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Synthesis of (*Z*)-6-Heneicosen-11-one, a Possible Pheromone Component of the Hickory Tussock Moth, *Lophocampa caryae*

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Abstract

The compound (*Z*)-6-heneicosen-11-one, a possible pheromone component of the hickory tussock moth, *Lophocampa caryae*, and a known pheromone component of the Douglas fir tussock moth, *Orgyia pseudotsugata*, was synthesized by a new four-step procedure with a 39% overall yield and a six-step procedure incorporating a protecting group with a 28% overall yield. This new synthesis is comparable to other similar syntheses for this molecule in the literature.

Keywords: (*Z*)-6-heneicosadien-11-one; synthesis; pheromone; tussock moths

1. Introduction

The hickory tussock moth (HTM, *Lophocampa caryae*) belongs in the Erebidae family, as does the whitemarked tussock moth (WMTM, *Orgyia leucostigma*) and the Douglas fir tussock moth (DFTM, *Orgyia pseudotsugata*), which share similar caterpillar characteristics. It is native to and widely spread throughout eastern North America [1]. HTM outbursts can cause local defoliation in their native range (i.e., occasionally stripping a tree) [2]. The favourite food plant of the HTM is hickory, but it feeds on a wide range of deciduous trees, such as birch, ash, elm, oak, willow, maple, walnut, pecan, quaking aspen, basswood and black locust [2], and anecdotal information. In addition, the setae (hairs) on the caterpillars as well as the cocoons that incorporate larval setae are urticating and can cause painful and itchy skin rashes in people sensitive to them [2].

Given the commercial value of its host plants and the threat imposed upon human health, the study of HTM's pheromone chemistry (semiochemistry) is of significant interest. To our knowledge, the semiochemistry of HTM is completely unknown. Since DFTM and HTM belong in the family Erebidae, it is plausible that they share similar semiochemicals. (*Z*)-6-Heneicosen-11-one (**1**) (Figure 1) has been identified as the principal component of the DFTM sex pheromone [3] as well as a sexual stimulant to males of seven tussock moth species in the *Orgyia* and *Daysychira* genera [4,5]. We are currently investigating possible pheromone candidates effective towards HTM, of which we speculate (*Z*)-6-heneicosen-11-one (**1**) to be a possibility.

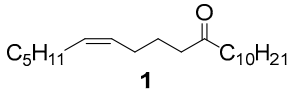


Figure 1. (*Z*)-6-heneicosen-11-one **1**.

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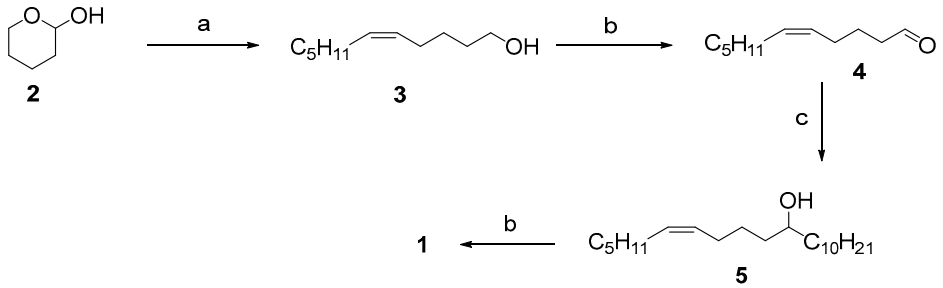
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2. Results

2.1. Background for the Synthesis of **1**

A literature search for the synthesis of **1** revealed several synthetic pathways [6–12]. Most of them were multistep procedures requiring the synthesis of either starting materials or intermediate reagents to establish the required 21-carbon chain. Two of these methods also involved the semihydrogenation of an acetylenic intermediate with Lindlar's catalyst or P2-nickel. Out of the reported methods, the most convenient was found to be the one explicated by Fetizon and Lazare [10]. The two key steps that implemented the 21-carbon chain in this reaction scheme were a Wittig reaction and a Grignard reaction (Scheme 1). Although the synthesis is only four steps, with 51% overall yield, their Wittig reaction is lengthened by the necessity of freshly distilled starting material, THF dried by distillation over KOH under N_2, and steam distillation during the workup phase. The three-step synthesis described by Herbert et al. [11] used a specialized ruthenium catalyst for the formation of the *Z*-alkene. However, their *Z*-alkene selectivity was 88%, much lower than the 98% reported by Fetizon and Lazare [10]. The synthesis reported by Jones et al. [12] involved the synthesis of an enol triflate as the starting material, on which the Grignard-triggered fragmentation as a key step to establishing the 21-carbon chain produced only a 58% yield.



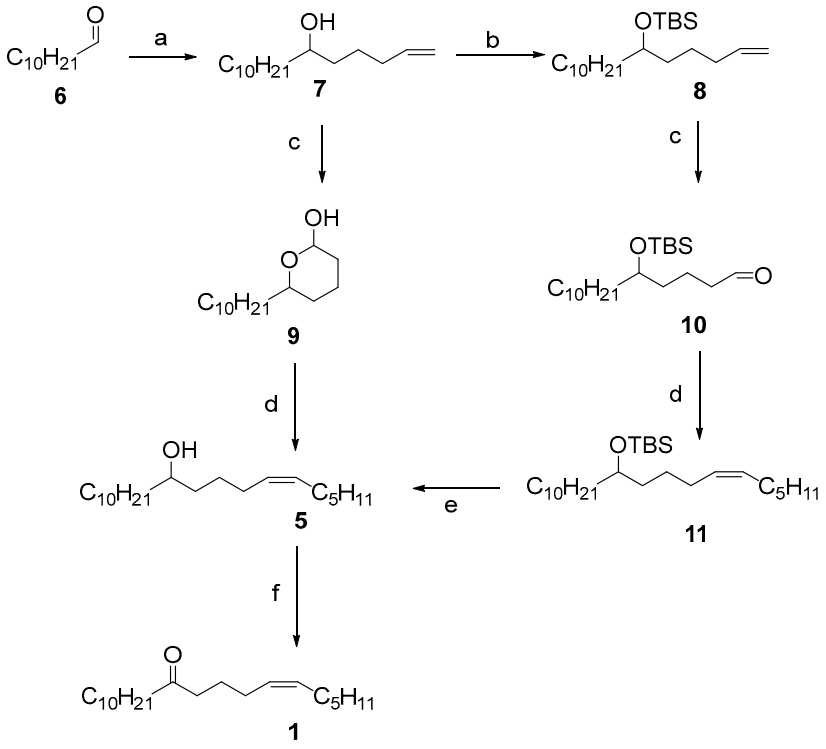
Scheme 1. Synthesis of **1** according to Fetizon and Lazare [10]. (a) Wittig reaction; *n*-hexyltriphenylphosphonium bromide, potassium *t*-butoxide, 60%. (b) Collins oxidation; CrO_3, pyridine, 90–95% for **4**, 95% for **1**. (c) Grignard reaction; *n*-decylmagnesium bromide, 100%.

We are reporting an alternative pathway for the synthesis of **1** (Scheme 2), which is similar to and runs parallel with the method shown in Scheme 1. We are confident of it being a fast, reliable and efficient pathway that is complimentary to the existing methodologies.

2.2. Proposed Synthesis of **1**

The synthesis begins with the Grignard coupling of commercially available *n*-undecanal **6** (Millipore-Sigma, St. Louis, MO, USA) with 5-bromo-1-pentene (Tokyo Chemical Industry, TCI, Portland, OR, USA) in Et_2O, resulting in an 85% yield of the secondary alcohol **7**. The Grignard reagent was easily prepared in situ from magnesium metal and 5-bromo-1-pentene. If preferred, it is also commercially available, but at a high price, CAD 702/100 mL, through Millipore Sigma. Thus, this was the only reagent we prepared in our synthetic process. Since TBS protection of the hydroxyl group could lead to a cleaner reaction and better yields, it was decided to approach the reaction scheme along two pathways. One is the shortest and most direct route that continued with the use of **7**, and the other is with the TBS-protected hydroxyl group in **8**. The TBS protection was accomplished through the reaction of **7** with TBSCl in CH_2Cl_2 in the presence of imidazole, resulting in a 69% yield of **8.** Ozonolysis of the terminal alkene in **7** and **8** resulted in the lactol **9** and an aldehyde **10** with 81% and 82% respective yields. Ozonolysis is a salient feature of our proposed synthesis that has not been reported thus far. The major advantage of this reaction is its very short reaction time of less than five minutes with a

high yield. To assemble the 21-carbon chain, **9** and **10** were subjected to lithium salt-free Wittig coupling, proven to have high stereoselectivity, with commercially available (TCI) *n*-hexyltriphenylphosphonium bromide. The ylide formation was accomplished with the use of sodium bis(trimethylsilyl)amide, which is a stronger base and preferred for its superior *Z* selectivity than the *t*-butoxide used by Fetizone and Lazare [10]. Both reactions successfully established the 21-carbon chain, with the internal *Z*-alkene resulting in 61% and 66% yields of **5** and **11**, respectively. GC/MS and NMR spectral data confirmed the near exclusivity of the Z-alkene, >95% in both cases. The *E*-isomer was undetectable by ^{1}H NMR (generally 5% detection limit) and GC/MS in either **5** or **11**, and it is known that lithium salt-free Wittig reactions of unstabilized ylides generally yields high *Z*-selectivity [13]. However, an advantage of the longer route is that the Wittig reaction on **10** to yield **11** produced fewer side-products and was generally cleaner than Wittig reaction on **9**, which also required two equivalents of the ylide because of the labile proton in **9**. The TBS-protecting group on **11** was easily cleaved with tetrabutylammonium fluoride to produce a 96% yield of **5**. It had identical spectroscopic properties to compound **5** prepared by the shorter route. Finally, pyridinium chlorochromate (PCC) oxidation of the secondary alcohol in **5** produced a 93% yield of the desired ketone **1**. The use of commercially available PCC with NaOAc as a buffer makes this reaction more convenient and efficient than the previously reported PCC oxidation with pyridine-chromic anhydride complex prepared at 0 °C [9,10]. Furthermore, it is much more efficient than the Swern oxidation reported by Herbert et. al. [11], which uses oxalyl chloride, Et_3N, and DMSO at −78 °C. It is highly exothermic and produces toxic and flammable CO as a byproduct.



Scheme 2. Proposed synthesis of (*Z*)-6-heneicosen-11-one (**1**). (a) Grignard reaction; 5-bromo-1-pentene + $Mg_{(s)}$, 85%. (b) Alcohol protection; TBSCl, imidazole, 69%. (c) Ozonolysis with reductive workup; (i) O_3, CH_2Cl_2, −78 °C; (ii) PPh_3, 81% for **9**, 82% for **10**. (d) Wittig reaction; *n*-hexyltriphenylphosphonium bromide + sodium bis(trimethylsilyl)amide, 61% for **5**, 66% for **11**. (e) Alcohol deprotection; tetrabutylammonium fluoride, 96%. (f) PCC oxidation; pyridinium chlorochromate, NaOAc, 93%.

3. Discussion

We have reported the synthesis of **1** through a short four-step synthesis and a six-step synthesis, with overall yields of 39% and 28%, respectively. Elimination of the TBS protection step to shorten the synthesis to four steps is a significant advantage, and this seemed to have negligible effects on the *Z*-selectivity of the crucial Wittig step to install the *Z*-alkene. Due to the commercial availability of all starting materials and reagents, as well as the easy execution of the reactions, this synthetic pathway is very convenient and reliable.

4. Materials and Methods

4.1. General Information

All nonaqueous reactions were conducted in flame-dried glassware under an argon atmosphere. Air-sensitive reagents were transferred via syringe through rubber septa (natural red rubber, Wheaton, Millville, NJ, USA). Reagents were purchased from commercial suppliers and directly used without further purification. Hexanes and ethyl acetate were obtained from VWR International, Mississauga, ON, Canada. Diethyl ether (anhydrous) was obtained from Millipore-Sigma, St. Louis, MO, USA. Tetrahydrofuran (anhydrous) was obtained from Millipore-Sigma. Dichloromethane (DCM) was obtained from Supelco, Burlington, MA, USA. Analytical thin-layer chromatography (TLC) was performed using SiliaPlate TLC plates (Silicycle, Québec, QC, Canada), precoated with 0.25 mm of UV_{254} silica gel on aluminum backing. Visualization was carried out by dipping the TLC plate in potassium permanganate solution (1.5 g $KMnO_4$ (Millipore-Sigma, St. Louis, MO, USA) 3.2 mL 1 M $NaOH_{(aq)}$ (Millipore-Sigma) 10 g K_2CO_3 (Millipore-Sigma) 200 mL H_2O), followed by baking with a heat gun. Flash column chromatography was performed using a glass column filled with 230- to 400-mesh Silia Flash® (Silicycle) without further purification. All compounds purified by SiO_2 chromatography were greater than 95% chemical purity as determined by GC/MS and/or NMR. Synthetic compounds were characterized by GC/MS on an Agilent 7890 GC and an Agilent 5975C inert mass selective detector (Agilent Technologies, Santa Clara, CA, USA) with a triple-axis detector in the electron impact (unit resolution, EI, 70 eV) mode to record low-resolution mass spectra (LRMS), with a GC column temperature program of 70 °C for 3 min, then an increase in temperature of 15 °C/min until the oven temperature reached 280 °C, which was then maintained for 23 min. An injector port temperature of 250 °C was used for all analyses, as was a Restek Rxi-17Sil MS capillary GC column (Chromatographic Specialties Inc., Brockville, ON, Canada, 30 m, 0.25 mm id, 0.25 μm film). NMR spectroscopy (^1H and ^{13}C, as well as 2-D NMR) was carried out on a Bruker Avance NEO 400 NMR spectrometer (Bruker Biospin Corporation, Billerica, MA, USA) in $CDCl_3$ with tetramethylsilane (TMS) or residual protic solvent (Millipore-Sigma) as internal standard. IR spectra were recorded on an Agilent Cary 630 FTIR spectrometer (Agilent Technologies) with ATR as neat samples. High-resolution mass spectra (HRMS) were obtained at Maritime Mass Spectrometry Laboratories, Halifax, NS, Canada, using a compact instrument from Bruker Daltonics (Bruker Corporation, Billerica, MA, USA). The ionization method used for low-resolution and high-resolution analysis was APCI (Atmospheric Pressure Chemical Ionization) or APPI (Atmospheric Pressure Photo Ionization), which generated the M + H ion (for APCI and APPI). $N_{2(g)}$ was used as the carrier gas for all samples.

4.2. Synthesis of Compounds **7**, **8**, **10**, **11**, **5**, **1** *and* **9**, *as Well as Wittig Reaction on Compound* **9**. *For Tabulated Spectral Data for All of These Compounds, as Well as the Spectra Themselves, See Supporting Information*

6-Hydroxy-1-hexadecene (**7**): A round-bottomed flask (rbf) containing $Mg_{(s)}$ (518 mg, 21.3 mmol, Millipore-Sigma) was flame-dried and cooled to RT. To this was added diethyl ether (Et_2O, 50 mL), 5-bromo-1-pentene (2.30 mL, 19.4 mmol, TCI), and $I_{2(s)}$ (10 mg, 0.039 mmol, Millipore-Sigma). The reaction was stirred at RT (it generated its own heat) for 1 h until most of the magnesium turnings were dissolved and the brown colour of the $I_{2(s)}$ was discharged. The Grignard reagent was then transferred via cannula with rinsing (Et_2O, 2 × 10 mL) to another flame-dried rbf. To this, undecanal (**6**) (1.00 mL, 4.84 mmol, Millipore-Sigma) was added slowly via syringe, and the reaction was stirred at RT for 24 h and then quenched with saturated $NH_4Cl_{(aq)}$ (50 mL, Millipore-Sigma). The organic layer was separated, and the aqueous layer was extracted with diethyl ether (Et_2O, 3 × 30 mL). The combined organic layers were washed with brine (50 mL) and dried (Na_2SO_4, Thermo Scientific, Mississauga, ON, Canada). Flash column chromatography on silica gel with 2:3 EtOAc:hexanes as eluent yielded **7** (990 mg, 4.13 mmol, 85%) as a white powder.

6-*tert*-Butyldimethylsilyloxy-1-hexadecene (**8**): An rbf containing **7** (4.06 g, 16.9 mmol) and CH_2Cl_2 (DCM, 150 mL) was charged with imidazole (4.61 g, 67.7 mmol, Millipore-Sigma) and then *tert*-butyldimethylsilyl chloride (TBSCl, 7.65 g, 50.8 mmol, TCI America). The reaction was stirred at RT for 24 h. The reaction was quenched with H_2O (70 mL), the layers were separated, and the aqueous layer was extracted with DCM (3 × 30 mL). The combined organic layers were washed with brine (50 mL) and dried (Na_2SO_4). Flash column chromatography on silica gel with 1:9 EtOAc:hexanes as eluent yielded **8** (4.12 g, 11.6 mmol, 69%) as a colourless, transparent liquid.

5-*tert*-Butyldimethylsilyloxypentadecenal (**10**): An rbf containing **8** (570 mg, 1.61 mmol), DCM (70 mL) and Sudan III (5.0 mg, Honeywell Fluka, Seelze, Germany) was cooled to −78 °C, and ozone was passed through it until the red colour of the Sudan III was discharged (5 min). Air was then passed through the reaction mixture for 15 min at −78 °C, then PPh_3 (422 mg, 1.61 mmol, Acros Organics, New Jersey, USA) was added, and the reaction was allowed to warm to RT and stirred for 2 h, then kept in the freezer overnight. After removal of the solvent in vacuo, flash column chromatography on silica gel with 1:9 EtOAc:hexanes as eluent yielded 5-*tert*-butyldimethylsilyloxypentadecenal **10** (470 mg, 1.32 mmol, 82%) as a colourless, transparent liquid.

(*Z*)-11-*tert*-Butyldimethylsilyloxy-6-heneicosene (**11**): A flame-dried rbf containing *n*-hexyltriphenylphosphonium bromide (1.10 g, 2.56 mmol, TCI America) and THF (30 mL,) was cooled to 0 °C and sodium bis(trimethylsilyl)amide (2.80 mL, 2.80 mmol, 1.0 M solution in THF, Millipore-Sigma) was added dropwise over 5 min. After stirring at 0 °C for 30 min, then RT for 1 h, the reaction mixture was cooled to −78 °C, and 5-*tert*-butyldimethylsilyloxypentadecenal **10** (600 mg, 1.69 mmol) in THF (15 mL) was added via cannula, with THF rinsing (2 × 5 mL). The reaction was allowed to warm to RT, then stirred for 24 h. At this point, H_2O (30 mL) was added, the layers were separated, and the aqueous layer was extracted with Et_2O (3 × 20 mL). The combined organic layers were washed with brine (50 mL), then dried (Na_2SO_4). Flash column chromatography on silica gel with 1:9 EtOAc:hexanes as eluent yielded **11** (474 mg, 1.12 mmol, 66%) as a colourless, transparent liquid.

(*Z*)-11-hydroxy-6-heneicosene (**5**): An rbf containing **11** (300 mg, 0.708 mmol) and THF (20 mL) was then charged with tetrabutylammonium fluoride (TBAF, 11.0 mL, 1.0 M, in THF, 11.0 mmol, Thermo Scientific), and the reaction was stirred at RT for 24 h. The reaction was quenched with H_2O (30 mL), the layers were separated, and the aqueous layer was extracted with Et_2O (3 × 20 mL). The combined organic layers were washed with

brine (50 mL) and dried (Na_2SO_4). Flash column chromatography on silica gel with 1:4 EtOAc:hexanes as eluent yielded **5** (211 mg, 0.681 mmol, 96%) as a colourless liquid.

(*Z*)-6-Heneiocosen-11-one (**1**): An rbf containing **5** (98.0 mg, 0.316 mmol), DCM (30 mL) and NaOAc (1.6 g, 20 mmol, Millipore-Sigma) was then charged with pyridinium chlorochromate (PCC, 2.0 g, 9.3 mmol, VWR International), and the reaction was stirred at RT for 24 h. After gravity filtration to remove most of the solids and removal of the solvent in vacuo, flash column chromatography on silica gel with 1:9 EtOAc:hexanes as eluent yielded **1** (90.8 mg, 0.295 mmol, 93%) as a colourless liquid.

6-^{n}Decyl-2-hydroxytetrahydropyran (**9**): An rbf containing **7** (990 mg, 4.13 mmol), DCM (75 mL) and Sudan III (10.0 mg) was cooled to −78 °C, and ozone was passed through it until the red colour of the Sudan III was discharged (10 min). Air was then passed through the reaction mixture for 15 min at −78 °C, then PPh_3 (1.08 g, 4.12 mmol) was added, and the reaction was allowed to warm to RT and stirred for 2 h, then kept overnight in the freezer. After removal of the solvent in vacuo, flash column chromatography on silica gel with 2:3 EtOAc:hexanes as eluent yielded **9** (810 mg, 3.35 mmol, 81%) as a white solid and as a mixture of two diastereomers in a 0.36:0.64 ratio. This is reflected in the ^{1}H and ^{13}C NMR spectra.

Wittig reaction on **9**: A flame-dried rbf was charged with *n*-hexyltriphenylphosphonium bromide (1.73 g, 4.03 mmol) and THF (30 mL), then cooled to 0 °C, and sodium bis(trimethylsilyl)amide (4.4 mL, 4.4 mmol, 1.0 M solution in THF) was added dropwise over 5 min. After stirring at 0 °C for 30 min, then RT for 1 h, the reaction mixture was cooled to −78 °C, and **9** (391 mg, 1.62 mmol) in THF (5 mL) was added via cannula, with THF rinsing (2 × 5 mL). The reaction was allowed to warm to RT, then stirred at RT for 24 h. At this point, H_2O (30 mL) was added, the layers were separated, and the aqueous layer was extracted with Et_2O (4 × 20 mL). The combined organic layers were washed with brine (40 mL), then dried (Na_2SO_4). Flash column chromatography on silica gel with 1:9 EtOAc:hexanes as eluent yielded **5** (305 mg, 98.4 mmol, 61%) as a colourless, transparent liquid.

Supplementary Materials: The following supporting information is available online. ^{1}H and ^{13}C NMR of **1** and all synthetic intermediates, as well as total ion chromatograms (TLCs) with low-resolution mass spectra (LRMS) of **1** and all synthetic intermediates.

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Data Availability Statement: All experimental data is available in the Section 4 and in the Supporting Information.

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Abbreviations

The following abbreviations are used in this manuscript:

HTM	Hickory tussock moth
WMTM	Whitemarked tussock moth
DFTM	Douglas fir tussock moth
THF	Tetrahydrofuran
TBSCl	*tert*-Butyldimethylsilyl chloride
TLC	Thin-layer chromatography
NMR	Nuclear magnetic resonance
GC/MS	Gas chromatography/mass spectrometry
LRMS	Low-resolution mass spectrum
HRMS	High-resolution mass spectrum
RBF	Round-bottomed flask
IR	Infrared
Et_2O	Diethyl ether
RT	Room temperature
DCM	Dichloromethane
PCC	Pyridinium chlorochromate
R_f	Retention factor
TIC	Total ion chromatogram

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