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# Allylstannation of α-, β- and γ-diketones mediated by allylbutyltin halides: $Bu_2(CH_2=CHCH_2)SnCl$ and $Bu(CH_2=CHCH_2)SnCl_2$

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**Abstract**

Butane-2,3- (**1a**), pentane-2,4- (**1b**) and hexane-2,5-dione (**1c**) react with $Bu_2(CH_2=CHCH_2)SnCl$ in the presence of water to give monoallylated keto-ols (**2a**, **2b**) and/or diallylated diols (**3a**, **3b**, **3c**), this depending upon the employed molar ratio [diketone]/[allyltin chloride]. $Bu(CH_2=CHCH_2)SnCl_2$ reacts with neat **1c** in a one-pot synthesis to give mixtures of heterocyclic compounds: 2,5-diallyl-2,5-dimethyltetrahydrofuran (**4**), and 3-chloro-1,5-dimethyl-8-oxabicyclo [3,2,1] octane (**5**). Compound **4** is also obtained in high yield from the corresponding diol **3c** by cyclodehydration promoted by $RSnCl_3$ (R = Me and Bu). © 1997 Elsevier Science S.A.

*Keywords:* Allylstannation; Allyldibutyltin chloride; Allylbutyltin dichloride; Diketones; Hydroxyketones; Diols

## 1. Introduction

Allylchlorotins, $R_{3-n}AllSnCl_n$ (R = alkyl, All = $CH_2=CHCH_2$ or allyl-like group, $n = 1, 2$) have been shown to be very versatile reagents for C–C and C–O–C bond-forming reactions [1], which take place under very mild conditions in the absence of solvent and catalysts, without any special precautions. The tin–allyl bond in such substrates is so kinetically inert that its cleavage does not occur in the presence of water at room temperature, at least over a long period [2]. Therefore our previous studies have dealt with addition of carbonyl compounds to allyltin chlorides in water under heterogeneous conditions [3]. Besides allystannation processes, propargyl- and allenyl-stannations, as well as allylation in acid media have been realized [4].

In the present paper, we wish to report reactions of dibutylallyltin chloride with butane-2,3- (**1a**), pentane-2,4- (**1b**) and hexane-2,5-dione (**1c**) in the presence of water (see Scheme 1).

Keto-ols of the type **2** having $n = 0$ and 1, and diols of the type **3** having $n = 0$, 1 and 2 have been isolated. Reactions performed neat between **1c** and allylbutyltin dichloride show the ability of this compound to perform allylstannation followed by intramolecular cyclization to give **4** and **5**. Compound **4** can be also obtained by catalytic cyclodehydration of the diol **3c** in the presence of $BuSnCl_3$, as has been pointed out for many 1,*n*-diols ($n = 4$ and 5) [5]. On the other hand, a direct cyclodehydration of diketone **1c** promoted by $RSnCl_3$ compounds (R = Me and Bu) allows one to prepare 2,5-dimethylfuran (**6**).

## 2. Results and discussion

Results dealing with allylstannation of **1a** and **1b** with allyldibutyltin chloride in the presence of water at ambient temperature at various $R$ = [**1**]:[organotin] molar ratios are given in Tables 1 and 2 respectively.

In both cases, it is possible to discriminate the preparation of the corresponding mono- and di-allylated products on varying the $R$ molar ratios of the two reactants, as described by Eqs. (1) and (2).

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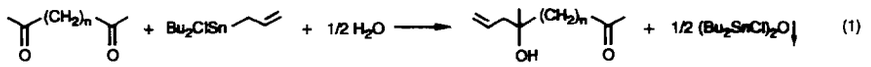
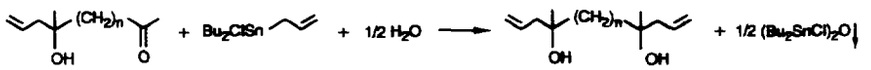


(1)



(2)

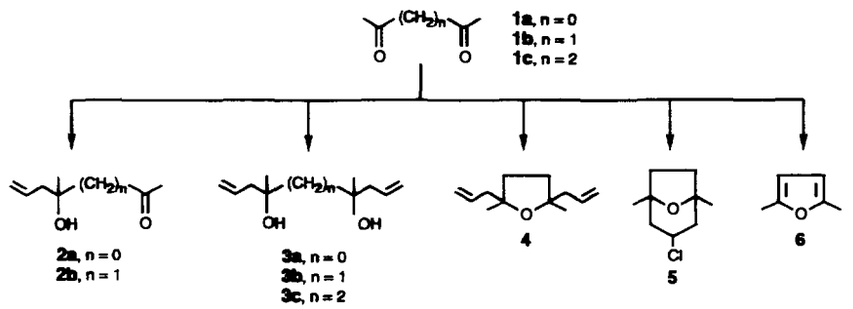
The overall process to form diols occurs in 5–6 days. In any case, a nearly quantitative amount of 1,3-dichloro-tetrabutyldistannoxane is recovered (95–98%). 3-Hydroxy-3-methylhex-5-en-2-one (**2a**) is the sole recovered product at $R = 1$ (entry 1), a mixture of **2a** and 4,5-dimethylocta-1,7-dien-4,5-ol (**3a**) is obtained at $R = 0.5$, and **3a** is the sole product at $R = 0.3$. The same trend is observed for the product arising from the allylstannation of **1b**: 4-hydroxy-4-methylhept-6-en-2-one (**2b**) is the major component in entries 4 and 5 ($R = 1.5$ and 1 respectively), whereas 4,6-dimethylnona-1,8-dien-4,6-diol (**3b**) is prevalent in entries 6 and 7 ($R = 0.5$ and 0.3 respectively). With regard to the reaction of **1c**, only the diallylated diol **3c** is recovered. In this case, the two carbonyl functions are so far away that interactions between them may be excluded and the allylation rate is the same for both. Indeed, complete allylstannation of both carbonyl functions of **1c** takes place at a higher rate than those dealing with diketones **1a** and **1b**. In all runs, the diols **3a**, **3b** and **3c** are obtained as mixtures of *meso*- and D,L-*racemate*-diastereoisomers with ratios 40:60, 55:45 and 50:50 respectively.

Allystannation promoted by allyldibutyltin chloride represents a very easy procedure able to prepare either compounds of the type **2** or **3**. It is noteworthy to mention that allylation of $\alpha$-diketones with $R_3Sn$–$CH_2$=$CHCH_2$ compounds mediated by Lewis acids or promoted via photoinduction [6] leads to the formation of the sole monoallylated $\alpha$-hydroxyketones **2**. These have also been prepared from [2,3]sigmatropic rearrangement of acetonylallylethers [7], by means of allylzinc reagent [8], and $\pi$-allylnickel bromide [9]. Diallylated diols of type **3** have been obtained via $CeCl_3$-mediated addition of Grignard reagents [10] and from diepoxides [11]. The palladium-catalysed allylation of diketones by allylic alcohols with $SnCl_2$ seems so far the sole procedure able to prepare both compounds **2** and **3** [12].

We have already shown that allylbutyltin dichloride and allyltin trichloride react with aldehydes to give 4-chlorotetrahydropyran derivatives [13]. These compounds arise from the allylstannation of the carbonyl site followed by an intramolecular cyclization process. Therefore, we have performed reactions between $Bu(CH_2$=$CHCH_2)SnCl_2$ and **1c**, since formation of cyclic products may be expected from this diketone after allylstannation. Results of these reactions, either at room temperature or at 80 °C, are listed in Table 3.

As one can see, mixtures of 2,5-diallyl-2,5-dimethyltetrahydrofuran (**4**) and 3-chloro-1,5-dimethyl-8-oxabicyclo[3,2,1]octane (**5**) are isolated. Compound **4** is the major component when $R = 0.5$ (entries 8 and 9), whereas both compounds **4** and **5** are obtained in equal amounts when $R = 1$ (entries 10 and 11).

The ability of organotin derivatives to promote the formation of cyclic compounds from **1c** has already been verified. Indeed, 2,5-dimethylfuran (**6**) (see Scheme 2) can be prepared in high yield by heating **1c** in the presence of a catalytic amount of $BuSnCl_3$ [14]. Additional data about the catalytic activity of $MeSnCl_3$ and $Bu_2SnCl_2$ in comparison with $BuSnCl_3$ are listed in Table 4. One can see that $Bu_2SnCl_2$ is a poor catalyst of this reaction while very good results are obtained with both organotin trichlorides. However, use of $BuSnCl_3$ is recommended since this compound can be better manipulated than $MeSnCl_3$. This reaction is an example of cyclodehydration via interaction of the enol




Scheme 1. Allylstannation and cyclization of diketones operated by organotin halides.

Table 1
Allylstannation of butane-2,3-dione (**1a**) with allyldibutyltin chloride in the presence of water at 25 °C [a]

| Entry | **1a** (g; (mmol)) | Molar ratio $R$ [b] | Recovered product (g) | Composition (%) | |
|---|---|---|---|---|---|
| | | | | **2a** | **3a** |
| 1 | 1.72; (20) | 1.0 | 1.98 [c] | 100 | — |
| 2 | 0.86; (10) | 0.5 | 1.42 | 15 | 85 |
| 3 | 0.86; (10) | 0.3 | 1.60 [d] | — | 100 |

[a] Systems were stirred for 6 days before work-up.
[b] $R$ = [**1a**]:[organotin].
[c] 77% yield.
[d] 93% yield.

Table 2
Allylstannation of pentane-2,4-dione (**1b**) with allyldibutyltin chloride in the presence of water at 25 °C [a]

| Entry | **1b** (g; (mmol)) | Molar ratio $R$ [b] | Recovered product (g) | Composition (%) | |
|---|---|---|---|---|---|
| | | | | **2b** | **3b** |
| 4 | 3.0; (30) | 1.5 | 2.45 | 93 | 7 |
| 5 | 2.0; (20) | 1.0 | 2.14 | 78 | 22 |
| 6 | 1.0; (10) | 0.5 | 1.87 | 32 | 68 |
| 7 | 1.0; (10) | 0.3 | 2.08 | 7 | 93 |

[a] Systems were stirred for 6 days before work-up.
[b] $R$ = [**1b**]:[organotin].

Table 3
Allylstannation of hexane-2,5-dione (**1c**) with allylbutyltin dichloride

| Entry | **1c** (g; (mmol)) | Molar ratio $R$ [a] | Temp.; time (°C; h) | Recovered product (g) | Composition (%) | |
|---|---|---|---|---|---|---|
| | | | | | **4** | **5** |
| 8 | 1.90; (16.5) | 0.5 | 25; 25 | 1.5 | 86 | 14 |
| 9 | 2.52; (22.1) | 0.5 | 80; 3 | 1.8 | 80 | 20 |
| 10 | 5.71; (50) | 1.0 | 25; 20 | 1.9 | 45 | 55 |
| 11 | 5.71; (50) | 1.0 | 80; 9 | 2.5 | 52 | 48 |

[a] $R$ = [**1c**]:[organotin].

Table 4
Catalytic conversion of hexane-2,5-dione (**1c**) to 2,5-dimethylfuran (**6**) by mono- and di-organotin chlorides, $R = 20$ [a]

| Entry | **1c** (g; (mol)) | Organotin chloride (g; (mmol)) | Temp.; time (°C; h) | Recovered **6** (g; (yield %)) |
|---|---|---|---|---|
| 12 | 25.0; (0.22) | $BuSnCl_3$ 3.1; (11) | 160; 3 | 18.2; (85) |
| 13 | 30.0; (0.26) | $MeSnCl_3$ 3.1; (13) | 170; 1 | 21.2; (84) |
| 14 | 30.0; (0.26) | $Bu_2SnCl_2$ 3.9; (13) | 220; 1 | 2.1; (8) |

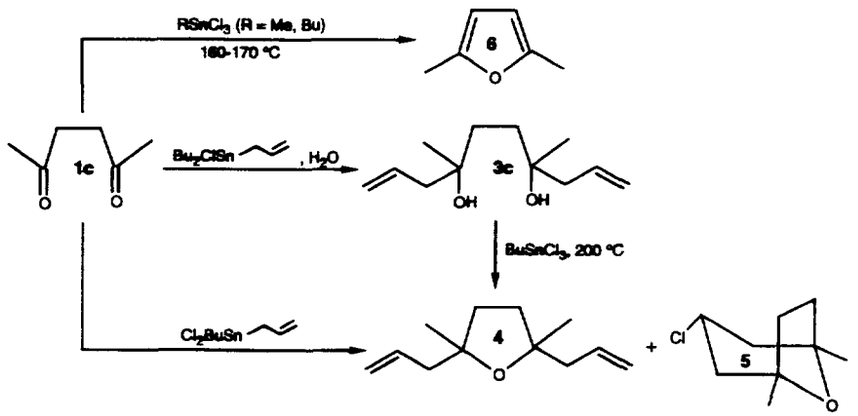
[a] $R$ = [**1c**]:[organotin chloride].

form of the diketone with alkyltin halides. It may be thought that also the interaction of the allylstannane with **1c** leads to an alkoxystannane which gives the cyclic compound **4** by internal rearrangement, as has been verified for many diols [5]. An alternative way to produce compound **4** is the cyclodehydration of the diol **3c** catalytically mediated by $BuSnCl_3$. Scheme 2 gathers together the different reactions dealing with the interaction of **1c** with allyl- and alkyl-tin halides.

## 3. Experimental section

Allyldibutyltin chloride and allylbutyltin dichloride were prepared and purified as previously described [3]. Dibutyltin dichloride, methyltin and butyltin trichlorides and diketones, commercially available from Aldrich, were purified before use by means of appropriate procedures.

The $^1H$ (89.55 MHz) and $^{13}C$ NMR (22.49 MHz) spectra were recorded with a Jeol FX90Q multinuclear spectrometer operating in Fourier transform mode. Chemical shifts are reported in parts per million down-




Scheme 2. Products arising from the interaction between **1c** and organotin halides.

field from internal TMS. $^1$H NMR measurements were made using $CDCl_3$ solutions. Off-resonance, insensitive nuclei enhanced polarization transfer (INEPT) and selective decoupling techniques were used to analyse and assign the $^{13}$C NMR signals. The gated decoupling method [15] was also employed for quantitative analyses of the $^{13}$C NMR spectra. The assignment of the $^{13}$C NMR signals dealing with the *meso*- and D,L-configurations of the diols **3a**, **3b** and **3c** were made on the basis of previous observations [16]. GC analyses were carried out with a Gas-Chromatograph Perkin–Elmer model 8310 equipped with an ionization flame detector using samples dissolved in diethyl ether (15 m length × 0.25 mm o.d. DB 225 capillary polar column, $T_d$ = 220 °C, $T_i$ = 200 °C, $T_o$ = 80–220 °C with a temperature programming of 10 °C min$^{-1}$, nitrogen as carrier-gas at 10 psi). Retention times $t_R$ were measured from the peak of the diethyl ether. GC technique is not able [16] to discriminate the two diastereoisomers of diols **3a**, **3b** and **3c** neither with packed columns nor with capillary columns having different polarities. The IR spectra were recorded with a Perkin–Elmer, model 599B spectrophotometer using KBr optics.

### 3.1. Reactions of $Bu_2(CH_2=CHCH_2)SnCl$ with diketones 1a, 1b and 1c in the presence of water

The following general procedure has been adopted. The appropriate diketone was added under stirring to allyldibutyltin chloride at room temperature, then 10 ml of pure water produced by a Millipore Milli-Q system were added. Generally, a white precipitate (1,3-dichloro-tetrabutyldistannoxane) appeared after 20–24 h. This shows that the rate of the reaction is low. Thus, stirring was prolonged for 5–6 days. After this time, the distannoxane was filtered off. Extraction with diethyl ether was made from the filtrate, then the product was isolated as heavy oil or low melting solid compound. Amounts of the reagents, their ratios, and quantities of the obtained products are listed in Tables 1 and 2.

#### 3.1.1. 3-Hydroxy-3-methylhex-5-en-2-one (2a)

IR (cm$^{-1}$) (film): $\nu$ (OH) 3460s, $\nu$ (CH=) 3080w, $\nu$ (C=O) 1705s, $\nu$ (C=C) 1640m. $^{13}$C NMR (ppm) (pure sample): $\delta$ 24.8 ($CH_3$–CO; $CH_3$–COH), 44.0 ($CH_2$), 79.0 (C–OH), 118.3 ($CH_2$=), 133.2 (CH=), 212.1(C=O). $t_R$: 1.48 min. Anal. Found: C, 65.30; H, 9.49. $C_7H_{12}O_2$. Calc.: C, 65.61; H, 9.44.

#### 3.1.2. 4-Hydroxy-4-methylhept-6-en-2-one (2b)

IR (cm$^{-1}$) (film): $\nu$ (OH) 3470s, $\nu$ (CH=) 3080w, $\nu$ (C=O) 1705s, $\nu$ (C=C) 1640m. $^1$H NMR (ppm) ($CDCl_3$ solution): $\delta$ 1.21 (s, 3H; $CH_3$–COH), 2.16 (s, 3H; $CH_3$–CO), 2.29 (d, 2H; $CH_2$–CO), 2.59 (m, 2H; $CH_2$–CH=), 4.6–6.2 (m, 3H; vinyl group). $^{13}$C NMR (ppm) (pure sample): $\delta$ 27.1 ($CH_3$–COH), 31.9 ($CH_3$–CO), 47.0 ($CH_2$–CO), 52.6 ($CH_2$–CH=), 71.4 (C–OH), 117.9 ($CH_2$=), 134.6 (CH=), 209.8 (C=O). $t_R$: 2.72 min. Anal. Found: C, 67.25; H, 9.91. $C_8H_{14}O_2$. Calc.: C, 67.54; H, 9.92.

#### 3.1.3. 4,5-Dimethylocta-1,7-dien-4,5-diol (3a)

IR (cm$^{-1}$) (film): $\nu$ (OH) 3400s, $\nu$ (CH=) 3080w, $\nu$ (C=C) 1640m. $^{13}$C NMR (ppm) ($CDCl_3$ solution): $\delta$ *meso*-derivative (40%) 21.5 ($CH_3$), 41.6 ($CH_2$), 76.6 (C–OH), 117.4 ($CH_2$=), 135.8 (CH=); $\delta$ D,L-mixture (60%) 21.8 ($CH_3$), 41.6 ($CH_2$), 76.6 (C–OH), 117.4 ($CH_2$=), 135.8 (CH=). $t_R$: 4.66 min. Anal. Found: C, 70.50; H, 10.78. $C_{10}H_{18}O_2$. Calc.: C, 70.55; H, 10.66.

#### 3.1.4. 4,6-Dimethylnona-1,8-dien-4,6-diol (3b)

IR (cm$^{-1}$) (film): $\nu$ (OH) 3350s, $\nu$ (CH=) 3080w, $\nu$ (C=C) 1640m. $^1$H NMR (ppm) ($CDCl_3$ solution): $\delta$ *meso*-derivative (55%) 1.27 (s, 6H; $CH_3$), 1.3–1.9 (q AB; $\nu_1$ = 1.58 ppm, 1H, $\nu_2$ = 1.78 ppm, 1H; $CH_2$–COH), 2.25 (d, 4H; $CH_2$–CH=) 4.6–6.2 (m, 6H; vinyl group); $\delta$ D,L-mixture (45%) 1.33 (s, 6H; $CH_3$), 1.67 (s, 2H; $CH_2$–COH), 2.34 (d, 4H; $CH_2$–CH=) 4.6–6.2 (m, 6H; vinyl group). $^{13}$C NMR (ppm) ($CDCl_3$ solution): $\delta$ *meso*-derivative (54%) 29.2 ($CH_3$), 47.8 ($CH_2$–COH), 50.2 ($CH_2$–CH=), 73.6 (C–OH), 117.7 ($CH_2$=), 134.8 (CH=); $\delta$ D,L-mixture (46%) 28.6 ($CH_3$), 48.1 ($CH_2$–COH), 49.1 ($CH_2$–CH=), 73.5 (C–OH), 117.7 ($CH_2$=), 134.6 (CH=). $t_R$: 6.06 min. Anal. Found: C, 71.35; H, 11.02. $C_{11}H_{20}O_2$. Calc.: C, 71.70; H, 10.94.

#### 3.1.5. 4,7-Dimethyldeca-1,9-dien-4,7-diol (3c)

IR (cm$^{-1}$) (film): $\nu$ (OH) 3355s, $\nu$ (CH=) 3080w, $\nu$ (C=C) 1640m. $^{13}$C NMR (ppm) (pure sample): $\delta$ *meso*-derivative (50%) 26.8 ($CH_3$), 35.1 ($CH_2$–$CH_2$), 46.8 ($CH_2$–CH=), 72.0 (C–OH), 117.3 ($CH_2$=), 135.0 (CH=); $\delta$ D,L-mixture (50%) 27.0 ($CH_3$), 35.1 ($CH_2$–$CH_2$), 47.1 ($CH_2$–CH=), 72.0 (C–OH), 117.3 ($CH_2$=), 135.0 (CH=). $t_R$: 7.67 min. Anal. Found: C, 72.48; H, 11.25. $C_{12}H_{22}O_2$. Calc.: C, 72.68; H, 11.18.

### 3.2. Reactions of allylbutyltin dichloride with hexane-2,5-dione (1c)

A mixture of the two reactants was allowed to react at room temperature or at 80 °C and the progress of the reaction was monitored by gas-chromatography. Formation of the cyclic products was observed after 1 day. After an appropriate time sufficient to reach a constant gas-chromatographic response, the system was hydrolysed with 20 ml of an $Na_2CO_3$ solution (2 M). The formed organotin oxides were filtered off, then the cyclic products were extracted from the filtrate with ethyl ether. After removal of the ether, crude colourless liquids were recovered and analysed. Generally, mix-

tures of both compounds **4**, present as cis- and trans-isomer, and compound **5**, present as one sole isomer, were obtained. Compounds **4** and **5** were isolated by means of gas-chromatography using a $\frac{1}{4}''$ inox column packed with 10% DEGS on chromosorbe. Amounts of the reagents, their ratios, reaction times, and quantities of the obtained products are listed in Table 3.

*3.2.1. 2,5-Diallyl-2,5-dimethyltetrahydrofuran (4)*

IR ($cm^{-1}$) (film): $\nu$ (CH=) 3080w, $\nu$ (C=C) 1640m. $^{13}C$ NMR (ppm) (pure sample): $\delta$ cis-isomer (60%) 27.5 ($CH_3$), 36.3 ($CH_2$–$CH_2$), 47.5 ($CH_2$–CH=), 83.5 (C–O–C), 116.8 ($CH_2$=), 135.5 (CH=); $\delta$ trans-isomer (40%) 27.8 ($CH_3$), 36.5 ($CH_2$–$CH_2$), 47.2 ($CH_2$–CH=), 82.5 (C–O–C), 116.8 ($CH_2$=), 135.5 (CH=). $t_R$: 3.08 min for both isomers (oven temperature: from 45 °C to 220 °C with a temperature programming of 15 °C $min^{-1}$). Anal. Found: C, 79.80; H, 11.25. $C_{12}H_{20}O$. Calc.: C, 79.95; H, 11.18.

*3.2.2. 3-Chloro-1,5-dimethyl-8-oxabicyclo[3,2,1]octane (5)*

IR ($cm^{-1}$) (film): $\nu$ $(C–Cl)_{eq}$ 745m, $\nu$ $(C–Cl)_{ax}$ 545m. $^1H$ NMR (ppm) ($CDCl_3$ solution): $\delta$ 1.33 (s, 6H; $CH_3$), 1.70–2.05 (m, 8H; $CH_2$), 4.20 (m, 1H; CH–Cl). $^{13}C$ NMR (ppm) (pure sample): $\delta$ 26.4 ($CH_3$), 36.4 ($CH_2$–$CH_2$), 48.0 ($CH_2$–CHCl), 52.9 (C–Cl), 80.5 (C–O–C). $t_R$: 4.54 min (oven temperature: from 45 °C to 220 °C with a temperature programming of 15 °C $min^{-1}$). Anal. Found: C, 61.75; H, 8.78; Cl, 20.05. $C_9H_{15}ClO$. Calc.: C, 61.89; H, 8.66; Cl, 20.30.

*3.3. Catalytic conversion of hexane-2,5-dione (1c) to 2,5-dimethylfuran (6)*

A mixture of **1c** and the appropriate organotin chloride at a given ratio $R$ = [**1c**]/[organotin chloride] (see Table 4) was heated at the appropriate temperature with stirring. The volatile product **6** together with water was distilled out by means of a Dean Stark apparatus. After separation from the water phase, the crude product was distilled to give pure samples boiling at 93 °C (Ref. [17] 93–94 °C). Both IR and $^1H$ NMR spectra were identical with those previously reported [18].

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