## Note

# Metathesis-Mediated Synthesis of (*R*)-10-Methyl-2-tridecanone, the Southern Corn Rootworm Pheromone<sup>\*</sup>

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(*R*)-10-Methyl-2-tridecanone, the female sex pheromone of the southern corn rootworm (*Diabrotica undecimpunctata howardi* Barber), was synthesized in 9 steps from methyl (*S*)-3-hydroxy-2-methylpropanoate in a 15.7% overall yield. Olefin cross metathesis between (*R*)-6-methyl-1-nonene and 5-hexen-2-one employing Grubbs' first-generation catalyst was the key step of the synthesis.

Key words: Diabrotica undecimpunctata howardi Barber; 10-methyl-2-tridecanone; Grignard coupling; metathesis; pheromone

The development of efficient synthetic methods for the pheromones of insect pests is a valuable task of chemists. *Diabrotica undecimpunctata howardi* Barber (Coleoptera: Chrysomelidae), known as the southern corn rootworm or cucumber beetle, is an economically important pest. Its female-produced pheromone was identified in 1983 as (*R*)-10-methyl-2-tridecanone (1) by Guss *et al.*,<sup>2)</sup> after examining the pheromone activity of both (*R*)- and (*S*)-1 of synthetic origin.<sup>3)</sup> A number of syntheses of (*R*)-1 have been reported<sup>4–8)</sup> since then, including our own.<sup>5)</sup> Chen and co-workers published in 2009 an asymmetric synthesis of (*R*)-1 employing a stoichiometric amount of a chiral auxiliary.<sup>8)</sup>

This Note reports our new synthesis of (R)-1 by using olefin cross metathesis<sup>9,10)</sup> as the key step and employing the readily available chiral building block, methyl (*S*)-3-hydroxy-2-methylpropanoate (**2**) as the starting material. The present synthesis is more economical than Chen's, because no chiral auxiliary is required.

Scheme 1 shows that the target pheromone (R)-1 could be prepared by olefin cross metathesis between (R)-6-methyl-1-nonene (A) and 5-hexen-2-one (B). The former (A) can be synthesized from readily available C (= 2), while the latter (B) is commercially available.

Scheme 2 summarizes the preparation procedure. Methyl (*S*)-3-hydroxy-2-methylpropanoate (**2**) was converted to known (*S*)-**3** in a 73% yield as previously reported.<sup>11)</sup> Chain-extension of (*S*)-**3** to (*R*)-**4** was achieved with ethylmagnesium bromide in the presence of dilithium tetrachlorocuprate in THF under Schlosser conditions<sup>12)</sup> to give (*R*)-**4** in a 63% yield. In this particular case, 2.8 equivalents of the Grignard reagent and 0.69 mol % of the catalyst were necessary to convert (*S*)-**3** to (*R*)-**4** in an acceptable yield of 63%, presumably due to steric congestion caused by methyl-branching at



Scheme 1. Retrosynthetic Analysis of (R)-1.

C-2 of (*S*)-**3**. Treatment of (*R*)-**4** with acidic methanol gave alcohol (*R*)-**5**. The enantiomeric purity of (*R*)-**5** was estimated as approximately 95% ee by a <sup>1</sup>H-NMR analysis of the corresponding Mosher ester [ $\alpha$ -methoxy- $\alpha$ -trifluoromethylphenylacetate (MTPA ester)].<sup>13</sup> Alcohol (*R*)-**5** was tosylated to give (*R*)-**6**. The second chain-extension of (*R*)-**6** with 3-butenyl bromide proceeded smoothly to give (*R*)-**7** (= **A**) in an 83% yield, employing relatively large amounts of the bromide (2.8 equivalents) and the catalyst (2.0 mol %) based on our previous experience with the conversion of (*S*)-**3**.

Olefin cross metathesis between (*R*)-7 and 5-hexen-2one (**B**) was best carried out using an excess (6.2 equivalents) of the latter to fully utilize (*R*)-7 to give the desired (*R*)-10-methyl-5-tridecen-2-one (**8**) as an *E*/*Z*mixture (*E*/*Z* = 2 : 1) in a 73% yield based on (*R*)-7. Hydrogenation of (*R*)-8 over 10% palladium-charcoal in ethyl acetate finally yielded (*R*)-10-methyl-2-tridecanone (**1**) as an oil,  $[\alpha]_D^{23} - 1.39$  (*c* 4.09, CHCl<sub>3</sub>) {ref. 5,  $[\alpha]_D^{23} - 1.4$  (*c* 3.017, CHCl<sub>3</sub>)}. Its IR and NMR data were identical to those previously reported.<sup>2,5)</sup> Although direct estimation of the enantiomeric purity of (*R*)-**1** could not be achieved by GC with a chiral stationary phase (Chiramix<sup>®</sup>), the purity would have been high enough to reflect the high enantiomeric purity of intermediate (*R*)-**5**.

In conclusion, female pheromone (*R*)-1 of the southern corn rootworm was synthesized in nine steps with a 15.7% overall yield, starting from readily available chiral building block (*S*)-2. The present Note provides another example of the usefulness of the olefin cross metathesis reaction in pheromone synthesis.<sup>11,14,15</sup>

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\* Pheromone Synthesis, Part 248, See Ref. 1 for Part 247.



#### Scheme 2. Synthesis of (R)-1.

Reagents: a) EtMgBr, Li<sub>2</sub>CuCl<sub>4</sub>, THF (63%); b) *p*-TsOH, MeOH (71%); c) *p*-TsCl, C<sub>5</sub>H<sub>5</sub>N (89%); d) CH<sub>2</sub>=CH(CH<sub>2</sub>)<sub>2</sub>MgBr, Li<sub>2</sub>CuCl<sub>4</sub>, THF (83%); e) CH<sub>2</sub>=CH(CH<sub>2</sub>)<sub>2</sub>COMe, Grubbs I catalyst [(Cy<sub>3</sub>P)<sub>2</sub>Ru(=CHPh)Cl<sub>2</sub>], CH<sub>2</sub>Cl<sub>2</sub> (73% of **8** based on **7**); f) H<sub>2</sub>, 10% Pd–C, EtOAc (89%).

#### Experimental

All boiling point (bp) data are uncorrected values. Refractive indices ( $n_D$ ) were measured with an Atago DR-M2 refractometer, and optical rotation values were measured with a Jasco P-1020 polarimeter. IR spectra were measured with a Jasco FT/IR-410 spectrometer, and <sup>1</sup>H-NMR spectra (400 MHz, TMS at  $\delta = 0.00$  as the internal standard) and <sup>13</sup>C-NMR spectra (100 MHz, CDCl<sub>3</sub> at  $\delta = 77.0$  as the internal standard) were recorded by a Jeol JNM-AL 400 spectrometer. GC-MS data were measured with an Agilent Technologies 5975 Inert XL instrument, and HRMS data were recorded on an Agilent Technologies 6530 Accurate Mass Q-TOF LC/MS instrument. Column chromatography was carried out on Merck Kieselgel 60 Art. 1.07734.

(R)-2-Methyl-1-pentanol THP ether (4). A solution of 3 (20.0 g, 60.9 mmol) in dry THF (60 mL) was added dropwise to a stirred and cooled solution of ethylmagnesium bromide in Et<sub>2</sub>O (3 M, 41 mL, 123 mmol, 2.02 eq.) and dry THF (80 mL) at -60--40 °C in an Ar atmosphere. A solution of Li2CuCl4 in THF (0.1 M, 3.1 mL, 0.31 mmol, 0.51 mol %) was subsequently added dropwise to the stirred mixture. and the temperature was gradually raised to room temperature. After stirring for 4 d, the mixture was cooled again to -70 °C. An additional solution of ethylmagnesium bromide in Et<sub>2</sub>O (3 M, 15 mL, 45 mmol, 0.74 eq.) and a solution of Li2CuCl4 in THF (0.1 M, 1.1 mL, 0.11 mmol, 0.18 mol %) were successively added to the stirred mixture which was left to stand overnight with a gradual increase in temperature to room temperature. The mixture was poured into an ice-cooled dil. NH<sub>4</sub>Cl solution and extracted with Et2O. The Et2O solution was successively washed with water and brine, dried (MgSO<sub>4</sub>), and concentrated in vacuo. The residue (20.2 g) was chromatographed over SiO<sub>2</sub> (150 g). Elution with hexane/EtOAc (15:1) gave 7.09 g (63%) of 4 as an oil,  $n_{\rm D}^{21}$  1.4385;  $[\alpha]_{\rm D}^{24}$  -0.05 (c 3.94, Et<sub>2</sub>O); IR  $\nu_{\rm max}$  (film) cm<sup>-1</sup>: 1128 (m, C-O-C), 1080 (m, C-O-C), 1063 (m, C-O-C), 1032 (s, C-O-C); <sup>1</sup>H-NMR  $\delta_{\rm H}$  (400 MHz, CDCl<sub>3</sub>): 0.88–0.94 (6H, m, CH<sub>3</sub> × 2), 1.04– 1.14 (1H, m), 1.23-1.45 (3H, m), 1.49-1.62 (4H, m), 1.67-1.78 (2H, m), 1.78-1.88 (1H, m), 3.11-3.26 (1H, m), 3.47-3.63 (2H, m), 3.83-3.90 (1H, m), 4.55-4.58 (1H, m); GC-MS [HP-5MS column, 5% phenylmethylsiloxane,  $30 \text{ m} \times 0.25 \text{ mm}$  i.d.; 60.7 kPa pressure; 70-230 °C +10 °C/min temperature)]:  $t_{\rm R}$  9.06 min (33.5%) and 9.07 min (65.6%, the diastereomer resulting from stereochemistry of the protective group). MS of 4 (former peak, 70 eV, EI): m/z: 185 (0.4)  $[(M^+ - 1), C_{11}H_{22}O_2], 115 (8), 101 (9), 85 (100), 56 (18), 43 (24).$  MS of the latter peak was indistinguishable from that of the former.

(R)-2-Methyl-1-pentanol (5). p-Toluenesulfonic acid monohydrate (2.04 g, 10.7 mmol) was added to a solution of 4 (17.3 g, 92.9 mmol) in methanol (170 mL). The solution was stirred and heated at reflux for 5 h, before being diluted with water and extracted with  $Et_2O$ . The combined  $Et_2O$  solution was successively washed with an NaHCO<sub>3</sub> solution and brine, dried (MgSO<sub>4</sub>), and concentrated under atmo-

spheric pressure. The residue (21.2 g) was chromatographed over SiO<sub>2</sub> (190 g), elution with pentane/Et<sub>2</sub>O (10:1-3:1) giving 6.78 g (71%) of 5 as a colorless oil. A portion of this oil was distilled to give an analytical sample, bp 91–92 °C at 98 Torr,  $n_D^{25}$  1.4153;  $[\alpha]_D^{25}$  +13.3 (c 4.08, Et<sub>2</sub>O); IR v<sub>max</sub> (film) cm<sup>-1</sup>: 3336 (s, O–H), 1045 (s, C–O); <sup>1</sup>H-NMR  $\delta_{\rm H}$  (400 MHz, CDCl<sub>3</sub>): 0.89–0.93 (6H, m, CH<sub>3</sub> × 2), 1.05–1.14 (1H, m), 1.25-1.44 (4H, m), 1.57-1.67 (1H, m), 3.38-3.54 (2H, m, CH<sub>2</sub>OH); GC-MS [same conditions as those for 4]:  $t_R$  2.9 min (99.4%). MS of **5** (70 eV, EI) m/z: 101 (0.1) [(M<sup>+</sup> - 1), C<sub>6</sub>H<sub>14</sub>O], 84 (17), 71 (50), 55 (34), 43 (100). HRMS of 5 [APCI ionization; positive polarity;  $6\,\mu A$  corona current;  $N_2$  (50 psi) nebulizer;  $N_2$  (5 L/min, 350 °C) drying gas; 350 °C vaporizer; 3500 V capillary current; 0.01 м HCO<sub>2</sub>H aq/MeOH (10/90) eluant]: calcd. for  $C_6H_{15}O$  [(M + H)<sup>+</sup>], 103.1117; found, 103.1117. The enatiomeric purity of 5 was determined by comparing the <sup>1</sup>H-NMR spectrum of the corresponding (R)-MTPA ester (4.15-4.16 ppm, m, CH<sub>2</sub>OMTPA) with that of the (S)-MTPA ester (4.05-4.09 and 4.22-4.26 ppm, m, CH<sub>2</sub>OMPTA). Neither spectrum exhibited any signals from the other isomer, indicating the enantiomeric purity of 5 to be over 95% ee.

(R)-2-Methylpentyl tosylate (6). Tosyl chloride (6.08 g, 31.9 mmol) was added portionwise to a stirred and ice-cooled solution of 5 (2.55 g, 25.0 mmol) in dry pyridine (25 mL) at 5-10 °C. After stirring for 3 h at 5-10°C, water was added to the mixture until the precipitate had dissolved, and stirring was continued for 5 min. It was then diluted with an ice-dil. HCl mixture and extracted with Et<sub>2</sub>O. The combined Et<sub>2</sub>O solution was successively washed with water, a dil. CuSO<sub>4</sub> solution, water, an NaHCO<sub>3</sub> solution and brine, dried (MgSO<sub>4</sub>), and concentrated in vacuo. The residue (5.89 g) was chromatographed over SiO<sub>2</sub> (90 g). Elution with hexane/EtOAc (15:1) gave 5.67 g (89%) of 6 as an oil,  $n_D^{23}$  1.4957;  $[\alpha]_D^{22}$  -1.91 (c 4.09, Et<sub>2</sub>O); IR  $\nu_{max}$  (film) cm<sup>-1</sup>: 1599 (m, arom. C=C), 1360 (s, SO<sub>2</sub>), 1178 (s, SO<sub>2</sub>), 1097 (m, C-O), 968 (s), 941 (m), 839 (m), 814 (m), 667 (s), 555 (s); <sup>1</sup>H-NMR  $\delta_{\rm H}$  $(400 \text{ MHz}, \text{ CDCl}_3)$ : 0.84 (3H, t,  $J = 7.0 \text{ Hz}, \text{ CH}_2\text{CH}_3$ ), 0.88 (3H, d, *J* = 6.8 Hz, CHC*H*<sub>3</sub>), 1.05–1.35 (4H, m), 1.74–1.83 (1H, m), 2.45 (3H, s, C<sub>6</sub>H<sub>4</sub>CH<sub>3</sub>), 3.78–3.90 (2H, m, CH<sub>2</sub>OTs), 7.34 (2H, d, J = 8.0 Hz), 7.79 (2H, d, J = 8.4 Hz).

(R)-6-Methyl-1-nonene (7). A solution of the Grignard reagent (2.8 eq.) was prepared from 3-butenyl bromide (3.27 g, 24.2 mmol) and Mg (0.65 g, 27 mmol) in dry THF (9 mL) in an Ar atmosphere. This was diluted with dry THF (8 mL), and the solution added dropwise to a stirred and cooled solution of **6** (2.22 g, 8.66 mmol) in dry THF (10 mL) at -60 - 40 °C in an Ar atmosphere. A solution of Li<sub>2</sub>CuCl<sub>4</sub> in THF (0.1 M, 1.7 mL, 0.17 mmol, 2.0 mol%) was subsequently added dropwise to the stirred mixture, and the temperature was gradually raised to room temperature. The mixture was stirred overnight, before being poured into an ice-dil. NH<sub>4</sub>Cl mixture and extracted with Et<sub>2</sub>O. The combined Et<sub>2</sub>O solution was successively washed with water and

brine, dried (MgSO<sub>4</sub>), and concentrated under atmospheric pressure. The residue (3.12 g) was chromatographed over SiO<sub>2</sub> (24 g), elution with pentane giving 1.00 g (83%) of **7** as an oil. A portion of this oil was distilled to give an analytical sample, bp (bath temperature) 120–130 °C at 100 Torr;  $n_D^{23}$  1.4208;  $[\alpha]_D^{25}$  –2.02 (*c* 4.07, pentane); IR  $\nu_{max}$  (film) cm<sup>-1</sup>: 3078 (m, C=CH<sub>2</sub>), 1641 (m, C=C), 993 (m), 910 (s), 739 (w); <sup>1</sup>H-NMR  $\delta_{\rm H}$  (400 MHz, CDCl<sub>3</sub>): 0.85 (3H, d, *J* = 7.2 Hz, CHCH<sub>3</sub>), 0.88 (3H, t, *J* = 6.8 Hz, CH<sub>2</sub>CH<sub>3</sub>), 1.05–1.15 (2H, m), 1.22–1.45 (7H, m), 1.99–2.08 (2H, m), 4.92–5.02 (2H, m, CH=CH<sub>2</sub>), 5.77–5.87 (1H, m, CH=CH<sub>2</sub>); <sup>13</sup>C-NMR  $\delta_{\rm C}$  (100 MHz, CDCl<sub>3</sub>): 14.4, 19.6, 20.1, 26.4, 32.4, 34.1, 36.5, 39.3, 114.0, 139.1; GC-MS [same conditions as those for **4**]:  $t_{\rm R}$  4.5 min (94.1%). MS of **7** (70 eV, EI) *m/z*: 140 (0.4) [M<sup>+</sup>, C<sub>10</sub>H<sub>20</sub>], 125 (0.5), 112 (2), 97 (76), 84 (33), 69 (49), 55 (100), 43 (55).

(R)-10-Methyl-5-tridecen-2-one (8). A solution of (Cy<sub>3</sub>P)<sub>2</sub>Ru-(=CHPh)Cl<sub>2</sub> (Grubbs I catalyst, 300 mg, 0.365 mmol; Aldrich), 5hexen-2-one (3.59 g, 36.6 mmol), and 7 (822 mg, 5.86 mmol) in dry CH2Cl2 (8 mL) was stirred and heated under reflux for 3.5 h in an Ar atmosphere. An additional solution of Grubbs I catalyst (150 mg, 0.182 mmol) in dry CH2Cl2 (2 mL) was added, and stirring and heating were continued for 6h. The mixture was left to stand for 2d at room temperature, before the solvent was removed, and the residue in a small amount of hexane was chromatographed over SiO<sub>2</sub> (40 g). Elution with hexane gave (4R,8EZ,13R)-4,13-dimethyl-8-hexadecene (9) and recovered 7 (140 mg total). Further elution with hexane/EtOAc (15:1) gave 900 mg (73%) of **8** as an oil. Properties of **8**:  $n_D^{17}$  1.4495;  $[\alpha]_D^{25}$  -1.84 (c 3.02, hexane); IR  $\nu_{max}$  (film) cm<sup>-1</sup>: 1720 (s, C=O), 1641 (w, C=C), 1161 (m), 968 (m), 739 (w); <sup>1</sup>H-NMR  $\delta_{\rm H}$  (400 MHz, CDCl<sub>3</sub>): 0.76-0.82 (6H, m, CH<sub>3</sub> × 2), 0.95-1.07 (2H, m), 1.13-1.36 (7H, m), 1.84-1.97 (2H, m), 2.06-2.08 (3H, m, COCH<sub>3</sub>), 2.15-2.28 (2H, m), 2.38-2.48 (2H, m, COCH<sub>2</sub>), 5.19-5.41 (2H, m, CH=CH);  $^{13}\text{C-NMR}~\delta_{\text{C}}$  (100 MHz, CDCl<sub>3</sub>): 14.4, 19.6, 20.1, 26.8, 26.9, 27.1, 27.5, 29.9, 32.3, 32.4, 32.8, 36.5, 36.6, 39.3, 43.58, 43.61, 127.4, 128.0, 131.2, 131.5, 208.3 (C=O); GC-MS [same conditions as those for 4]: t<sub>R</sub> 12.78 min [31.0%, (Z)-isomer], 12.84 min [65.8%, (E)-isomer]. MS of (E)-8 (70 eV, EI) m/z: 210 (1) [M<sup>+</sup>, C<sub>14</sub>H<sub>26</sub>O], 192 (1), 167 (2), 149 (9), 139 (2), 123 (9), 110 (20), 97 (28), 84 (17), 69 (18), 55 (25), 43 (100). MS of (Z)-8 was indistinguishable from that of (E)-8. HRMS of 8 [same conditions as those for 5]: calcd. for  $C_{14}H_{27}O$  [(M + H)<sup>+</sup>], 211.2056; found, 211.2054.

(R)-10-Methyl-2-tridecanone (1). 10% Palladium on charcoal (170 mg) was added to a solution of **8** (320 mg, 1.52 mmol) in EtOAc (10 mL). The suspension was stirred in an H<sub>2</sub> atmosphere (balloon) for 5.5 h at room temperature. The mixture was filtered through Celite, and the catalyst and Celite were washed with Et<sub>2</sub>O. The filtrate was concentrated *in vacuo*, and the residue (455 mg) was chromatographed over SiO<sub>2</sub> (7 g). Elution with hexane/EtOAc (50:1) gave 286 mg (89%) of **1** as an oil. A portion of this oil was distilled to give an analytical

sample, bp 98 °C at 3 Torr;  $n_D^{18}$  1.4387;  $[\alpha]_D^{23}$  –1.39 (*c* 4.09, CHCl<sub>3</sub>), {ref. 5:  $[\alpha]_D^{23}$  –1.4 (*c* 3.017, CHCl<sub>3</sub>)}; IR  $\nu_{max}$  (film) cm<sup>-1</sup>: 1720 (s, C=O), 1466 (m), 1360 (m), 1163 (m), 719 (w); <sup>1</sup>H-NMR  $\delta_{\rm H}$ (400 MHz, CDCl<sub>3</sub>): 0.83 (3H, d, *J* = 6.4 Hz, CHCH<sub>3</sub>), 0.87 (3H, t, *J* = 6.8 Hz, CH<sub>2</sub>CH<sub>3</sub>), 1.01–1.14 (2H, m), 1.15–1.43 (13H, m), 1.52– 1.62 (2H, t-like), 2.13 (3H, s, COCH<sub>3</sub>), 2.42 (2H, t, *J* = 7.2 Hz, COCH<sub>2</sub>); <sup>13</sup>C-NMR  $\delta_{\rm C}$  (100 MHz, CDCl<sub>3</sub>): 14.4, 19.6, 20.1, 23.9, 27.0, 29.2, 29.4, 29.79, 29.81, 32.4, 37.0, 39.4, 43.8, 209.1 (C=O); GC-MS [same conditions as those for **4**]:  $t_{\rm R}$  13.0 min (98.7%). MS of **1** (70 eV, EI) *m/z*: 212 (8) [M<sup>+</sup>, Cl<sub>4</sub>H<sub>28</sub>O], 194 (7), 169 (1), 151 (4), 123 (7), 110 (17), 96 (14), 85 (22), 71 (60), 58 (100), 43 (81). HRMS of **1** [same conditions as those for **5**]: calcd. for Cl<sub>4</sub>H<sub>29</sub>O [(M + H)<sup>+</sup>], 213.2213; found, 213.2211.

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