# Efficient telemerization of 1,3-butadiene with alcohols in the presence of in situ generated palladium(0)carbene complexes

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## Abstract

The palladium-catalyzed telemerization of 1,3-butadiene with alcohols has been studied in presence of palladium and imidazolium salts, which form in situ carbene ligands. Among the different imidazolium salts tested 1,3-dimesitylimidazolium chloride (**7**), 1,3-bis(ferrocenylmethyl)benzimidazolium tetraphenylborate (**12**) and 1,3-bis(2-ferrocenylethyl)benzimidazolium bromide (**13**) gave the best yields of the desired octadienyl ethers. Significantly improved regioselectivities for the linear octadienylethers are obtained in the reaction of 1,3-butadiene and methanol compared to the previously optimized palladium/triphenylphosphine catalyst. Using *n*-butanol and *iso*-propanol the palladium carbene catalysts lead to a considerable increase in the corresponding telemerization products compared to standard palladium/triphenylphosphine catalysts. © 2002 Elsevier Science B.V. All rights reserved.

*Keywords:* Palladium; Homogeneous catalysis; Telemerization; Octadienyl ethers; Carbenes

## 1. Introduction

An important challenge in catalysis is the development of "clean" and practical technologies for existing and new industrial products. As far as possible the desired compound should be synthesized from cheap available raw materials with high selectivity and avoiding waste or by-products. The control of product selectivity can be achieved, for example, by employing an appropriate catalyst and by adjusting the crucial reaction parameters.

During the course of our studies [1] into palladium-catalyzed C–C coupling reactions, we became interested in the telemerization of 1,3-butadiene. This reaction assembles simple starting materials in a 100% atom efficient [2] manner to give octadienes [3]. In general, the telemerization reaction is the dimerization of two molecules of a 1,3-diene in the presence of an appropriate nucleophile HX, e.g. alcohols [4], to give substituted octadienes (1-substituted 2,7-octadiene, 3-substituted 1,7-octadiene). The resulting compounds are useful as intermediates in the total synthesis of several natural products [5] as well as in industry, as precursors for plasticizer alcohols [6], important monomers, solvents, corrosion inhibitors and non-volatile herbicides [7].

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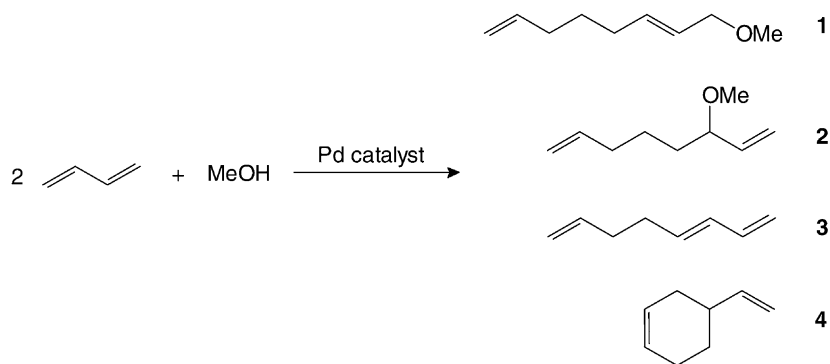
Scheme 1.

From an industrial point of view, 1,3-butadiene and methanol are the most attractive starting materials for this reaction due to their ready availability and their exceedingly low price.[1] Apart from the desired product 1-methoxy-2,7-octadiene (**1**), the 3-substituted 1,7-octadiene (**2**) (*iso*-product), 1,3,7-octatriene (**3**) (formed by the linear dimerization of butadiene) and less importantly 4-vinylcyclohexene (**4**) (formed by the Diels–Alder reaction of two molecules of butadiene) are observed as major by-products in this reaction (Scheme 1).

The mechanism of the palladium-catalyzed telomerization reaction has been carefully examined (Scheme 2) [8]. Recently, we were able to show that the main principle which governs the *n*/*iso* (l/b) selectivity of the octadienyl products is an internal coordination of the olefinic side-chain. Hence, one should avoid a large excess of coordinating ligands. Unfortunately using phosphines as ligands it is necessary to use an excess of phosphine in order to stabilize the palladium catalyst appropriately at low catalyst concentration.

We envisioned that a more strongly bound and sterically hindered ligand on the palladium center should give a productive and more selective catalyst system compared to the known palladium/phosphine catalysts. Obviously carbene ligands fulfill these requirements and have been proven successful for a number of palladium-catalyzed coupling reactions

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[1] The current industrial price for butadiene is in the range 0.55–0.65 DM/kg; mixtures of butadiene with other $C_4$-olefins are even cheaper.
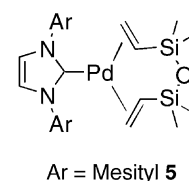
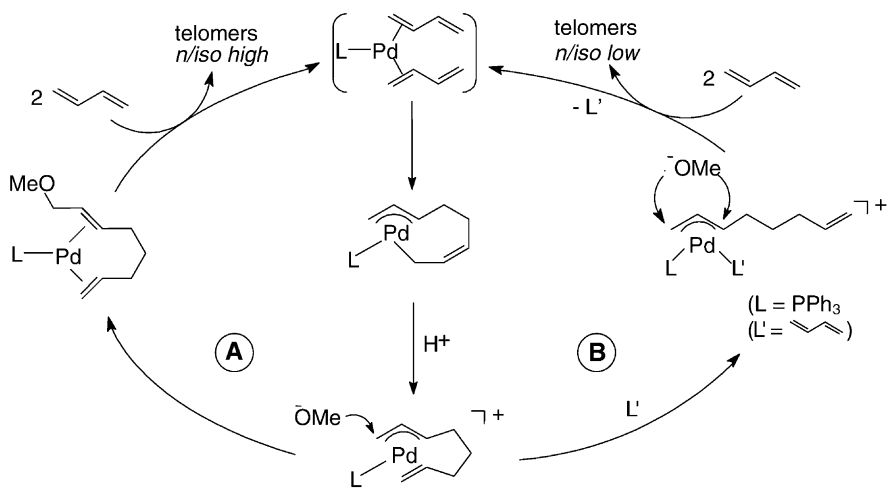

Ar = Mesityl **5**

Fig. 1.

such as the Heck, Suzuki and Sonogashira reaction, copolymerizations and amination of chloroarenes [9]. Recently, we succeeded in synthesizing defined monocarbenepalladiumdiolefin complexes such as 1,3-dimesitylimidazol-2-ylidene-palladium(0)-$\eta^2$ + $\eta^2$-1,1,3,3-tetramethyl-1,3-divinyl-disiloxane (**5**) (dmiPd(0)dvds) (Fig. 1), which show remarkable catalyst productivities and selectivities for different telomerization reactions [10].

A drawback of this class of catalysts is the laborious multi-step synthesis of the corresponding complex. In this paper, we describe for the first time the use of simple in situ generated palladium carbene catalysts for the telomerization of 1,3-butadiene with methanol and other alcohols.

## 2. Results and discussion

As a starting point of our investigation, we tested several imidazolium salts (**6**–**13**) (Fig. 2) in the presence of Pd(OAc)$_2$ and Pd(dba)$_2$ at 90 °C. 1,3-Dimesitylimidazolium chloride (**7**), 1,3-bis(ferrocenyl)imidazolium tetraphenylborate (**8**), 1-ferrocenyl-
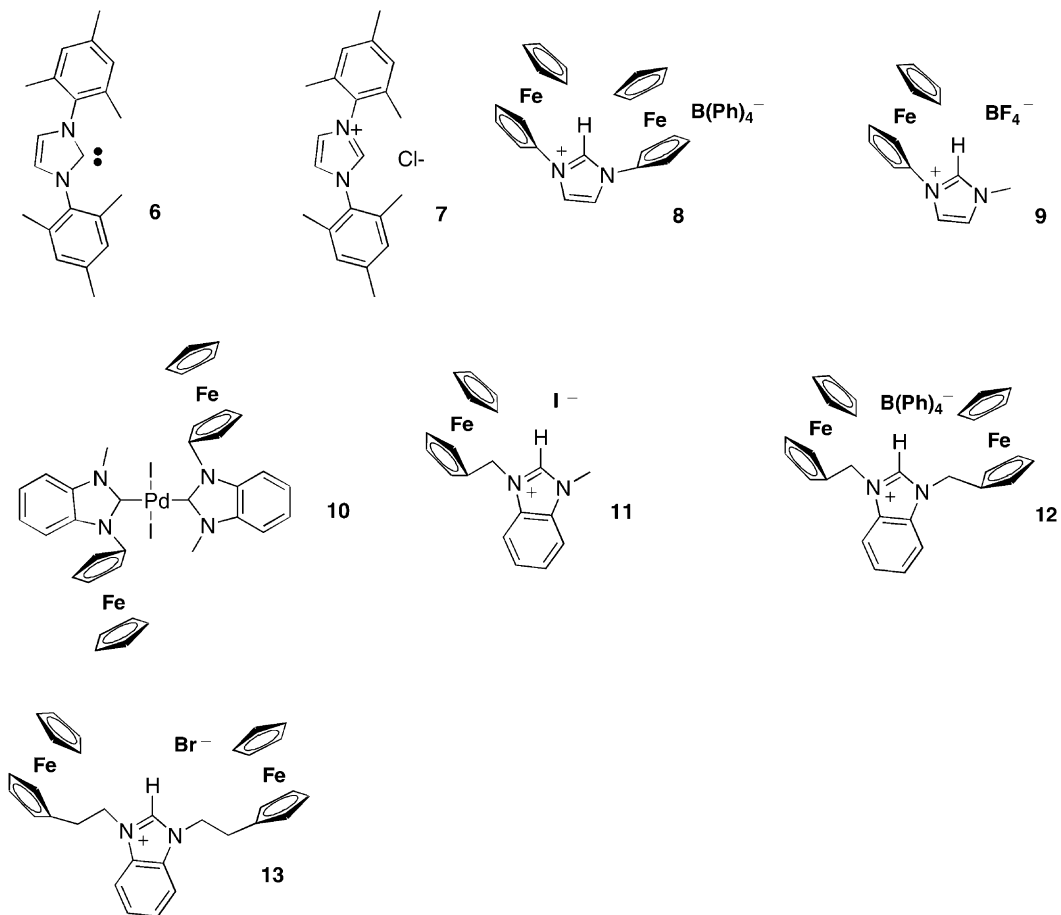
Scheme 2.

Fig. 2.

3-methylimidazolium tetrafluoroborate (**9**), 1-ferrocenylmethyl-3-methylbenzimidazolium iodide (**11**), 1,3-bis(ferrocenylmethyl)benzimidazolium tetraphenylborate (**12**), 1,3-bis(2-ferrocenylethyl)benzimidazolium bromide (**13**) were applied as ligand precursors. In addition, the palladium complex **10** was also used. The imidazolium salts and **10** were synthesized as described previously [11]. Of special interest to us was the question whether redox-active ferrocenylated imidazolium and benzimidazolium salts exhibit unusual catalytic features.

A prerequisite for an industrial application of telomerization reactions is a high catalyst efficiency, therefore only a small amount of palladium (1,3-butadiene:catalyst = 330,000:1–100,000:1) was used in the test reaction. In general, 2 or 4 equiv. of ligand relative to Pd and a 1,3-butadiene to methanol ratio of 1:2 were employed. Previously we found a positive influence of a catalytic amount of base on the catalyst activity. Hence, 1 mol% of NaOH was added to the reaction mixture. For comparison catalytic reactions were also performed in the presence of triphenylphosphine [12] and the free carbene **6** under identical reaction conditions.

As shown in Table 1 (entry 1) the previously optimized palladium phosphine catalyst system

(Pd(dba)$_2$/2PPh$_3$) gave methoxyoctadienes **1** and **2** in a total yield of 86% (catalyst turnover number TON = 86000). The linear to branched ratio (l/b = **1/2**) is 14:1 and the chemoselectivity is 92%. Decreasing the palladium concentration to 0.0003 mol% led to a lower yield (69%; Table 1, entry 2). Among the various imidazolium salts tested **7** and **12** gave higher product yields compared to triphenylphosphine (Table 1, entries 5, 7, and 15). Remarkably, in the presence of all imidazolium salts except for **8** improved linear to branched ratios were obtained. Using **9**, **10** and **11** l/b ratios >60:1 were observed. These are the highest regioselectivities known that we are aware for this type of reaction at 90 °C. Apparently, unsymmetrically substituted carbenes are especially suited to give high regioselectivities. If two ferrocenyl units are directly bound to the imidazolium salt **8** no conversion was observed. The resulting carbene will not bind to the palladium center due to steric reasons.

To prove whether the free carbene ligand gives similar results compared to the imidazolium salt 1,3-dimesitylimidazol-2-ylidene (**6**) was prepared from **7** in 70% yield. Addition of 0.002 mol% of ligand **6** resulted in 92% (93%) yield (chemoselectivity = 98%; l : b = 36 : 1) of methoxyoctadienes **1** and **2** (Table 1, entries 3 and 4). These results are nearly

Table 1
Telomerization of 1,3-butadiene and methanol[a]

| Entry | Pd catalyst | Pd (mol%) | L | Pd:L | Yield (%)[b] | Chemoselectivity (%)[c] | l/b | TON |
|---|---|---|---|---|---|---|---|---|
| 1 | A | 0.001 | PPh$_3$ | 1:2 | 86 | 92 | 14:1 | 86000 |
| 2 | A | 0.0003 | PPh$_3$ | 1:2 | 69 | 92 | 14:1 | 230000 |
| 3 | A | 0.001 | **6** | 1:2 | 92 | 98 | 35:1 | 92000 |
| 4 | B | 0.001 | **6** | 1:2 | 93 | 98 | 35:1 | 93000 |
| 5 | A | 0.001 | **7** | 1:2 | 95 | 98 | 35:1 | 95000 |
| 6 | B | 0.001 | **7** | 1:2 | 87 | 98 | 36:1 | 87000 |
| 7 | A | 0.0003 | **6** | 1:2 | 83 | 97 | 39:1 | 278000 |
| 8 | A | 0.0003 | **6** | 1:4 | 89 | 98 | 39:1 | 296000 |
| 9 | A | 0.0003 | **7** | 1:4 | 92 | 98 | 39:1 | 308000 |
| 10 | B | 0.0003 | **7** | 1:4 | 94 | 98 | 39:1 | 314000 |
| 11 | A | 0.001 | **8** | 1:2 | 0 | – | – | – |
| 12 | A | 0.001 | **9** | 1:2 | 73 | 96 | 61:1 | 73000 |
| 13 | **10** | 0.001 | – | – | 48 | 94 | 61:1 | 48000 |
| 14 | A | 0.001 | **11** | 1:2 | 8 | 75 | 99:1 | 8000 |
| 15 | A | 0.001 | **12** | 1:2 | 88 | 97 | 42:1 | 88000 |
| 16 | A | 0.0003 | **12** | 1:4 | 41 | 94 | 25:1 | 137000 |
| 17 | A | 0.001 | **13** | 1:2 | 82 | 97 | 36:1 | 82000 |

[a] General conditions: 16 h, 90 °C, 1 mol% NaOH. A = Pd(dba)$_2$, B = Pd(OAc)$_2$; MeOH:butadiene = 2:1.

[b] Yield of **1** + **2**.

[c] Chemoselectivity = **1** + **2**/**1** + **2** + **3** + **4** × 100.

Table 2
Telomerization of 1,3-butadiene and different alcohols[a]

| Entry | Pd catalyst | ROH | Pd (mol%) | L | Pd:L | Yield 1+b (%)[b] | Chemoselectivity (%)[c] | l/b | TON |
|-------|-------------|-----|-----------|---|------|------------------|--------------------------|-----|-----|
| 1 | A | $n$-C$_4$H$_9$OH[d] | 0.001 | PPh$_3$ | 1:4 | 20 | 36 | 16:1 | 20000 |
| 2 | A | $i$-C$_3$H$_7$OH[e] | 0.005 | PPh$_3$ | 1:4 | 3 | 29 | – | 600 |
| 3 | A | $n$-C$_4$H$_9$OH[d] | 0.001 | **7** | 1:4 | 90 | 93 | 44:1 | 90000 |
| 4 | A | $n$-C$_4$H$_9$OH[d] | 0.001 | **11** | 1:4 | 15 | 90 | 99:1 | 15000 |
| 5 | A | $n$-C$_4$H$_9$OH[d] | 0.001 | **12** | 1:4 | 85 | 86 | 70:1 | 85000 |
| 6 | A | $n$-C$_4$H$_9$OH[d] | 0.001 | **13** | 1:4 | 79 | 84 | 70:1 | 79000 |
| 7 | A | $i$-C$_3$H$_7$OH[e] | 0.005 | **7** | 1:4 | 71 | 75 | 52:1 | 14200 |
| 8 | A | $i$-C$_3$H$_7$OH[e] | 0.005 | **11** | 1:4 | 2 | 29 | – | 400 |
| 9 | A | $i$-C$_3$H$_7$OH[e] | 0.005 | **12** | 1:4 | 35 | 40 | 99:1 | 7000 |
| 10 | A | $i$-C$_3$H$_7$OH[e] | 0.005 | **13** | 1:4 | 27 | 32 | 99:1 | 5400 |

[a] General conditions: 16 h, 90 °C, 1 mol% base. ROH:1,3-butadiene = 2:1.

[b] Yield of **1** + **2**.

[c] Chemoselectivity = $\mathbf{1} + \mathbf{2}/\mathbf{1} + \mathbf{2} + \mathbf{3} + \mathbf{4} \times 100$.

[d] Base: C$_4$H$_9$ONa.

[e] Base: NaOCH(CH$_3$)$_2$.

identical to the ones obtained in the presence of the corresponding imidazolium salt, making a fast and complete transformation of the **7** to **6** under the reaction conditions very likely.

It is noteworthy that using imidazolium salt **7** as ligand even with a substrate to catalyst ratio of 330000:1 almost full conversion was obtained (yield **1** + **2** = 94%; catalyst turnover number TON = 314,000). To the best of our knowledge, this is the highest catalyst productivity reported for any telomerization reaction.

Regarding the palladium source Pd(OAc)$_2$ and Pd(dba)$_2$ gave in most cases very similar results. There is no general advantage to start with a Pd(II) or Pd(0) precursor complex.

Next we were interested in the coupling of 1,3-butadiene with other alcohols apart from methanol. In general, higher alcohols react in the presence of palladium/phosphine systems much less efficient compared to methanol and ethanol. Here, molecular-defined carbene complexes proved to be much better catalysts [10]. As test substrates for other alcohols $n$-butanol and $iso$-propanol were used under the standard conditions as described above. The results are summarized in Table 2.

Using $n$-butanol and $iso$-propanol in the presence of Pd(dba)$_2$/2PPh$_3$ only low yields of 1-butoxy-2,7-octadiene (**14**) (20%) and 1-$iso$-propoxy-2,7-octadiene (**15**) (3%) were observed. The in situ generated

carbenepalladium complexes from imidazolium salts **7**, **12** and **13** were significantly more productive and selective compared to the standard catalyst. For example using $n$-butanol and $iso$-propanol in the presence of 0.001 mol% (0.005 mol%) of **7** the telomerization products were obtained in 90 and 71% yield, respectively (Table 2, entries 3 and 7). Applying the imidazolium salts **12** and **13** for the telomerization of $n$-butanol also high yields of **14** were obtained (85 and 79%, respectively). However in case of $iso$-propanol the yield of **15** is significantly diminished. In agreement with the results obtained for the coupling of methanol the highest regioselectivity was observed using the imidazolium salt **11**.

## 3. Conclusion

In summary we have demonstrated for the first time that carbenepalladium complexes generated in situ from imidazolium salts show remarkable catalyst productivities and regioselectivities for different telomerization reactions. The efficiency of the catalyst system as well as the simplicity of the reaction makes these transformation interesting for industrial applications. Further studies on the development of other carbenepalladium catalysts are in progress.

# 4. Experimental

## 4.1. General procedure for the telomerization

1.6 mg ($2.77 \times 10^{-6}$ mol) of Pd(dba)$_2$ are dissolved in 17.8 g (0.555 mol) of methanol under argon. The corresponding amount of the ligand and 111.1 mg of NaOH ($2.77 \times 10^{-3}$ mol) are added. The mixture is transferred under argon into a secured 100 ml stainless steel Parr autoclave. After cooling with dry ice 15 g ($2.77 \times 10^{-1}$ mol) of 1,3-butadiene are condensed into the autoclave (mass control) and the vessel is heated to the reaction temperature. After the reaction is finished the autoclave is cooled down and the remaining 1,3-butadiene is condensed. The conversion is determined by the mass difference of 1,3-butadiene before and after the reaction. The yield of telomerization products is determined by GC (with isooctane as the internal standard) using an HP 6869A gas chromatograph. In order to isolate the different octadienyl ethers the reaction mixture is distilled in vacuo.
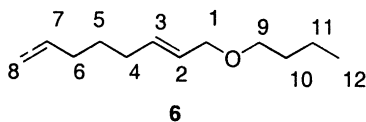
*cis/trans*-1-Methoxy-2,7-octadiene (**1**): ($M = 140.2$ g/mol, Kp$_5 = 30$ °C.



**1**
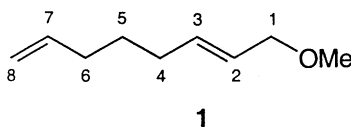
$^1$H NMR (CDCl$_3$, 400 MHz): $\delta = 1.39$ (quint, $^3J_{5,4 \text{ and } 6} = 8$ Hz, 2H, 5-H), 1.9 (m, 4H, 4-H and 6-H), 3.2 (s, 3H, OCH$_3$), 3.7 (d, $^3J_{1,2} = 6$ Hz, 2H, 1-H), 4.75–4.9 (m, 2H, 8-H), 5.35–5.45 (m, 1H, 7-H), 5.5–5.7 (m, 2H, 2-H and 3-H).
$^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta = 28.6$ (C-5), 32.0 (C-4), 33.5 (C-6), 57.9 (OCH$_3$), 73.5 (C-1), 114.9 (C-8), 126.9 (C-2), 134.8 (C-3), 138.8 (C-7).

1-Butoxy-2,7-octadiene (**14**): ($M = 182.3$ g/mol, Kp$_5 = 80$ °C.



**6**

$^1$H NMR (CDCl$_3$, 400 MHz): $\delta = 0.75$ (t, $J = 7.3$ Hz, 3H, 12-H), 1.25 (sext, $J = 7.1$ Hz, 2H, 11-H),

1.39 (q, $^3J_{5,4 \text{ and } 6} = 7$ Hz, 2H, 5-H), 1.42 (quint, $J = 7.1$ Hz, 2H, 10-H), 1.9 (m, 4H, 4-H and 6-H), 3.26 (t, $J = 6.7$ Hz, 2H, 9-H), 3.7 (dd, $J = 6$, 1 Hz, 2H, 1-H), 4.76–4.9 (m, 2H, 8-H), 5.36–5.45 (m, 1H, 7-H), 5.5–5.7 (m, 2H, 2-H and 3-H).
$^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta = 13.6$ (C-12), 19.1 (C-11), 28.05 (C-5), 31.4 (C-10), 31.6 (C-4), 32.9 (C-6), 69.1 (C-9), 71.3 (C-1), 114.3 (C-8), 126.7 (C-2), 133.5 (C-3), 138.2 (C-7).
MS *m/z* (%): 182 [M$^+$] (1.4), 139 (4.3), 126 (10.6), 108 (24), 101 (3.9), 97 (11), 93 (27), 82 (35), 67 (72), 57 (100).
HRMS. Calc. C$_{12}$H$_{22}$O: 182.16707, found: 182.16460.

1-Isopropoxy-2,7-octadiene (**15**): ($M = 168.3$ g/Mol, Kp$_5 = 50$ °C).



**7**
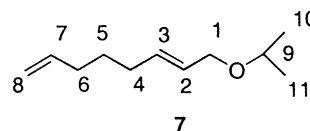
$^1$H NMR (CDCl$_3$, 400 MHz): $\delta = 1.05$ (d of s, 6H, 10-H, 11-H), 1.4 (quint, $J = 7.5$ Hz, 2H, 10-H), 1.9 (m, 4H, 4-H and 6-H), 3.5 (sept, $J = 6.1$ Hz, 2H, 9-H), 3.82 (dd, $J = 6.2$ Hz, $J = 1$ Hz, 2H, 1-H), 4.76–4.9 (m, 2H, 8-H), 5.36–5.45 (m, 1H, 7-H), 5.5–5.75 (m, 2H, 2-H and 3-H).
$^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta = 21.7$ (C-11, C-10), 27.9 (C-5), 31.3 (C-4), 32.9 (C-6), 69.1 (C-9), 70.8 (C-1), 114.8 (C-8), 127.7 (C-2), 133.5 (C-3), 138.7 (C-7).
MS *m/z* (%): [M$^+$] 168 (0.11), 126 (12.5), 109 (30.6), 97 (13), 93 (25), 82 (68), 67 (95), 55 (76), 43 (100).
EA. Calc. C$_{11}$H$_{20}$O: C, 78.51; H, 11.98; found: C, 78.56; H, 11.95.

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