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Preparation of Cycloheptane Ring by Nucleophilic Cyclopropanation of 1,2-Diketones with Bis(iodozincio)methane

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The nucleophilic cyclopropanation of hexa-1,5-diene-3,4-diones with bis(iodozincio)methane afforded the Zn alkoxides of cis-dialkynylcyclopropane-1,2-diols stereoselectively. The subsequent oxy-Cope rearrangement afforded the corresponding Zn alkoxides of 5,6-dialkylocyclohepta-3,7-diene-1,3-diols.

Introduction
The Cope rearrangement of cis-divinylcyclopropanes has been recognized as an efficient route to synthesize cycloheptane rings. The disadvantageous entropic factor for a seven-membered ring construction is overcome by coming close of both ends caused from the rigid configuration of cyclopropane.1 The difficulty in the stereoselective preparation of the cis-isomer of the substrate, however, often makes the transformation less valuable. Although some practical methods have been developed for the preparation of the cis-isomers,2 most of the methods afforded the trans-isomers that required a temperature of >100 °C to perform the Cope rearrangement.3 Thus, a direct route to synthesize cis-isomers stereoselectively is desirable in order to construct cycloheptane rings easily. During our studies on bis(iodozincio)methane (1),4 we found that the nucleophilic cyclopropanation of 1,2-diketones afforded cis-cyclopropane-1,2-diols stereoselectively.5 The mechanism of the reaction was elucidated by a computational method; the cis-selectivity was attributed to the face-to-face coordination of 1 with the diketones.6 We envisioned that the reaction of 1,6-dialkyhexa-1,5-diene-3,4-diones 2 with 1 would afford the Zn alkoxides of cis-divinylcyclopropane-1,2-diols 4, via the face-to-face coordination 3, thus facilitating the oxy-Cope rearrangement of 4 to 5, with the additional acceleration by the alkoxide groups (Scheme 1).7

Results and Discussion
1. As one-pot reaction
The reaction of (1E,5E)-1,6-diphenyhexa-1,5-diene-3,4-dione (R1, R1 = Ph, R2 = H, 2a) with dizinc 1 at −20 °C, however, afforded a complex mixture, even though it contained a small amount of the desired cycloheptane-1,3-diene 6a after the hydrolysis. The main byproduct was the adduct of an enolate 5a with the substrate 2a. This result indicates that the first reaction, i.e., the cyclopropanation of 2a with 1 should be completed before the start of Cope rearrangement to prevent the side reactions of the rearranged product 5 with substrate 2. For this purpose, we reacted diketone 2 with 1 at the lower temperatures, which do not allow Cope-rearrangement, for an appropriate period, until the complete conversion of 2; the resulting mixture was warmed up to promote the subsequent Cope-rearrangement. In fact, the reaction of 2a with 1 for 3 h at −78 °C, followed by warming up the resulting mixture to 25 °C afforded the seven-membered ring 6a in 78% yield.5 Moreover, instead of the simple heating...
to 25 °C, the addition of THF (25 °C) to the reaction mixture afforded 6a in 84% yield, because the dilution suppressed the intermolecular side reactions without affecting the rate of intramolecular rearrangement reaction. Some examples of the preparation of cycloheptane-1,3-diones are shown in Table 1. Various cycloheptane-1,3-diones substituted with two cis-aryl groups 6 were prepared and isolated in good yields (Table 1, entries 1–4). The presence of an electron-withdrawing group on the benzene ring resulted in a low yield (entry 5). The presence of bulky groups such as 1-naphthyl also resulted in a low yield (entry 7). The presence of alky groups as the substituents (R1, R2, and R3) did not hinder the reaction (Table 1, entries 8–11). These transformations were stereospecific. As shown in entries 8 and 9, the cis- and trans-isomers were obtained specifically depending on the E,Z-configuration of the substrate.

### Table 1. Preparation of Cycloheptane-1,3-diones

<table>
<thead>
<tr>
<th>entry</th>
<th>R1</th>
<th>R2</th>
<th>R3</th>
<th>6 (yield %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ph</td>
<td>Ph</td>
<td>H</td>
<td>6a (84%)</td>
</tr>
<tr>
<td>2</td>
<td>4-Me-C6H4</td>
<td>4-Me-C6H4</td>
<td>H</td>
<td>6b (93%)</td>
</tr>
<tr>
<td>3</td>
<td>4-MeO-C6H4</td>
<td>4-MeO-C6H4</td>
<td>H</td>
<td>6c (98%)</td>
</tr>
<tr>
<td>4</td>
<td>4-Br-C6H4</td>
<td>4-Br-C6H4</td>
<td>H</td>
<td>6d (96%)</td>
</tr>
<tr>
<td>5</td>
<td>4-F-C6H4</td>
<td>4-F-C6H4</td>
<td>H</td>
<td>6e (47%)</td>
</tr>
<tr>
<td>6</td>
<td>2-Furyl</td>
<td>2-Furyl</td>
<td>H</td>
<td>6f (78%)</td>
</tr>
<tr>
<td>7</td>
<td>1-Naphthyl</td>
<td>1-Naphthyl</td>
<td>H</td>
<td>6g (41%)</td>
</tr>
<tr>
<td>8</td>
<td>Me</td>
<td>Me</td>
<td>H</td>
<td>6h (99%)</td>
</tr>
<tr>
<td>9</td>
<td>Me</td>
<td>H</td>
<td>Me</td>
<td>6i (65%)</td>
</tr>
<tr>
<td>10</td>
<td>Me</td>
<td>Ph</td>
<td>H</td>
<td>6j (88 %)</td>
</tr>
<tr>
<td>11</td>
<td>Me</td>
<td>Me</td>
<td>Me</td>
<td>6k (86%)</td>
</tr>
</tbody>
</table>

*The reaction was performed with the following scale: 1 (1.2 mmol, 0.35 M THF solution), 2 (1.0 mmol, 0.35 M in THF). After 3 h at ~78 °C, 10 mL of THF (25 °C) was added in one portion. Isolated yields. The diastereomer was not detected.

The Zn-enolate intermediate 5 in Scheme 1 was able to be trapped with chlorotrimethylsilane (TMSCl) or acetic anhydride (Ac₂O). As shown in Scheme 2, after the treatment of 2h with dzcine 1 at ~78 °C for 3 h and at 25 °C for 1 h with additional THF, TMSCl was added. The corresponding silyl enol ether 7 was obtained in 96% isolated yield. Instead of TMSCl, the addition of Ac₂O afforded the corresponding enol acetate 8 in 82% isolated yield.

![Scheme 2. Trapping of Zn-enolate Intermediate from 2h with Electrophiles](image)

### 2. In a Microflow reactor

This [6+1] transformation contains two reactions, that is, the nucleophilic cyclopropanation of 1,2-diketone and the oxy-Cope rearrangement of cis-1,2-divinylcyclopropane-1,2-diol. As the activation energy for the first step is smaller than the second one, the first reaction can be completed before the second reaction proceeds at ~78 °C. Otherwise, the formed zinc enolate 5 reacts with the diketone 2. Therefore, careful temperature control made the entire transformation proceed reasonably to obtain the 7-membered product in good yields. In other words, two reactions were differentiated by the reaction temperature. Instead of the temperature control, it is possible to differentiate the two reactions with space. The microflow system (space integration) may improve the reaction arising from a preemptive start of the second reaction, because it can supply a minimum amount of the substrate to be consumed at the micromixer spontaneously. Thus, as shown in Figure 1, we constructed a microflow system consisting of two T-shaped SUS micromixers (M1 and M2, \( \phi = 0.5 \) mm) and SUS microtube reactors (R1, \( \phi = 1.0 \) mm). A THF solution of 1 (0.16 M, 3.92 mL/min) and a THF or CH2Cl2 solution of 1,2-diketone (0.09 M, 3.92 mL/min) were introduced by a syringe pump; after passage through a reactor R1, the enolate 5 and the excess amount of 1 were quenched with methanol in M2. The residence time was optimized by varying the length of the microtube reactor (See, the Supporting Information). In the flow system, that of 6 seconds (1-m length, \( \phi = 1.0 \) mm, SUS microtube reactor (R1)) afforded the products in good yields continuously. In this case, the residence time of the reaction mixture of 1 and 2 in R1 was 6 s. The period was calculated from the flow late (3.92 × 2 mL/min) and the inner volume of R1. The results are summarized in Table 2.

### Table 2. Preparation of Cycloheptane-1,3-diones Using a Microflow System

<table>
<thead>
<tr>
<th>entry</th>
<th>R1</th>
<th>R2</th>
<th>R3</th>
<th>6 (yield %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ph</td>
<td>Ph</td>
<td>H</td>
<td>6a (&gt;99%)</td>
</tr>
<tr>
<td>2</td>
<td>4-Me-C6H4</td>
<td>4-Me-C6H4</td>
<td>H</td>
<td>6b (&gt;99%)</td>
</tr>
<tr>
<td>3</td>
<td>4-MeO-C6H4</td>
<td>4-MeO-C6H4</td>
<td>H</td>
<td>6c (92%)</td>
</tr>
<tr>
<td>4</td>
<td>4-Br-C6H4</td>
<td>4-Br-C6H4</td>
<td>H</td>
<td>6d (81%)</td>
</tr>
<tr>
<td>5</td>
<td>4-F-C6H4</td>
<td>4-F-C6H4</td>
<td>H</td>
<td>6e (82%)</td>
</tr>
<tr>
<td>6</td>
<td>2-Furyl</td>
<td>2-Furyl</td>
<td>H</td>
<td>6f (77%)</td>
</tr>
<tr>
<td>7</td>
<td>Me</td>
<td>Me</td>
<td>H</td>
<td>6h (70%)</td>
</tr>
</tbody>
</table>

*The reaction was performed using the microflow system shown in Figure 2: T-shaped SUS micromixer: M1 (inner diameter: 0.5 mm) and M2 (inner diameter: 0.5 mm), SUS microtube reactor: R1 (\( \phi = 1.0 \) mm, length = 1 m), a solution of 1: 3.92 mL/min, 0.16 M; a solution of 2 in CH2Cl2: 3.92 mL/min, 0.09 M; methanol: 7.25 mL/min. THF was used as the solvent for the solution of 2 instead of CH2Cl2.
As shown in Table 2, the products were obtained in reasonable yields at 25 °C for 6 s continuously. Except entries 5 and 6, dichloromethane was used as the solvent to prepare the solution of diketones 2, because the corresponding diketones except 2e and 2f were not very soluble to THF. Notably, the microflow system allowed us to use dichloromethane as a cosolvent for reactions using a fairly basic dizinc reagent 1. Moreover, dichloromethane is difficult to use as a cosolvent in a batch reaction, because the monomeric structural of dizinc 1 in THF is changed into polymethylenezinc form through the Schlenk equilibrium by the addition of any other less polar solvent such as dichloromethane. The polymeric structure often loses the nucleophilicity.

In the microflow system shown in Figure 1, Zn-enolate 5a and an excess amount of dizinc 1 was protonated with methanol in M2. Subsequently, instead of protonation, the resulting reaction mixture via R1 was introduced to a THF solution of ketones 9a-c as shown in Scheme 3. Although dienolate 5 was treated with an excess amount of ketone, 5 reacted with only one molar equivalent of ketone to afford the corresponding aldol adducts 10a-c diastereoselectively.13

Nuclear magnetic resonance spectra were taken on Varian UNITY INOVA 500 (1H, 500 MHz; 13C, 125.7 MHz) spectrometer using tetramethylsilane for 1H NMR as an internal standard (δ = 0 ppm), CDCl3 for 13C NMR as an internal standard (δ = 77.0 ppm).

1H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), coupling constants (Hz), and integration. High-resolution mass spectra were obtained with a Thermo Fisher SCIENTIFIC EXACTIVE (ESI, APCI). Infrared (IR) spectra were determined on a SHIMADZU FTIR-8200PC spectrometer. Melting points were determined using a YANAKO MP-500D. TLC analyses were performed by means of Merck Kieselgel 60 F254 (0.25 mm) Plates. Visualization was accomplished with UV light (254 nm) and an aqueous vanillin solution followed by heating. Flash column chromatography was carried out using Kanto Chemical silica gel (spherical, 40–100 μm).

Unless otherwise noted, commercially available reagents were used without purification. Tetrahydrofuran, Dehydrated stabilizer free —Super— was purchased from Kanto Chemical Co., stored under argon, and used as it is. Zinc powder was used after washing with 10% HCl according to the reported procedure.15

Preparation of bis(iodozincio)methane (1)
A mixture of pure zinc dust (150 mmol), diiodomethane (1.0 mmol), and PbCl2 (0.005 mmol) in THF (5.0 mL) was sonicated for 1 h in an ultrasonic cleaner bath under Ar. When pyrometallurgy zinc dust was used instead of pure zinc, it was not necessary to add PbCl2. Both pure zinc and pyrometallurgy zinc are commercially available. Diodiomethane (50 mmol) in THF (45 mL) was added dropwise to the mixture over 30 min at 0 °C with vigorous stirring. The mixture was then stirred for 4 h at 25 °C.

After the stirring was stopped, the reaction vessel was allowed to stand undisturbed for several hours. Excess zinc was separated by sedimentation. 1H NMR spectra of the obtained supernatant showed a broad singlet at −1.2 ppm at 0 °C, which corresponded to the methylene proton of 1. The supernatant was used in further reactions as a solution of 1 in THF (0.1–0.5 M). The concentration of 1 was estimated by 1H NMR analysis using 2,2,3,3-tetramethylbutane as an internal standard. Bis(iodozincio)methane in THF can be kept for at least a month in a sealed reaction vessel.

(1E,5E)-1,6-diphenylhexa-1,5-diene-3,4-dione (2a) [CAS RN [126201-33-0]].
Yellow solid. mp. 163.2–164.6 °C. 1H NMR (500 MHz, CDCl3) δ 7.87 (d, J = 16.5 Hz, 2H), 7.69–7.64 (m, 4H), 7.48 (d, J = 16.5 Hz, 2H), 7.46–7.41 (m, 6H). 13C NMR (125 MHz, CDCl3) δ 189.1, 147.8, 134.5, 131.3, 129.04, 128.95, 119.7. IR (KBr) 1706, 1669, 1607, 1595, 1574, 1449, 1032, 1001, 988, 755, 722, 698, 688, 557, 434 cm⁻¹.

(1E,5E)-1,6-Bis(4-methylphenyl)hexa-1,5-diene-3,4-dione (2b) [CAS RN [263249-11-2]].
Yellow solid. mp. 182.5–185.3 °C. 1H NMR (500 MHz, CDCl3) δ 7.84 (d, J = 16.5 Hz, 2H), 7.55 (d, J = 8.0 Hz, 4H), 7.41 (d, J = 16.0 Hz, 2H), 7.32 (d, J = 8.0 Hz, 4H), 2.40 (s, 6H). 13C NMR (125 MHz, CDCl3) δ 189.5, 147.9, 142.1, 131.8, 129.8, 129.0, 118.9, 21.6. IR

Conclusion
In conclusion, the reaction of bis(iodozincio)methane 1 with divinyl-1,2-diketones 2 afforded cycloheptene-1,3-diones 6 efficiently via a reactive cis-divinylcyclopropane derivative as the key-intermediate. Bis(iodozincio)methane was found to be a unique reagent to perform a nucleophilic cyclopropanation reaction with vicinal electrophiles such as 1,2-diketone, and affords reactive cyclopropanol derivatives efficiently.14 Although classical batch reactions required a careful temperature control to suppress the side-reactions of the product with the starting substrate, the microflow system removed the reactive product from the reaction site continuously, thus improving the yield of the desired product.

Experimental Section

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(KBr) 1669, 1595, 1560, 1512, 1307, 1328, 1295, 1183, 993, 806, 684, 428 cm⁻¹.

**(&)**

**Chemistry**

8.82 (dq, J = 16.0 Hz, 2H), 7.62 (dt, J = 9.0, 2.5 Hz, 4H), 7.33 (d, J = 16.0 Hz, 2H), 6.94 (dt, J = 9.0, 2.5 Hz, 4H), 3.86 (s, 6H). ¹³C NMR (125 MHz, CDCl₃) δ 189.6, 162.4, 147.5, 130.9, 119.7, 114.5, 55.5. IR (KBr) 1680, 1671, 1593, 1568, 1509, 1421, 1282, 1253, 1175, 1030, 996, 816, 790, 552 cm⁻¹.

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**Preparation of (5R*,6S*)-5,6-diphenylcycloheptane-1,3-dione (6a)**

(37.5%) mixture was then purified by column chromatography. The combined organic layers were washed with brine and dried over sodium sulfate. Purification by silica gel column chromatography (hexane/ethyl acetate) gave the title compound in 84% yield (233 mg).

White solid. mp. 106.7–112.8 °C. ¹¹H NMR (500 MHz, CDCl₃) δ 7.18–7.10 (m, 6H), 6.74–6.69 (m, 4H), 3.95 (d, J = 17.5 Hz, 1H), 3.74–3.67 (m, 2H), 3.57 (d, J = 17.5 Hz, 1H), 3.22 (dd, J = 15.5, 11.0 Hz, 2H), 2.79 (dd, J = 15.5, 5.0 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 204.5, 139.3, 128.2, 128.1, 127.1, 58.2, 46.2, 45.8. IR (KBr) 3060, 3030, 2969, 2889, 1710, 1600, 1491, 1452, 1383, 1284, 1255, 1240, 1220, 1133, 1076, 806, 755, 738, 712, 537, 462 cm⁻¹.

**(5R*,6S*)-5,6-Bis(4-methylphenyl)cycloheptane-1,3-dione (6b)**

Yellow solid. mp. 129.2–131.3 °C. ¹¹H NMR (500 MHz, CDCl₃) δ 6.45 (d, J = 8.0 Hz, 4H), 6.62 (d, J = 8.0 Hz, 4H), 3.95 (d, J = 17.0 Hz, 1H), 3.69–3.62 (m, 2H), 3.55 (d, J = 17.0 Hz, 1H), 3.17 (dd, J = 15.5, 11.0 Hz, 2H), 2.77 (dd, J = 15.0, 5.0 Hz, 2H), 2.27 (s, 6H). ¹³C NMR (125 MHz, CDCl₃) δ 204.7, 136.6, 136.3, 128.8, 128.2, 58.3, 46.1, 45.8, 20.9. IR (KBr) 3024, 2954, 1715, 1513, 1248, 1196, 1019, 806, 480 cm⁻¹. HRMS Calcd for C₂₃H₂₂O₂: M⁺ 306.1620. Found: m/z 306.1625.
(55,6S*)-5,6-Dimethylcycloheptane-1,3-dione (6i).
Colorless liquid. Ref. 0.28 (Hexane/EOAC = 3/1). 13C NMR (500 MHz, CDCl3) δ 3.43 (s, 2H), 2.59 (dd, J = 14.0, 3.0 Hz, 2H), 2.44 (dd, J = 14.0, 8.5 Hz, 2H), 1.87 – 1.77 (m, 2H), 1.09 (d, J = 6.5 Hz, 6H). 13C NMR (125 MHz, CDCl3) δ 205.4, 59.1, 50.3, 38.0, 20.7. IR (neat) 2963, 2360, 1698, 1611, 1459, 1388, 1252 cm−1. HRMS Calcd for C10H14O3: M+ 154.0994. Found: m/z 154.0998.

(55S*,6R*)-5,6-Dimethylcycloheptane-1,3-dione (6j).
Colorless liquid. Ref. 0.28 (Hexane/EOAC = 3/1). 13C NMR (500 MHz, CDCl3) δ 3.43 (s, 2H), 2.59 (dd, J = 14.0, 3.0 Hz, 2H), 2.44 (dd, J = 14.0, 8.5 Hz, 2H), 1.87 – 1.77 (m, 2H), 1.09 (d, J = 6.5 Hz, 6H). 13C NMR (125 MHz, CDCl3) δ 205.4, 59.1, 50.3, 38.0, 20.7. IR (neat) 2963, 2360, 1698, 1611, 1459, 1388, 1252 cm−1. HRMS Calcd for C10H14O3: M+ 154.0994. Found: m/z 154.0990.
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d(\text{J} = 17.5 \text{ Hz}, 1H), 3.37 (dd, \text{J} = 9.0, 2.0 \text{ Hz}, 1H), 3.11 (dd, \text{J} = 17.5, 15.0 \text{ Hz}, 1H), 2.61 (dd, \text{J} = 17.5, 4.0 \text{ Hz}, 1H), 1.63–1.72 (2m, 2H) 1.24–1.46 (4m, 4H), 1.15 (dd, \text{J} = 16 \text{ Hz}, 1H), 0.98–1.07 (3m, 2H) 0.47 (t, \text{J} = 11.5 \text{ Hz}, 1H). \text{13}C NMR (CDCl3) δ 211.3, 205.3, 140.6, 137.7, 129.5, 128.0, 127.9, 127.0, 126.8, 77.2, 73.8, 60.9, 49.3, 43.8, 43.8, 37.5, 35.9, 25.4, 21.8, 21.1. IR (KBr) 3510.6, 2960.9, 2938.7, 2852.8, 1692.6, 1495.7, 1457.3, 1398.5, 1380.1, 1359.9, 1287.2, 1175.7, 1156.4, 1139.0, 1076.3, 982.8, 845.8, 765.8, 708.9. cm–1. HRMS (ESI) Calcd for C25H26O3Cl: [M+Cl]+, 411.1721. Found: m/z 411.1736.

\( \text{(5S*,6S*)-4-(1-hydroxycyclohexyl)-5,6-diphenylcycloheptane-1,3-dione (10b)} \)

Yellow solid. mp. 185.0–185.5 °C. \text{1H NMR (CDCl3)} δ 7.10–7.16 (m, 6H), 6.73 (d, \text{J} = 6.5 \text{ Hz}, 2H), 6.66–6.64 (m, 2H), 3.89 (dd, \text{J} = 9.5, 4.0 \text{ Hz}, 1H), 3.77–3.84 (m, 2H), 3.59 (dd, \text{J} = 17.5, 10.0 \text{ Hz}, 1H), 3.46 (d, \text{J} = 2.5 \text{ Hz}, 1H), 3.28 (dd, \text{J} = 9.0, 2.0 \text{ Hz}, 1H) 3.21 (dd, \text{J} = 17.5, 15.5, 10.0 \text{ Hz}, 1H), 2.64 (ddd, \text{J} = 17.5, 4.0, 1.0 \text{ Hz}, 1H), 1.84–1.89 (m, 1H) 1.71–1.78 (m, 1H) 1.74–1.89 (m, 4H) 0.93–0.97 (m, 1H) 0.41–0.47 (m, 1H). \text{13}C NMR (CDCl3) δ 210.5, 205.6, 140.6, 137.6, 129.6, 128.0, 127.9, 127.8, 127.0, 82.7, 77.2, 61.7, 59.5, 50.4, 43.9, 41.0, 38.3, 23.4, 22.3 IR (KBr) 3525.1, 2974.4, 2924.2, 2869.2, 1714.8, 1693.6, 1493.9, 1456.3, 1382.1, 1266.3, 1456.3, 1382.1, 1266.3, 1232.6, 1139.0, 1096.6, 1003.0, 767.7, 708.9. cm–1. HRMS (ESI) Calcd for C25H26O3Cl: [M+Cl]+, 397.1565. Found: m/z 397.1579.

\( \text{(5S*,6S*)-4-(2-hydroxypropan-2-yl)-5,6-diphenylcycloheptane-1,3-dione (10c)} \)

Yellow solid. mp. 111.2–112.0 °C. \text{1H NMR (CDCl3)} δ 7.11–7.18 (m, 6H), 6.67 (d, \text{J} = 7.0 \text{ Hz}, 2H), 6.63–6.30 (m, 2H), 3.86–3.80 (m, 3H), 3.75 (dd, \text{J} = 9.0, 3.5 \text{ Hz}, 1H), 3.61 (dd, \text{J} = 18.0, 1.0 \text{ Hz}, 1H), 3.35 (dd, \text{J} = 9.0, 2.0 \text{ Hz}, 1H), 3.14 (ddd, \text{J} = 18.0, 1.4, 1.0 \text{ Hz}, 1H), 2.62 (ddd, \text{J} = 18.0, 3.5, 1.0 \text{ Hz}, 1H) 1.20 (s, 3H) 0.64 (s, 3H). \text{13}C NMR (CDCl3) δ 210.9, 205.3, 140.5, 137.7, 129.4, 128.0, 127.9, 127.1, 126.9, 72.4, 61.2, 60.4, 50.2, 43.9, 43.8, 30.2, 28.5. IR (KBr) 3500.6, 2974.4, 2712.5, 1685.9, 1495.9, 1455.4, 1397.2, 1380.1, 1362.8, 1235.5, 1159.7, 1158.3, 1136.1, 1095.6, 1078.3, 958.7, 767.7, 704.1. cm–1. HRMS (ESI) Calcd for C25H26O3Cl: [M+Cl]+, 371.1408. Found: m/z 371.1422.

Notes and references


13. The structure of 10a was determined by a single crystal X-ray analysis (see ref 10). The structure of the other products 10b,c was determined by analogy of the structure of 10a.