SUPPORTING INFORMATION

Palladium nanoparticles stabilised by cinchona-based alkaloids in glycerol: efficient catalysts for surface assisted processes
A. Reina, C. Pradel, E. Martin, E. Teuma and M. Gómez

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General Experimental Part

Unless otherwise stated, all chemical reagents were obtained from commercial suppliers and used without further purification. All manipulations were performed using standard Schlenk techniques under argon atmosphere. Glycerol was dried under vacuum at 80 °C for 18h prior to use. NMR spectra were recorded on a Bruker Advance 300 spectrometer at 293 K (300 MHz for $^1$H NMR, 75.5 MHz for $^{13}$C NMR and 50.6 MHz for $^{15}$N NMR). GC analyses were carried out on a GC Perkin Elmer Clarus 500 with ionization flame detector, using a SGE BPX5 column composed by 5% phenylmethylsiloxane and a Perkin Elmer Clarus MS560 mass detector. The determination of enantiomeric excesses was carried out by GC analyses on a HP 6890 Series GC instrument with a FS-cyclodex beta I/P chiral column. TEM images of particles in the solid state and dispersed in glycerol were obtained from transmission electron microscopes JEOL JEM 1400 running at 120 kV and HR-TEM from JEOL JEM 2100F running at 200 kV equipped with X PGT (detection of light elements, resolution 135 eV). The nanoparticles size, distribution and average diameter were determined from TEM images with Image-J software associated to a Microsoft Excel macro developed by Christian Pradel. XPS experiments were performed in a PHI 5500 Multi-technique System (from Physical Electronics) with a monochromatic X-ray source (Al $K\alpha$ line of 1486.6 eV energy, 350 W), placed perpendicular to the analyser axis and calibrated using the 3$d_{5/2}$ line of Ag with a full width at half maximum (FWHM) of 0.8 eV. The analysed area was a circle of 0.8 mm diameter. The selected resolution for the survey spectra was 187.85 eV of Pass Energy and 0.8 eV/step, while a Pass Energy of 23.5 eV and 0.1 eV/step was used for the high-resolution spectra of the main orbitals of the different elements. In order to avoid possible contamination in the analysis chamber, samples remained long times (> 12 h) in a pre-chamber under high vacuum (ca. 10^{-7} mbar). As a result of it, little but perceptible optical changes were noticed in samples, and low and high-resolution analyses could be performed working on ultra-high vacuum (under 10^{-9} mbar) without evaporation of the liquid phase. IR spectra were recorded in the range of 4000-400 cm^{-1} on a Varian 640-IR FTIR Spectrometer. The powder X-ray diffraction patterns were collected on a XPert (Theta-Theta mode) Panalytical diffractometer with $\alpha$(Cu $K\alpha_1$, $K\alpha_2$)=1.54060, 1.54443 Å. High-pressure reactions were carried out in a Top Industrie Autoclave suitable from 0-50 bar and from 15-150 °C. Elemental and ICP-AES analyses were carried out at the “Service d’Analyse” of Laboratoire de Chimie de Coordination (Toulouse) using a Perkin Elmer 2400 series II analyser and an iCAP 6300 ICP Spectrometer.
Table S1. TEM images of PdxL nanoparticles stabilised by alkaloids (cinchonidine and quinidine) in glycerol, using different palladium precursors ($x = I$, Pd(OAc)$_2$; $x = II$, [PdCl$_2$(cod)]; $x = III$, [Pd$_3$(dba)$_3$])

<table>
<thead>
<tr>
<th>Stabiliser (right)/Pd precursor (bottom)</th>
<th>Cinchonidine (a)</th>
<th>Quinidine (d)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pd(OAc)$_2$ (I)</td>
<td>Not well-dispersed nanoparticles, tending to be aggregated:</td>
<td>Well-dispersed nanoparticles, quite homogeneous in size:</td>
</tr>
<tr>
<td></td>
<td><img src="image1.png" alt="TEM image" /></td>
<td><img src="image2.png" alt="TEM image" /></td>
</tr>
<tr>
<td></td>
<td>$d_{mean} = 2.1 \pm 0.6 \text{ nm (1759 particles)}$</td>
<td>$d_{mean} = 1.4 \pm 0.3 \text{ nm (954 particles)}$</td>
</tr>
<tr>
<td>[PdCl$_2$(cod)] (II)</td>
<td>Not well-dispersed nanoparticles, tending to be aggregated:</td>
<td>Well-dispersed nanoparticles, quite homogeneous in size:</td>
</tr>
<tr>
<td></td>
<td><img src="image3.png" alt="TEM image" /></td>
<td><img src="image4.png" alt="TEM image" /></td>
</tr>
<tr>
<td></td>
<td>$d_{mean} = 1.6 \pm 0.3 \text{ nm (642 particles)}$</td>
<td></td>
</tr>
<tr>
<td>[Pd$_3$(dba)$_3$] (III)</td>
<td>Well-dispersed nanoparticles ($d_{mean} = 1.5 \pm 0.3 \text{ nm (1524 particles)}$), together with agglomerates</td>
<td>No colloidal dispersion, only precipitate</td>
</tr>
<tr>
<td></td>
<td><img src="image5.png" alt="TEM image" /></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table S2. Hydrogenation of compounds containing C=C or C≡C bonds and nitroarenes catalysed by Pdla.\textsuperscript{a}

<table>
<thead>
<tr>
<th>Entry</th>
<th>Substrate</th>
<th>Product</th>
<th>Pd mol% (pH\textsubscript{2})</th>
<th>Conv. (yield)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td><img src="image1.png" alt="Substrate 1" /></td>
<td><img src="image2.png" alt="Product 1" /></td>
<td>0.1\textsuperscript{c} (1)</td>
<td>&gt;99 (96)</td>
</tr>
<tr>
<td>2</td>
<td><img src="image1.png" alt="Substrate 2" /></td>
<td><img src="image2.png" alt="Product 2" /></td>
<td>0.1\textsuperscript{c} (1)</td>
<td>86 (80)</td>
</tr>
<tr>
<td>3</td>
<td><img src="image1.png" alt="Substrate 3" /></td>
<td><img src="image2.png" alt="Product 3" /></td>
<td>1 (1)</td>
<td>83 (9/1)\textsuperscript{d}</td>
</tr>
<tr>
<td>4</td>
<td><img src="image1.png" alt="Substrate 4" /></td>
<td><img src="image2.png" alt="Product 4" /></td>
<td>1 (3)</td>
<td>99 (93)\textsuperscript{e}</td>
</tr>
<tr>
<td>5</td>
<td><img src="image1.png" alt="Substrate 5" /></td>
<td><img src="image2.png" alt="Product 5" /></td>
<td>1 (1)</td>
<td>95 (92)</td>
</tr>
<tr>
<td>6</td>
<td><img src="image1.png" alt="Substrate 6" /></td>
<td><img src="image2.png" alt="Product 6" /></td>
<td>1 (1)</td>
<td>99 (96)</td>
</tr>
<tr>
<td>7</td>
<td><img src="image1.png" alt="Substrate 7" /></td>
<td><img src="image2.png" alt="Product 7" /></td>
<td>1 (10)</td>
<td>53 (50)</td>
</tr>
<tr>
<td>8</td>
<td><img src="image1.png" alt="Substrate 8" /></td>
<td><img src="image2.png" alt="Product 8" /></td>
<td>1 (1)</td>
<td>&gt;99 (94)</td>
</tr>
<tr>
<td>9</td>
<td><img src="image1.png" alt="Substrate 9" /></td>
<td><img src="image2.png" alt="Product 9" /></td>
<td>0.1\textsuperscript{c} (1)</td>
<td>47 (44)\textsuperscript{f}</td>
</tr>
<tr>
<td>10</td>
<td><img src="image1.png" alt="Substrate 10" /></td>
<td><img src="image2.png" alt="Product 10" /></td>
<td>1 (1)</td>
<td>&gt;99 (96)</td>
</tr>
<tr>
<td>11</td>
<td><img src="image1.png" alt="Substrate 11" /></td>
<td><img src="image2.png" alt="Product 11" /></td>
<td>0.5\textsuperscript{g} (1)</td>
<td>80 (84)\textsuperscript{h}</td>
</tr>
<tr>
<td>12</td>
<td><img src="image1.png" alt="Substrate 12" /></td>
<td><img src="image2.png" alt="Product 12" /></td>
<td>1 (3)</td>
<td>98 (70)\textsuperscript{i,k}</td>
</tr>
<tr>
<td>13</td>
<td><img src="image1.png" alt="Substrate 13" /></td>
<td><img src="image2.png" alt="Product 13" /></td>
<td>0.5\textsuperscript{g} (1)</td>
<td>97 (93)\textsuperscript{j}</td>
</tr>
</tbody>
</table>
Results from duplicated experiments. Reaction conditions: 1 mmol of substrate (2-16) and 1 mL of the catalytic glycerol solution of PdIa (10⁻² mol⁻¹, 0.01 mmol of total Pd). 

- Determined by GC and GC-MS using decane as internal standard.
- 10 mmol of substrate for 1 mL of catalytic solution (0.01 mmol of total Pd).
- In brackets, 4H/4I ratio.
- For 12h.
- At 30 °C for 24h.
- 5 mmol of substrate for 1 mL of catalytic solution (0.01 mmol of total Pd).
- 16% of 9H was detected.
- Determined by GC and GC-MS using cyclooctane as internal standard.
- At 35 °C for 6h.

\[ \text{For } 12h. \]
\[ \text{At } 30^\circ \text{C for } 24h. \]
\[ \text{5 mmol of substrate for } 1 \text{ mL of catalytic solution (0.01 mmol of total Pd).} \]
\[ \text{16\% of } 9H \text{ was detected.} \]
\[ \text{Determined by GC and GC-MS using cyclooctane as internal standard.} \]
\[ \text{At } 35^\circ \text{C for } 6h. \]
\[ \text{25\% of } 10\text{HE} \text{ was detected.} \]
Scheme S1. Hydrogenation (top) and hydrodeboronation (bottom) catalysed by PdIdH. Conversions and yields were determined by GC and GC-MS using decane as internal standard.

Scheme S2. Hydrogenation of ketones (top) and aldehydes (bottom) catalysed by PdIId. Conversions and yields were determined by GC and GC-MS using decane as internal standard.

Scheme S3. Pd-catalysed hydrogenation of N-benzylideneaniline (24) by PdIa and PdIId. Conversions and yields were determined by GC and GC-MS using decane as internal standard.
Scheme S4. Pathway of Pd-catalysed synthesis of secondary and tertiary amines from aldehyde and amine reagents under hydrogen pressure, showing the formation of imine and iminium ion intermediates.
Figure S1. TEM images in glycerol corresponding to PdIa, PdIb and PdIc after centrifugation (isolation of PdNPs at the solid state) and redispersion in glycerol.

Figure S2. TEM images for PdIa and PdId after centrifugation and redispersion in ethanol (analyses at solid state).

PdIa + d $\rightarrow_{\text{glycerol}}^{\text{Pd/L = 1/1}}$ PdId

Pd(OAc)$_2$ + d $\rightarrow_{\text{glycerol}}^{\text{Pd/L = 1/1}}$ PdId

$\text{Pd} = 10^{-2} \text{ mol/L}$

80 °C, overnight

Figure S3. TEM images for PdId working at different dihydrogen pressures.
Figure S4. TEM images for PdId working at different temperatures.
Figure S5. TEM images for PdId working at different metal concentrations.
Figure S6. Exchange ligand reaction between Pd1L (L = a, d) and dodecanethiol (a). $^1$H NMR spectra (300 MHz, CDCl$_3$, 298 K) corresponding to the extracted organic product from the reaction involving Pd1a (b) and Pd1d (c). * Denotes CHCl$_3$. 
Figure S7. $^1$H NMR spectra (300 MHz, CDCl$_3$, 298 K) for commercially available cinchonidine (top) and quinidine (bottom).

Figure S8. TEM images in glycerol of PdNPs stabilised by dH (PdIdH).

$d_{\text{mean}} = 2.5 \pm 0.9$

(1283 nanoparticles)
(a) \[ \text{Pd} \text{L} \xrightarrow{\text{THF-d}_8, 298 \text{ K}} \text{cyclooctane} \rightarrow \]

(b) 

\[ t = 0 \]

\[ t = 1 \text{h} \]

Pd/H = 2/1
Figure S9. (a) Titration of hydrides present on Pd1L (s) (L = a, d) in THF-d$_8$ using maleic anhydride and cyclooctane as internal standard. $^1$H NMR spectra (300 MHz, CDCl$_3$, 298 K) for PdLa (b) and Pdld (c).
Figure S10. (a): IR spectra (KBr pellets) for cinchona-based stabilisers (a, d) with the corresponding PdNPs (Pdla, Pddl); (b) IR region corresponding to the double bonds and quinuclidine fragment. The intense absorption at ca. 1620 cm$^{-1}$ for the free ligands a and d corresponds to the bending of water.
Figure S11. XRD diffractograms (red traces) for PdIa (left) and PdId (right) showing the peaks corresponding to fcc structure. Sharp lines correspond to the pattern of bulk fcc Pd(0).

Figure S12. XPS survey spectra for PdIa (top) and PdId (bottom) at solid state. Insets correspond to high-resolution XPS spectra of Pd 3d binding region.
Figure S13. High-resolution XPS spectra: (a) Pd 3d binding energy region for **Pdla** and **Pdlid** at the solid state; the corresponding spectrum for Pd(OAc)$_2$ is included for comparison purposes. (b) N 1s binding region for **Pdla** and **Pdlid**.
**Figure S14.** High-resolution XPS spectra in the N1s binding energy region for cinchonidine (a) and quinidine (d) at the solid state.

**Figure S15.** XPS survey spectra for PdIa (top) and PdId (bottom) in glycerol.
**Figure S16.** Heteronuclear Multiple Bond Correlation (HMBC) NMR experiment between $^1$H and $^{15}$N for quinidine (d) in a) CD$_3$OD; b) CD$_3$OD/glycerol (1:1) and c) THF-d$_8$/glycerol (1/1) at room temperature. Arrows show the quinoline and quinuclidine nitrogen atoms.

**Figure S17.** TEM images after the first (left) and the fourth (right) recycling of the hydrogenation of 4-phenylbut-3-en-2-one catalysed by Pdld (see Table 1 and Fig. 4 in the main text).
Figure S18. (a) $^1$H NMR spectrum (300 MHz, CDCl$_3$, 298 K) corresponding to the product 12HE; (b) NOESY-NMR analysis of 12HE showing the exclusive formation of the ($Z$)-stereoisomer. Arrows indicate the NOE contact between both ethylenic hydrogens. * Denotes CH$_2$Cl$_2$. 
(a) 

\[
\text{O} \quad \text{H}_2 (3 \text{ bar}) \\
\text{glycerol} \\
80 \text{ ºC}, 2\text{h}
\]

\[ \text{A} \rightarrow \text{AH} \]

<table>
<thead>
<tr>
<th>Entry</th>
<th>Catalyst</th>
<th>$\text{pH}_2$</th>
<th>$T (\text{º C})$</th>
<th>$t (\text{h})$</th>
<th>Conv. (% yield) &amp; ($\text{ee} %$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>PdIa</td>
<td>3</td>
<td>100</td>
<td>6</td>
<td>90 (91) &amp; 0</td>
</tr>
<tr>
<td>2</td>
<td>PdId</td>
<td>3</td>
<td>100</td>
<td>6</td>
<td>99 (94) &amp; 0</td>
</tr>
<tr>
<td>3</td>
<td>PdIa</td>
<td>3</td>
<td>40</td>
<td>6</td>
<td>32 (29) &amp; &lt;5</td>
</tr>
<tr>
<td>4</td>
<td>PdId</td>
<td>3</td>
<td>40</td>
<td>6</td>
<td>88 (84) &amp; &lt;5</td>
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<tr>
<td>6</td>
<td>PdId</td>
<td>10</td>
<td>40</td>
<td>6</td>
<td>&gt;99 (95) &amp; &lt;5</td>
</tr>
<tr>
<td>7</td>
<td>PdId</td>
<td>15</td>
<td>40</td>
<td>6</td>
<td>&gt;99 (94) &amp; &lt;5</td>
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</table>

At milder conditions, no conversion was obtained.

(b) 

\[
\text{CH}_3 \text{CO} \\
\text{H}_2 (20 \text{ bar}) \\
\text{glycerol} \\
80 \text{ ºC}, 24\text{h}
\]

\[ \text{B} \rightarrow \text{BH} \]

<table>
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<tr>
<th>Entry</th>
<th>Catalyst</th>
<th>$\text{pH}_2$</th>
<th>$T (\text{º C})$</th>
<th>$t (\text{h})$</th>
<th>Conv. (% yield) &amp; ($\text{ee} %$)</th>
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<tbody>
<tr>
<td>1</td>
<td>PdIa</td>
<td>10</td>
<td>40</td>
<td>6</td>
<td>&gt;99 (95) &amp; &lt;5</td>
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<tr>
<td>2</td>
<td>PdId</td>
<td>15</td>
<td>40</td>
<td>6</td>
<td>&gt;99 (94) &amp; &lt;5</td>
</tr>
</tbody>
</table>

At milder conditions, no conversion was obtained.

(c) 

\[
\text{OH} \\
\text{glycerol} \\
T (\text{º C}), t (\text{h})
\]

\[ \text{C} \rightarrow \text{CH} \]

<table>
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<tr>
<th>Entry</th>
<th>Catalyst</th>
<th>$\text{pH}_2$</th>
<th>$T (\text{º C})$</th>
<th>$t (\text{h})$</th>
<th>Conv. (% yield) &amp; ($\text{ee} %$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>PdIa</td>
<td>3</td>
<td>100</td>
<td>6</td>
<td>90 (86) &amp; 0</td>
</tr>
<tr>
<td>2</td>
<td>PdId</td>
<td>3</td>
<td>100</td>
<td>6</td>
<td>99 (94) &amp; 0</td>
</tr>
<tr>
<td>3</td>
<td>PdIa</td>
<td>3</td>
<td>40</td>
<td>6</td>
<td>32 (29) &amp; &lt;5</td>
</tr>
<tr>
<td>4</td>
<td>PdId</td>
<td>3</td>
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<td>6</td>
<td>88 (84) &amp; &lt;5</td>
</tr>
<tr>
<td>6</td>
<td>PdId</td>
<td>10</td>
<td>40</td>
<td>6</td>
<td>&gt;99 (95) &amp; &lt;5</td>
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<tr>
<td>7</td>
<td>PdId</td>
<td>15</td>
<td>40</td>
<td>6</td>
<td>&gt;99 (94) &amp; &lt;5</td>
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</table>

Results from duplicated experiments. Reaction conditions: 1 mmol of substrate (A-C) and 1 mL of the catalytic glycerol solution of PdIa (10-2 mol.L-1, 0.01 mmol of total Pd). $^b$ Determined by GC and GC-MS using decane as internal standard. $^c$ Enantiomeric excess determined by chiral GC using a FS-Cyclodex beta I/P column. The addition of L-proline did not favour the asymmetric induction.

Figure S19. Pd-catalysed hydrogenation of prochiral substrates dimethyl itaconate A (a), ethyl pyruvate B (b) and isophorone C (c) by PdIa and PdId. Conversions and yields were determined by GC and GC-MS using decane as internal standard and enantiomeric excesses were determined by chiral GC (FS-Cyclodex beta I/P column).
Figure S20. $^1$H NMR spectrum (300 MHz, CDCl$_3$, 298 K) corresponding to the extraction of stabiliser from the catalytic glycerol phase PdId, after hydrodehalogenation of 4-bromophenol.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 1H.
$^{13}$C[1H] Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 1H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 2H.
$^{13}$C$^1$H Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 2H.
GC-MS chromatogram for 3H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 4H.
$^{13}$C$(^1$H$)$ Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 4H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 5H.
$^{13}$C\(^1\)H Jmod NMR (75 MHz, 298 K) spectrum in CDCl\(_3\) for \textbf{5H}. 
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 6H.
$^{13}$C($^1$H) Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 6H.
GC-MS chromatogram for 7H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 8H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 9H.
$^{13}\text{C}^{(1)}\text{H}$ Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 9H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 9HE.
$^{13}\text{C}^{[1\text{H}]}$ Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 9HE.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 10H.
$^{13}$C(¹H) Jmod NMR (75 MHz, 298 K) spectrum in CDCl₃ for 10H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for **10HE**.
$^{13}$C{H} Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 10HE.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 11H.
$^{13}\text{C}[^{1}H]$ Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 11H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 11HE.
$^{13}\text{C}({}^1\text{H})$ Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for **11HE**.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 12H.
$^{13}$C{$^1$H} Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 12H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 12HE.
\(^{13}\text{C}^{1\text{H}}\) Jmod NMR (75 MHz, 298 K) spectrum in CDCl\(_3\) for 12HE.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 13H.
$^{13}$C$^{1}$H Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 13H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 14H.
$^{13}$C{\(^1\)H} Jmod NMR (75 MHz, 298 K) spectrum in CDCl\(_3\) for 14H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 15H.
$^{13}$C($^1$H) NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 15H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 16H.
$^{13}$C$^{1}$H Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 16H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 17H.
$^{13}$C($^1$H) Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 17H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 20H.
$^{13}$C$^1$H Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 20H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 20e.
$^{13}\text{C}[^1\text{H}]$ Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 20e.
GC-MS chromatogram for 20h.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 20g.
$^{13}\text{C}^{[1]}\text{H}$ Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 20g.
GC-MS chromatogram for 20g.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 21e.
$^{13}$C{\textsuperscript{1}}H Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 21e.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 21g.
$^{13}$C$^1$H Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 21g.
GC-MS chromatogram for 21g.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 21h.
$^{13}$C\(^{(1)}\) J-mod NMR (75 MHz, 298 K) spectrum in CDCl\(_3\) for 21h.
GC-MS chromatogram for 21h.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 22e.
GC-MS chromatogram for 22e.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 22f.
$^{13}$C{^1}H Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 22f.
GC-MS chromatogram for 22f.
¹H NMR (300 MHz, 298 K) spectrum in CDCl₃ for 22g.
$^{13}C(\text{H})$ Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 22g.
GC-MS chromatogram for 22g.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 24H.
$^{13}$C(1H) Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 24H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 24Im.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 25H.
$^1$H J-mod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 25H.
GC-MS chromatogram for 25H.
GC-MS chromatogram for 25Im.
$^{13}$C(J) NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 26H.
GC-MS chromatogram for 26H.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 27H.
$^{13}$C\(^{1}H\) Jmod NMR (75 MHz, 298 K) spectrum in CDCl\(_3\) for 27H.
GC-MS chromatogram for 27H.
GC-MS chromatogram for 27Im.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 33.
$^{13}$C$^1$H Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 33.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 34.
$^{13}$C($^1$H) Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 34.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for 35.
$^{13}$C$^1$H Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for 35.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for AH.
$^{13}$C$^1$H Jmod NMR (75 MHz, 298 K) spectrum in CDCl$_3$ for AH.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for BH.
$^1$H NMR (300 MHz, 298 K) spectrum in CDCl$_3$ for CH.
GC-MS chromatogram for CH.