), 200 MHz): \( \delta \) 5.50-5.20 (m, 2H), 3.71 (bt, \( J = 7.1 \) Hz, 2H), 1.78-1.50 (m, 5H), 1.14 (s, 6H).

MS (EI; 70 eV) 128(1) [M]+, 110(2), 95(17), 83(71), 69(32); 55(100).

2-(3-Methylbut-2-enyl)phenol (2-prenylphenol)\(^6\)\(^8\)

\[ R_f = 0.71 \text{ (cyclohexane, EtOAc = 9/1)} \]

\(^1\)H NMR (CDCl\(_3\), 200 MHz): \( \delta \) 7.20-7.00 (m, 2H), 6.96-6.68 (m, 2H), 5.40 (m, 1H), 3.36 (d, \( J = 7.1 \) Hz, 2H), 1.78 (s, 3H).

MS (EI; 70 eV) 162(40) [M]+, 147(34), 119(17), 107(100), 91(45); 77(39), 65(14), 51(19), 41(26).
2-(3-Methylbut-2-enyl)phenol (2-geranylphenol)

Rf = 0.37 (cyclohexane, EtOAc = 9/1)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 7.20-7.00 (m, 2H), 6.95-6.65 (m, 2H), 5.32 (m, 1H), 5.07 (m, 1H), 3.37 (d, $J$=7.1 Hz, 2H), 2.09 (m, 4H), 1.77 (s, 3H), 1.69 (s, 3H), 1.60 (s, 3H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 157.05, 130.05, 127.65, 123.97, 122.56, 121.77, 120.84, 115.92, 39.82, 29.89, 26.53, 25.83, 17.84, 16.29.

MS (EI; 70 eV) 230(7) [M]$^+$, 161(30), 145(21), 133(26), 123(71); 109(12), 107(68), 91(28), 81(18), 69(77), 41(100).

1-Methoxy-3,7-dimethyloct-6-ene

Rf = 0.62 (cyclohexane, EtOAc = 9/1)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 5.09 (m, 1H), 3.40 (t, $J$=6.8 Hz, 2H), 3.33 (s, 3H), 1.97 (m, 2H), 1.67 (s, 3H), 1.59 (s, 3H), 1.50-1.00 (m, 5H), 0.89 (d, $J$=6.4 Hz, 3H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 131.00, 124.95, 71.32, 58.70, 37.40, 36.74, 29.71, 25.86, 25.62, 19.66, 17.77.

MS (EI; 70 eV) 170(1) [M]$^+$, 138(21), 123(33), 109(17), 95(59), 85(65); 81(87), 67(79), 55(43), 45(86), 41(100).

2-Methyldec-2-ene

Rf = 0.90 (cyclohexane, EtOAc = 9/1)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 5.11 (m, 1H), 2.09-1.87 (m, 2H), 1.68 (s, 3H), 1.60 (s, 3H), 1.50-1.00 (m, 10H), 0.89 (t, $J$=6.5 Hz, 3H).
MS (EI; 70 eV) 154(11) [M]⁺, 83(10), 69(100), 56(69), 41(69).

2-Methylnon-2-ene

Rᵣ = 0.90 (cyclohexane, EtOAc = 9/1)

¹H NMR (CDCl₃, 200 MHz): δ 5.12 (m, 1H), 2.09-1.82 (m, 2H), 1.69 (s, 3H), 1.60 (s, 3H), 1.48-1.10 (m, 8H), 0.88 (t, J=6.5 Hz, 3H).

¹³C NMR (CDCl₃, 50 MHz): δ 131.25, 125.13, 32.01, 30.05, 29.21, 28.23, 25.87, 22.85, 17.79, 14.26.

MS (EI; 70 eV) 140(16) [M]⁺, 69(100), 56(69), 41(70).

5. Final Products Characterization

2,2,6-Trimethyltetrahydro-2H-pyran.³ The compound was obtained from cyclisation of 1a (1 mmol) in deuterated dimethylcarbonate. The reaction was run to completion under TLC and GC monitoring. NMR analyses were carried out on samples directly taken from the reaction medium by using benzene as internal standard (calculated yield = 98%).

Rᵣ = 0.53 (Cyclohexane, EtOAc = 9/1)

¹H NMR (DMC-d₆, 200 MHz): δ 3.78-3.54 (m, 1H), 1.75 – 1.53 (m, 2H), 1.52-1.25 (m, 4H), 1.17 (s, 3H), 1.13(s, 3H), 1.04 (d, J = 6.1 Hz, 3H).

¹³C NMR (DMC-d₆, 50 MHz): δ 71.64, 66.65, 36.43, 33.99, 31.84, 22.53, 21.67, 20.59.

MS (EI; 70 eV) 128(0) [M]⁺, 113(47), 95(7), 70(10), 59(100), 56(36), 43(80).

2,2-Dimethyl-5,5-diphenyltetrahydro-2H-pyran. Spectral data matched literature reference.²

Rᵣ = 0.64 (cyclohexane, EtOAc = 4/1)
$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 7.37-7.10 (m, 10H), 4.06 (s, 2H), 2.50-2.33 (m, 2H), 1.40 (dd, $J$ = 7.3, 5.0 Hz, 1H), 1.23 (s, 6H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): 146.54, 128.21, 128.02, 126.05, 71.32, 69.06, 45.96, 32.65, 30.91, 26.46.

MS (EI; 70 eV) 266(5) [M]$^+$, 236(15), 180(100), 165(36), 152 (4); 129(2), 115(13), 103(4), 91(14), 73(8), 65(3), 43(10), 41(5).

(4,4-Dimethyl-1-phenyl-1,2,3,4-tetrahydronaphthalen-1-yl)methanol. Isolated by column chromatography (silica gel) eluting with petroleum ether: diethylether = 90:10.

R$_f$ = 0.40 (Cyclohexane, EtOAc = 4/1)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 7.47 (d, $J$ = 7.8 Hz, 1H), 7.33-7.11 (m, 6H), 7.10-6.98 (m, 2H), 4.28-4.02 (m, 2H), 2.58-2.28 (m, 1H), 1.94 (dt, $J$ = 9.2, 4.2 Hz, 1H), 1.54 (dd, $J$ = 6.7, 5.4 Hz, 2H), 1.46 (s, 1H), 1.33 (s, 3H), 1.30 (s, 3H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): 148.28, 147.66, 137.81, 128.39, 128.18, 128.90, 127.42, 126.96, 126.14, 125.99, 69.83, 49.66, 34.38, 34.21, 32.16, 31.94, 31.36.

MS (EI; 70 eV) 266(0) [M]$^+$, 248(2), 233(1), 219(1), 205(4), 197(52), 180(7), 165(11), 157(7), 152 (4), 142(4), 129(5), 119(9), 155(10), 105(100), 91(100), 77(25), 65(8), 51 (5), 41(17).

2,2-Dimethyl-6-pentyltetrahydro-2H-pyran. The compound was obtained from cyclisation of ac (1 mmol). Final product was isolated by column chromatography (silica gel) eluting with petroleum ether: diethylether = 95:5 as a colourless liquid (0.89 mmol, 89%).

R$_f$ = 0.79 (Cyclohexane, EtOAc = 9/1)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 3.50-3.41 (m, 1H), 1.67-0.94 (m, 14H), 1.19 (s, 3H), 1.17 (s, 3H), 0.87 (t, $J$ = 6.8 Hz, 3H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 71.57, 70.46, 37.10, 36.45, 32.10, 31.66, 25.39, 22.79, 22.13, 20.28, 14.22.

MS (EI; 70 eV) 184(0) [M]$^+$, 169(16), 151 (4), 126(3), 113(63), 99 (15), 95(61), 83(15), 69(38), 59(100), 56(72), 43(57).

HRMS calculated for C$_{12}$H$_{23}$O (M-H)$^+$: 183.1749; found: 183.1743, $|\Delta|= 3.3$ ppm.
2,2-Dimethyl-6-(2-phenylethyl)-tetrahydro-2H-pyran. The compound was obtained from cyclisation of \textbf{1d} (1 mmol). Final product was isolated by column chromatography (silica gel) eluting with petroleum ether: diethylether = 9:1 as a colourless liquid (0.89 mmol, 89%).

\[ R_f = 0.64 \] (Cyclohexane, EtOAc = 9/1)

\[ \begin{align*}
\delta & 7.43\text{-}7.01 \text{ (m, 5H)} , 3.66\text{-}3.30 \text{ (m, 1H)} , 2.89\text{-}2.46 \text{ (m, 2H)} , 1.90\text{-}0.99 \text{ (m, 8H)} , 1.23 \text{ (s, 3H)} , 1.17 \text{ (s, 3H).}
\end{align*} \]

\[ \begin{align*}
\delta & 142.64, 128.62, 128.23, 125.60, 77.80, 77.16, 76.53, 71.62, 69.19, 38.49, 36.39, 32.05, 31.76, 22.07, 20.18.
\end{align*} \]

\[ \text{MS (EI; 70 eV) 218(12) [M]\textsuperscript{+}, 185(6), 157 (7), 140(25), 129(14), 113(34), 104(36), 91(100), 69(22), 56(14), 43(29).} \]

\[ \text{HRMS calculated for C}_{15}\text{H}_{23}\text{O (M+H)}\text{+}: 219.1749; \text{found: 203.1751.} \Delta m_1 = 0.9 \text{ ppm.} \]

2,2-Dimethyl-6-(p-tolyl)tetrahydro-2H-pyran. The compound was obtained from cyclisation of \textbf{1e} (1 mmol). Final product was isolated by column chromatography (silica gel) eluting with petroleum ether: diethylether = 98:2 as colourless liquid (0.38 mmol, 38%).

\[ R_f = 0.67 \] (Cyclohexane, EtOAc = 9/1)

\[ \begin{align*}
\delta & 7.25 \text{ (d, } J = 8 \text{ Hz, 2H)} , 7.12 \text{ (d, } J = 8 \text{ Hz, 2H)} , 4.55 \text{ (dd, } J = 11.5, 1.9 \text{ Hz, 1H)} , 2.31 \text{ (s, 3H)} , 1.82\text{-}1.41 \text{ (m, 6H)} , 1.30 \text{ (s, 3H)} , 1.29 \text{ (s, 3H).}
\end{align*} \]

\[ \begin{align*}
\delta & 141.33, 136.76, 129.04, 126.15, 72.90, 72.42, 36.19, 34.21, 32.13, 22.07, 21.24, 20.61.
\end{align*} \]

\[ \text{MS (EI; 70 eV) 204(14) [M]\textsuperscript{+}, 189(5), 146 (25), 131(15), 121(100), 105 (5), 91(19), 84(4), 77(6), 69(6), 56(31), 43(11).} \]

\[ \text{HRMS calculated for C}_{14}\text{H}_{19}\text{O (M-H)}\text{+}: 203.1436; \text{found: 203.1436.} \Delta m_1 = 0.0 \text{ ppm.} \]

cis/trans-(2-Isopropyl-1,3-dioxolan-4-yl)methanol (\textbf{2f}) and cis,trans-2-isopropyl-1,3-dioxan-5-ol (\textbf{2f'}). The compounds were obtained from cyclisation of \textbf{1f} (1 mmol). Final products were isolated as 1/1
mixture of stereoisomers (colourless oil) by column chromatography (silica gel) eluting with petroleum ether:diethylether with gradient elution from 4:1 to 10:0 (isolated yields: 51 and 31% respectively). Spectral data matched literature reference.10

R_f = 0.71 (Cyclohexane, EtOAc = 1/1) [cis,trans-2f]

R_f = 0.79 (Cyclohexane, EtOAc = 1/1) [cis,trans-2f’]

[cis/trans-2f] 1H NMR (CDCl₃, 200 MHz): δ 4.75 (d, J = 4.7 Hz, 1H), 4.66 (d, J = 4.6 Hz, 1H), 4.32-4.02 (m, 2H), 4.00-3.16 (m, 7H), 2.04 (br s, 2H), 1.94-1.70 (m, 2H), 0.97 (d, J = 5.6 Hz, 6H), 0.94 (d, J = 6.6 Hz, 6H).

13C NMR (CDCl₃, 50 MHz): δ 108.79, 108.65, 76.52, 76.29, 66.80, 66.55, 63.52, 62.76, 32.30, 31.85, 16.99, 16.95, 16.89, 16.80.

MS (EI; 70 eV) 146(0) [M]+, 145(1), 115(3), 103(82), 97(7), 86(2), 71(5), 69(2), 57(100), 55(31), 43(24), 41(15). (I stereoisomer)

[cis/trans-2f’] 1H NMR (CDCl₃, 200 MHz): δ 4.27 (d, J = 5 Hz, 1H), 4.20 (d, J = 5 Hz, 1H), 4.13 (t, J = 4.6 Hz, 2H), 4.01 (d, J = 11.2 Hz, 1H), 3.93-3.76 (m, 3H), 3.86 (d, J = 11.2 Hz, 1H), 3.54-3.45 (m, 1H), 3.36 (d, J = 10.9 Hz, 1H), 3.31 (d, J = 10.2 Hz, 1H), 2.02 (br s, 2H), 1.91-1.66 (m, 2H), 0.94 (d, J = 6.8 Hz, 6H), 0.92 (d, J = 6.8 Hz, 6H).

13C NMR (CDCl₃, 50 MHz): δ 106.35, 105.69, 71.88, 71.70, 64.28, 61.61, 32.82, 32.35, 17.31, 16.94.

MS (EI; 70 eV) 146(0) [M]+, 145(1), 116(1), 103(100), 73(53), 57(63), 55(31), 43(42), 41(15). (II stereoisomer)

[cis/trans-(2-Isopropyl-1,3-dioxolan-4-yl)-methyl acetate. The compound was obtained from cyclisation of 1g (1 mmol). Final product was isolated as mixture of stereoisomers (colourless oil) by column chromatography (silica gel) eluting with 9:1 petroleum ether:diethylether (isolated yield: 59%).

R_f = 0.52 (Cyclohexane, EtOAc = 4/1)

1H NMR (CDCl₃, 200 MHz): δ 4.73 (d, J = 4.6 Hz, 1H), 4.66 (d, J = 4.4 Hz, 1H), 4.37-4.18 (m, 2H), 4.17-3.98 (m, 3H), 3.91 (dd, J= 8.3, 6.9 Hz, 1H), 3.74 (dd, J= 8.3, 4.9 Hz, 1H), 3.58 (dd, J= 8.4, 6.7 Hz, 1H), 2.08 (s, 3H), 2.06 (s, 3H), 1.94-1.67 (m, 2H), 0.97-0.88 (m, 12H).
$^{13}$C NMR (CDCl$_3$, 50 MHz): 170.89, 170.84, 109.02, 108.51, 73.70, 73.53, 67.27, 67.19, 64.72, 64.30, 32.06, 31.77, 20.89, 20.86, 16.93, 16.87, 16.59, 16.55.

MS (EI; 70 eV) 188(0) [M$^+$], 187(0), 145(43), 115(1), 97(4), 86(1), 71(3), 61(4), 57(18), 43(100), 41(7). (I stereoisomer)

MS (EI; 70 eV) 188(0) [M$^+$], 187(1), 145(46), 115(2), 97(45), 86(1), 71(4), 69(2), 57(19), 43(100), 41(7). (II stereoisomer)

HRMS calculated for C$_9$H$_{15}$O$_4$ (M-H)$^+$: 187.0970; found: 187.0967. $|\Delta|=1.6$ ppm.

2-Hexyltetrahydrofuran (2i) and 2-pentyltetrahydro-2H-pyran (2i'). The compounds were obtained from cyclisation of 1i (1 mmol). Final products were isolated by column chromatography (silica gel) eluting with petroleum ether:diethylether = 95:5 as colourless liquids (isolated yields: 2 and 62% respectively). Spectral data matched literature reference.$^3,11$

R$_f$ = 0.64 (Cyclohexane, EtOAc = 9/1) [THP-ether/9b]

R$_f$ = 0.52 (Cyclohexane, EtOAc = 9/1) [THF-ether/9b]

[THF-ether/2i] $^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 3.97-3.59 (m, 3H), 2.08-1.69 (m, 3H), 1.69-1.03 (m, 11H), 0.86 (t, $J = 6.8$ Hz, 3H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 79.59, 67.70, 35.89, 31.97, 31.51, 29.55, 26.50, 25.84, 22.73, 14.19.

MS (EI; 70 eV) 156(1) [M$^+$], 138 (1), 96 (1), 81(1), 71(100), 55(6), 43(31), 41(31).

[THP-ether/2i'] $^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 4.01-3.88 (m, 1H), 3.40 (td $J = 10.9$, 3.6 Hz, 1H), 3.29-3.08 (m, 1H), 2.17-1.04 (m, 14H), 0.87 (t, $J = 6.5$ Hz, 3H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 78.08, 68.63, 36.78, 32.12, 32.09, 26.40, 25.36, 23.76, 22.77, 14.19.

MS (EI; 70 eV) 156(1) [M$^+$], 138 (1), 95 (1), 85(100), 67(16), 57(15), 43(21), 41(24).

2-Phenyltetrahydro-2H-pyran. The compound was obtained from cyclisation of 1j (1 mmol). Final product was isolated by column chromatography (silica gel) eluting with petroleum ether:diethylether = 98:2 as colourless liquid (0.97 mmol, 97%). Spectral data matched literature reference.$^{12}$

R$_f$ = 0.33 (Cyclohexane, EtOAc = 9/1)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 7.39-7.15 (m, 5H), 4.36-4.24 (m, 1H), 4.12 (dd, $J = 11.4$, 3.5 Hz, 1H), 3.76-3.46 (m, 1H), 2.01-1.46 (m, 6H).
$^{13}$C NMR (CDCl$_3$, 50 MHz): 143.45, 128.38, 127.38, 125.95, 80.26, 69.11, 34.14, 26.02, 24.14.

MS (EI; 70 eV) 162(54) [M]$^+$, 161 (46), 144(2), 133(4), 129(4), 115 (6), 105 (100), 91(29), 77(45), 65(8), 55(26), 51(19), 41(31).

2-Methyl-2,3-dihydrobenzofuran. The compound was obtained from cyclisation of 11 (1 mmol). Final product was isolated by column chromatography (silica gel) eluting with petroleum ether:diethylether = 9:1 as a colorless liquid (0.31 mmol, 31%). Spectral data matched literature reference.$^{13}$

R$_f$ = 0.69 (Cyclohexane, EtOAc = 4/1)

$^1$H NMR (CDCl$_3$, 200 MHz): $^\delta$ 7.23-7.05 (m, 2H), 6.85 (dd, $J = 7.4$, 0.9 Hz, 1H), 6.77 (d, $J = 7.9$ Hz, 1H), 4.93 (ddq, $J = 8.7$, 7.7, 6.2 Hz, 1H), 3.32 (dd, $J = 15.4$, 8.8 Hz, 1H), 2.82 (dd, $J = 15.4$, 7.7 Hz, 1H), 1.48 (d, $J = 6.2$ Hz, 3H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $^\delta$ 159.63, 128.07, 127.15, 125.08, 120.28, 109.44, 76.52, 37.25, 21.88.

MS (EI; 70 eV) 134(100) [M]$^+$, 133(42), 119(66), 115(27), 113(34), 119(11), 107(100), 91(22), 77(25), 65(6), 51(13), 41(11).

2,2-Dimethylchromane. The compound was obtained from cyclisation of 1m (1 mmol). Final product was isolated by column chromatography (silica gel) eluting with petroleum ether:diethylether = 9:1 as a colorless liquid (0.63 mmol, 63%). Spectral data matched literature reference.$^{7,8}$

R$_f$ = 0.67 (Cyclohexane, EtOAc = 9/1)

$^1$H NMR (CDCl$_3$, 200 MHz): $^\delta$ 7.15-7.05 (m, 2H), 6.90-6.70 (m, 2H), 2.80 (t, $J = 6.7$ Hz, 2H), 1.83 (t, $J = 6.7$ Hz, 2H), 1.36 (s, 6H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $^\delta$ 153.48, 128.93, 126.73, 120.39, 119.08, 116.72, 73.56, 32.29, 26.37 (2), 21.94.

MS (EI; 70 eV) 162(11) [M]$^+$, 147(17), 133(17), 119(40), 107(100), 91(5), 77(8), 51(8), 41(9).

2-Methyl-2-(4-methylpent-3-enyl)chromane (2n) and cis/trans-1,1,4a-trimethyl-2,3,4,4a,9,9a-hexahydro-1H-xanthene (3n). The compounds were obtained from cyclisation of 1n (1 mmol). Final
products were isolated as single isomer (\textit{cis-3n}, white crystals) or as mixture of isomers (\textit{2n, trans-3n}, colourless oil) by column chromatography (silica gel) eluting with petroleum ether:diethyl ether with gradient elution from 10:0 to 9:1 (isolated yields: 13 and 65% respectively). Spectral data matched literature reference.\textsuperscript{14}

\[ R_f = 0.80 \text{ (Cyclohexane, EtOAc = 95/5)} \] [\textit{cis-3n}]

\[ R_f = 0.66 \text{ (Cyclohexane, EtOAc = 95/5)} \] [\textit{2n, trans-3n}]

\[ 2n \] \textsuperscript{1}H NMR (CDCl\textsubscript{3}, 200 MHz): \( \delta \) 7.06 (d, \( J = 7.3 \) Hz, 2H), 6.88-6.70 (m, 2H), 5.17-5.06 (m, 1H), 2.82-2.52 (m, 2H), 2.08-1.39 (m, 6H), 1.72 (s, 3H), 1.64 (s, 3H), 1.34 (s, 3H).

\[ 13C \text{ NMR (CDCl3, 50 MHz): } \delta 154.09, 129.50, 127.33, 124.38, 121.22, 119.64 117.41, 76.02, 39.66, 31.06, 25.80, 24.37, 22.40, 22.24, 17.71. \]

MS (EI; 70 eV) 230(52) [M]+

\[ [\textit{cis-3n}] \] \textsuperscript{1}H NMR (CDCl\textsubscript{3}, 200 MHz): \( \delta \) 7.05 (d, \( J = 7.1 \) Hz, 2H), 6.88-6.69 (m, 2H), 3.05 (dd, \( J = 17.7, 7.9 \) Hz, 1H), 2.76 (d, \( J = 17.7 \) Hz, 1H), 2.08-1.20 (m, 7H), 1.21 (s, 3H), 0.97 (s, 3H), 0.65 (s, 3H).

\[ 13C \text{ NMR (CDCl3, 50 MHz): } \delta 154.64, 129.04, 126.81, 122.18, 119.94, 117.23, 75.36, 44.58, 41.80, 39.71, 34.12, 32.41, 27.16, 23.74, 21.54, 18.23. \]

MS (EI; 70 eV) 230(31) [M]+

\[ [\textit{trans-3n}] \] \textsuperscript{1}H NMR (CDCl\textsubscript{3}, 200 MHz): \( \delta \) 7.09 (d, \( J = 7.1 \) Hz, 2H), 6.81-6.72 (m, 2H), 2.73 (dd, \( J = 16.2, 6.7 \) Hz, 1H), 2.57 (d, \( J = 16.4 \) Hz, 1H), 2.17-1.16 (m, 7H), 1.23 (s, 3H), 1.04 (s, 3H), 0.94 (s, 3H).

\[ 13C \text{ NMR (CDCl3, 50 MHz): } \delta 154.38, 129.75, 127.23, 122.72, 119.74, 117.15, 76.29, 48.19, 41.63, 40.13, 33.49, 32.22, 23.37, 20.80, 19.96, 19.91. \]

MS (EI; 70 eV) 230(54) [M]+

2-Methyl-2-(2-(2,6,6-trimethylcyclohex-1-en-1-yl)ethyl)tetrahydrofuran. The compound was obtained from cyclisation of \textit{1o} (1 mmol). Final product was isolated as a mixture of isomers, \textit{2o} being the major isomer, by column chromatography (silica gel) eluting with petroleum ether:diethyl ether = 9 : 1 as a colourless liquid (0.71 mmol, 71%). Spectral data matched literature reference.\textsuperscript{15}

\[ R_f = 0.76 \text{ (Cyclohexane, EtOAc = 4/1)} \]

\textsuperscript{1}H NMR (CDCl\textsubscript{3}, 200 MHz): \( \delta \) 3.97-3.68 (m, 1H), 2.17-1.33 (m, 14H), 1.59 (s, 1H), 1.21 (s, 1H), 0.98 (s, 1H).
$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 137.12, 126.90, 82.88, 67.20, 41.13, 40.05, 36.83, 35.22, 32.92, 28.78, 26.24, 25.48, 23.63, 19.84, 19.69.

MS (EI; 70 eV) 236(2) $[M]^{+}$., 221(2), 203(2), 177(4), 161(2), 149(2), 136(18), 123(15), 121(25), 107(15), 95(17), 93(17), 85(100), 81(14), 69(9), 67(10), 55(14), 43(52), 41(24).

1,7-Dimethoxy-3,7-dimethyloctane$^9$

R$_f$ = 0.42 (cyclohexane, EtOAc = 8/2)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 3.39 (t, $J$=6.7 Hz, 2H), 3.32 (s, 3H), 3.16 (s, 3H), 1.79-1.20 (m, 9H), 1.12 (s, 6H), 0.88 (d, $J$=6.4 Hz, 3H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 74.6, 71.2, 58.6, 49.1, 40.1, 37.7, 36.7, 30.2, 21.2, 19.6.

MS (EI; 70 eV) 202(0) $[M]^{+}$., 187(4), 138(11), 123(29), 109(14), 95(50), 81(95), 73(100), 55(94).

7-Ethoxy-1-methoxy-3,7-dimethyloctane$^9$

R$_f$ = 0.45 (cyclohexane, EtOAc = 8/2)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 3.50-3.25 (m, 7H); 1.70-1.00 (m, 18H); 0.82 (d, $J$=6.4 Hz, 3H).

MS (EI; 70 eV) 216(0) $[M]^{+}$., 87(100), 59(59), 55(9), 45(13), 43(13), 41(9).

1-Methoxy-7-propyloxy-3,7-dimethyloctane$^9$

R$_f$ = 0.57 (cyclohexane, EtOAc = 8/2)
$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 3.39 (t, $J$=6.7 Hz, 2H), 3.31 (s, 3H), 3.23 (t, $J$=6.7 Hz, 3H), 1.80-1.10 (m, 11H), 1.06 (s, 6H), 0.90-0.75 (m, 6H).

MS (EI; 70 eV) 230(0) [M]$^+$, 215(1), 101(74), 83(9), 69(7), 59(100), 55(11), 45(16), 43(21), 41(14).

7-Butoxy-1-methoxy-3,7-dimethyloctane$^9$

R$_f$ = 0.60 (cyclohexane, EtOAc = 8/2)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 3.40-3.10 (m, 7H); 1.70-1.00 (m, 22H); 0.82 (d, $J$=6.4 Hz, 3H).

MS (EI; 70 eV) 244(0) [M]$^+$, 229 (1), 115(55), 83(7), 69(7), 59(100), 55(10), 45(15), 43(10), 41(16).

2-Methoxy-2-methyldecane

R$_f$ = 0.47 (cyclohexane, EtOAc = 98/2)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 3.17 (s, 3H), 1.52-1.37 (m, 2H), 1.37-1.20 (m, 12H), 1.12 (s, 6H), 0.88 (d, $J$=6.4 Hz, 3H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 74.79, 49.20, 39.97, 32.06, 30.41, 29.79, 29.47, 25.12 (2), 24.03, 22.82, 14.26.

MS (EI; 70 eV) 186(0) [M]$^+$, 171(3), 73(100), 69(4), 55(8), 43(10).

2-Ethoxy-2-methyldecane

R$_f$ = 0.50 (cyclohexane, EtOAc = 98/2)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 3.29 (q, $J$=7.0 Hz, 2H), 1.45-1.30 (m, 2H), 1.30-1.15 (m, 12H), 1.08 (t, $J$=7.0 Hz, 3H), 1.07 (s, 6H), 0.81 (d, $J$=6.4 Hz, 3H).
$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 74.59, 56.38, 40.29, 32.06, 30.40, 29.79, 29.47, 25.85 (2), 24.06, 22.82, 16.36, 14.25.

MS (EI; 70 eV) 200(0) [M]$^+$, 185(3), 87(100), 59(57), 43(16).

2-Propyloxy-2-methylnonane

R$_f$ = 0.70 (cyclohexane, EtOAc = 9/1)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 3.25 (t, $J$=6.8 Hz, 2H), 1.66-1.38 (m, 4H), 1.38-1.19 (m, 10H), 1.13 (s, 6H), 0.90 (t, $J$=7.3 Hz, 3H), 0.88 (d, $J$=6.4 Hz, 3H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 74.39, 62.93, 40.44, 32.04, 30.38, 29.51, 25.78 (2), 24.05, 23.99, 22.83, 14.26, 10.94.

MS (EI; 70 eV) 200(0) [M]$^+$, 171(4), 87(100), 69(13), 59(64), 43(19).

2-Butyloxy-2-methylnonane

R$_f$ = 0.70 (cyclohexane, EtOAc = 9/1)

$^1$H NMR (CDCl$_3$, 200 MHz): $\delta$ 3.28 (t, $J$=6.4 Hz, 2H), 1.61-1.34 (m, 6H), 1.34-1.18 (m, 10H), 1.12 (s, 6H), 1.01-0.81 (m, 6H).

$^{13}$C NMR (CDCl$_3$, 50 MHz): $\delta$ 74.37, 60.91, 40.43, 32.96, 32.04, 30.38, 29.50, 25.76 (2), 24.04, 22.83, 19.65, 14.25, 14.13.

MS (EI; 70 eV) 214(0) [M]$^+$, 199(1), 173(22), 115(95), 101(68), 85(28), 71(31), 57(84), 43(100).

2,2-Dimethyl-4-(2-methylnonan-2-yloxy)methyl)-1,3-dioxolane
$^1\text{H} \text{NMR (CDCl}_3, \text{ 200 MHz)}$: $\delta$ 4.18-4.11 (m, 1H), 4.10-4.01 (m, 1H), 3.80-3.69 (m, 1H), 3.50-3.38 (m, 1H), 3.32-3.19 (m, 1H), 1.52-1.38 (m, 5H), 1.36 (s, 3H), 1.30-1.18 (m, 10H), 1.13 (s, 6H), 0.88 (t, J=6.4 Hz, 3H).

$^{13}\text{C} \text{NMR (CDCl}_3, \text{ 50 MHz)}$: $\delta$ 109.25, 75.36, 75.14, 67.68, 62.98, 40.35, 32.03, 30.32, 29.49, 26.95, 25.62, 25.56 (2), 24.01, 22.82, 14.25.

MS (El; 70 eV) 272(0) [M]$^+$, 257(8), 173(19), 115(84), 101(65), 85(25), 71(28), 57(77), 43(100).
6. Recycling studies

![Chemical reaction diagram]

Figure S4. Recycling studies of Fe-MMT catalyst

7. Hot filtration experiment

![Graph showing yield vs time for Fe-MMT and Bi-MMT]

Figure S5. Hot filtration experiments with Fe-MMT and Bi-MMT. In dashed lines are the expected yields for each system at completion of the reaction. Filtration performed at 80 °C over porosity 4 fritted glass.
8. NMR spectra
THF-ether

2i
\[ 21 \]
$^1$H NMR (500 MHz, CDCl$_3$) of 2rc