idazinecarboxylic Acid (10e). Saponification and workup as above for the hydrolysis of **10a** yields, from 1.0 g (2.64 mmol) of pyridazine ester **10d**, 0.75 g (81%) of acid **10e** as an off-white solid, mp 214–215 °C dec (with gas evolution). Crystallization from ethanol yields a colorless analytical sample: mp 215–215.5 °C dec (with gas evolution); IR ~3500 (br), 1710, 1370, 1280 (br), 1220 cm^{-1}; ^1H NMR ($CDCl_3$ + Me_2SO-d_6) δ 7.3 (m, 13 H, aromatic), 7.8 (m, 2 H, ortho protons, 6-phenyl), 9.09 (br s, 1 H, CO_2H); mass spectrum *m/e* (% base) 352 (12.7) M^+, 308 (90.3), 307 (100), 178 (99.0). M^+, calcd for $C_{23}H_{16}N_2O_2$: 352.12. Found: 352.15.

Decarboxylation of 10e. Preparation of 3,4,6-Triphenylpyridazine (10f). In a 25-mL round-bottom flask fitted with a $CaCl_2$ drying tube is placed 300 mg (0.855 mmol) of pyridazinecarboxylic acid **10e**. The flask is immersed in a 220 °C oil bath for 30 min. As the sample melts, vigorous gas evolution is observed. The yield of slightly yellow solid, mp 170–175 °C, is 0.26 g (99%). Recrystallization from ethanol affords colorless material: mp 170–172 °C (lit.[5] 171–172 °C); IR (KBr) 1580, 1560, 1395 cm^{-1}; ^1H NMR δ 7.2 (br m, 13 H, aromatic), 7.59 (s, 1 H, pyridazine ring H), 7.97 (m, 2 H, ortho protons, 6-phenyl ring); mass spectrum *m/e* (% base) 309 (54.0), 308 (99.6), 307 (100), 178 (99.4).

Preparation of Benzilacetylacetone Azine (11). A mixture of 4.5 g (0.02 mol) of benzil monohydrazone and 4.0 g (0.04 mol) of acetylacetone (**8c**) is heated at reflux in ethanol containing 100 mg of benzoic acid for 60 h. On cooling an orange solid precipitates which is isolated by filtration, yielding 4.48 g (73%) of **11**, mp 122–159 °C. ^1H NMR indicates that this is a 1.5:1 mixture of two isomers, A and B. Careful fractional crystallization from ethanol or carbon tetrachloride allows the isolation of both isomers. Isomer A: light yellow plates, mp 161–162 °C; IR ($CHCl_3$) 3100 (br), 3010, 1635, 1565, 1490 cm^{-1}; ^1H NMR δ 1.90 (s, 3 H, CH_3C=C), 1.99 (s, 3 H, $COCH_3$), 5.11 (s, 1 H, C=CH), 7.3 (m, 8 H, aromatic), 7.8 (m, 2 H, aromatic ortho to C=O), 12.91 (br s, 1 H, NH). Anal. Calcd for $C_{19}H_{18}N_2O_2$: C, 74.49; H, 5.92. Found: C, 74.25; H, 5.90. Isomer B: orange-yellow chunky crystals, mp 146–149 °C; IR ($CHCl_3$) 3100 (br), 3020, 1675, 1620, 1570 cm^{-1}; ^1H NMR δ 1.88 (s, 3 H, CH_3C=C), 2.18 (s, 3 H, CH_3C=O), 5.00 (s, 1 H, C=CH), 7.3 (m, 8 H, aromatic), 7.8 (m, 2 H, aromatic ortho to C=O), 13.47 (br s, 1 H, NH). Either pure isomer is converted to the equilibrium mixture (~1.5:1, A:B) on heating in ethanol for several hours.

Cyclization of 11. Preparation of 3,4-Diphenyl-5-acetyl-6-methylpyridazine (12) and 3,4-Diphenyl-6-methylpyridazine (10c). To a gently boiling solution of 115 mg (~1.78 mmol) of potassium hydroxide in 15 mL of ethanol is added 0.46 g (1.50 mmol) of azine **11** (mixture of isomers). A deep red color develops which fades rapidly. After heating for 5 min, the bulk of the ethanol is removed in vacuo and the residue partitioned between ether and 5% NaOH. The layers are separated, the organic layer thoroughly extracted with 5% HCl, and the acid extracts reserved. The ether layer is dried (Na_2SO_4) and evaporated in vacuo to yield 50 mg (12%) of **12** as a pale yellow solid. Purification by sublimation (140 °C, 1 mm) and recrystallization (CH_2Cl_2/heptane) yields an analytical sample: mp 131.5–132.5 °C; IR (KBr) 1695, 1440, 1375 cm^{-1}; ^1H NMR δ 1.86 (s, 3 H, CH_3C=O), 2.62 (s, 3 H, ring CH_3), 7.2 (br s, 10 H, aromatic); mass spectrum *m/e* (% base) 288 (87.9) M^+, 287 (100), 245 (75.1), 178 (28.3). Anal. Calcd for $C_{19}H_{16}N_2O$: C, 79.14; H, 5.59. Found: C, 79.17; H, 5.86.

Basification of the acid extracts (above) and extraction with methylene chloride yields, after drying (Na_2SO_4) and removal of the CH_2Cl_2 in vacuo, 0.29 g (79%) of **10c**, identical in all respects with the material prepared by decarboxylation of **10b**.

Registry No.—**7**, 5344-88-7; **8a**, 141-97-9; **8b**, 94-02-0; **8c**, 105-45-3; **9a**, 62139-81-5; **10a**, 62139-82-6; **10b**, 62139-83-7; **10c**, 13340-82-4; **10d**, 62139-84-8; **10e**, 62139-85-9; **10f**, 2272-58-4; **11a**, 62139-86-0; **11b**, 62139-87-1; **12**, 62139-88-2.

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2-Phenylthio-2-cyclopentenone, a Useful Synthon for 2,3-Disubstituted Cyclopentanones. Synthesis of *dl*-Methyl Dehydrojasmonate

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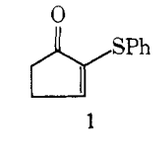
Received December 7, 1976

Recently, there has been an increasing amount of research devoted to the development of useful synthetic routes to 2,3-disubstituted cyclopentanones, mainly because of the interest in the pharmacologically important prostaglandins. However, most of the published routes either use as starting material the commercially expensive 2-cyclopentenone[1] or 2-alkylated 2-cyclopentenones,[2] which are not themselves readily available. 2-Phenylthio-2-cyclopentenone (**1**) is expected to be a very useful synthon for the preparation of several 2,3-disubstituted cyclopentanones, since it bears the adequate functionalities for conjugate alkylations,[1,3] or Michael-type additions followed by a regiospecifically directed[4] alkylation at the 2 position ensured by the thioether function. The described[5] syntheses of **1** are too laborious and expensive; therefore, a facile preparation was sought.

Oki[6] described the formation of 2-methylthio-2-cyclohexenone by the reaction of phenylsulfenyl chloride with 2-methylthiocyclohexanone, but did not exploit the preparative aspects of this interesting transformation. We reasoned that, since 2-phenylthiocyclopentanone is expected[7] to be the primary product from the reaction of phenylsulfenyl chloride with cyclopentanone, **1** could be prepared in a single step by reaction of the ketone with an excess of the sulfenyl chloride. Indeed, treatment of cyclopentanone with phenylsulfenyl chloride in dry acetonitrile, followed by chromatography on silica gel, afforded 55–65% yields of the pure unsaturated ketone **1**, based on cyclopentanone. The method[8] is quite economical, since the diphenyl disulfide formed in the reaction can be recovered and reconverted into phenylsulfenyl chloride.

Initial alkylation experiments of **1** with *n*-amylmagnesium



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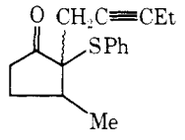

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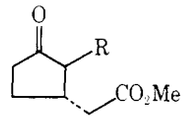

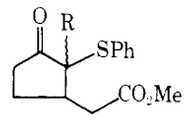

3, R = CH_2CH=CHEt-*cis*
4, R = CH_2C≡CEt
5, R = H
6, R = CH_2C≡CEt

bromide without copper salt catalysis resulted in about a 45% yield of 1,4-addition, as evidenced by spectroscopic analyses of the reaction products. This large proportion of conjugate addition may be possibly caused by the inductive or steric effects of the 2-phenylthioether group.

On the other hand, alkylation of **1** with lithium dimethylcuprate followed by quenching of the enolate with 2-pentynyl bromide, according to Coates' procedure,[3a] afforded, after preparative TLC, good yields of the ketone **2** as an epimeric mixture. No 5-alkylated ketone could be detected among the minor products, indicating that enolate equilibration[1,4] does not take place under the reaction conditions.

To further test the synthetic potentialities of the ketone **1**,

and also encouraged by its ready availability, we turned our attention to a practical synthesis of methyl jasmonate (**3**), a useful raw material in the perfume industry.[9]

Michael reaction of **1** with dimethyl malonate and sodium methoxide in methanol, followed by acid hydrolysis and decarboxylation of the crude adduct, and subsequent esterification of the resulting acid with methanol gave the expected ester **5** in 84% overall yield. The phenylthioether group in **5** was ready to serve its purpose, which was to guarantee the regiospecific alkylation[4] at the sulfur bearing carbon atom. Thus, alkylation of the keto ester **5** with 2-pentynyl bromide and sodium hydride in glyme gave the expected ester **6**, which was isolated from polymeric and other, nonidentified, minor products on preparative TLC, in 63% yield. Although compound **6** appeared homogeneous on analytical TLC, its NMR spectrum showed it to be a mixture of the two expected epimers at the newly alkylated center in the approximate ratio of 1.6:1. As the phenylthioether grouping was to be removed in the next step, generating a new epimeric center susceptible to equilibration, no endeavor was made to separate and further characterize the isomers. Instead, the epimeric mixture of **6** was treated with aluminum amalgam, a smooth cleavage of the thioether function taking place to afford, after purification on preparative TLC, a 92% yield of pure *dl*-methyl dehydrojasmonate (**4**), which was identical with the material previously prepared by other workers.[9a-c,g] Since **4** can be readily hydrogenated to *dl*-methyl jasmonate (**3**),[9a-c,g] its synthesis of the above procedure constitutes a new and efficient route to that interesting substance, and exemplifies a useful method of preparation of 2,3-disubstituted cyclopentanones.

Experimental Section

General. All solvents were distilled before use. Melting points were measured in a Kofler block and are uncorrected. Analytical and preparative TLC were run on fluorescent (GF_{254}) or Rhodamine 6G dyed Merck silica gel plates. ^1H NMR spectra were recorded on a Varian A-60D spectrometer using carbon tetrachloride as solvent and tetramethylsilane as internal standard. Infrared spectra were recorded on a Perkin-Elmer 137 instrument. Mass spectra were obtained on Varian MAT CH-5 and 731 spectrometers.

2-Phenylthio-2-cyclopentenone (1). Phenylsulfenyl chloride (4.40 g, 33 mmol) was added to a solution of cyclopentanone (0.84 g, 10 mmol) in dry acetonitrile (15 mL), cooled by a water bath. After stirring at room temperature for 2 h, the mixture, protected from moisture, was filtered and the residue washed with acetonitrile. The filtrate was evaporated under vacuum on the steam bath. To the residue was added boiling methanol (20 mL), the mixture reevaporated under vacuum, and the whole process repeated a second time. Chromatography of the oily residue on a silica gel column (eluting solvents: petroleum ether and petroleum ether–ether, 9:1) gave diphenyl disulfide (1.58 g) and **1** (1.07 g, 56.5%), identical with the authentic material.[5]

2-Phenylthio-2-(2′-pentynyl)-3-methylcyclopentanone (2). A solution of the ketone **1** (190 mg, 1.0 mmol) in anhydrous ether (5 mL) was added dropwise, under stirring and argon, to a chilled solution of lithium dimethylcuprate prepared from methyllithium (2.2 mL of 1.1 M ether solution, 2.4 mmol) and cuprous iodide (230 mg, 1.2 mmol) in dry ether (5 mL). After the addition, the bath was removed and the solvent evaporated almost to dryness under vacuum. The flask was again chilled, and the residue dissolved in dry glyme (5 mL). 2-Pentynyl bromide (500 mg, 3.4 mmol) was then added, and the mixture left stirring at room temperature for 2 h. After cooling in an ice bath, the reaction was quenched with aqueous saturated NH_4Cl and extracted with ether. The extract was washed successively with brine and 5% NaOH, dried (Na_2SO_4), and evaporated. Preparative TLC of the oily residue (eluting solvent: ether–petroleum ether, 1:9) yielded **2** (176 mg, 65%). An analytical sample was prepared by bulb-to-bulb distillation at 0.005 mm (120 °C bath temperature): IR (film) 1740 cm^{-1}; ^1H NMR 0.95–1.40 (m, 6 H, CH_3CH_2 and CH_3CH), 1.50–2.70 (m, 9 H), 7.10–7.60 ppm (m, 5 H, Ar). Anal. Calcd for $C_{17}H_{20}OS$: *m/e* 272.1230. Found: *m/e* 272.1242.

2-Phenylthio-3-methoxycarbonylmethylcyclopentanone (5). Dimethyl malonate (393 mg, 2.98 mmol) was added to a solution of sodium (65 mg, 2.82 mmol) in methanol (5 mL). To this mixture, under argon and chilled in an ice bath, was added a solution of **1** (500 mg, 2.63 mmol) in methanol (4 mL). After stirring for 1.5 h, the reaction was quenched with 10 N HCl (10 mL), the methanol distilled off, and the aqueous mixture refluxed for 24 h. The cold reaction mixture was extracted with ether, the ethereal extracts washed with 10% $NaHCO_3$, and the basic washings acidified with 10 N HCl. Extraction with ether, followed by washing of the organic phase with brine, drying (Na_2SO_4), and evaporation under vacuum, gave the crude acid. The residue was dissolved in dry methanol (6 mL) containing a drop of concentrated sulfuric acid. After standing overnight under argon at room temperature $NaHCO_3$ (200 mg) was added, and the methanol removed under vacuum. The oily residue was taken into ether, washed successively with 10% $NaHCO_3$ and brine, and dried (Na_2SO_4). Evaporation of the solvent left practically pure **5** (586 mg, 84%). An analytical sample was prepared by bulb-to-bulb distillation at 0.005 mm (125 °C bath temperature): IR (film) 1740 cm^{-1}; ^1H NMR 1.17–3.22 (m, 8 H), 3.47 (s, 3 H, CH_3O), 7.12–7.57 ppm (m, 5 H, Ar). Anal. Calcd for $C_{14}H_{16}O_3S$: *m/e* 264.0816. Found: *m/e* 264.0855. Semicarbazone: mp 185–187 °C (from MeOH). Anal. Calcd for $C_{15}H_{19}N_3O_3S$: *m/e* 321.1147. Found: *m/e* 321.1151.

2-Phenylthio-2-(2′-pentynyl)-3-methoxycarbonylmethylcyclopentanone (6). Sodium hydride (89 mg, 3.70 mmol) was added to a solution of **5** (890 mg, 3.37 mmol) in dry glyme (10 mL), kept in an ice bath with efficient stirring under an argon atmosphere. After the evolution of hydrogen subsided, 2-pentynyl bromide (660 mg, 4.50 mmol) was added, and the mixture left stirring for 2 h at 0 °C and 16 h at room temperature. The dark brown reaction mixture was then chilled in an ice bath, quenched with saturated aqueous NH_4Cl, and extracted with ether. The ether extracts were washed successively with brine, 5% NaOH, and brine, and dried (Na_2SO_4). Evaporation of the solvent under vacuum gave a dark oil, which was extracted with hot *n*-hexane. Evaporation of the hexane extract and preparative TLC (eluting solvent: ether–petroleum ether, 3:7) gave **6** (696 mg, 63%). An analytical sample was prepared by bulb-to-bulb distillation at 0.001 mm (125 °C bath temperature): IR (film) 2250, 1740 cm^{-1}; ^1H NMR 1.12, 1.17 (two overlapping t, 3 H, J = 7 Hz, CH_3CH_2), 1.75–3.15 (m, 11 H), 3.67, 3.70 (two overlapping s, 3 H, CH_3O), 7.32 ppm (s, 5 H, Ar). Anal. Calcd for $C_{19}H_{22}O_3S$: *m/e* 330.1284. Found: *m/e* 330.1261.

2-(2′-Pentynyl)-3-methoxycarbonylcyclopentanone (*dl*-Methyl Dehydrojasmonate, 4). A solution of **6** (162 mg, 0.49 mmol) in 10% aqueous THF (50 mL) was added to aluminum amalgam (1.75 g, 65 mmol) prepared according to Fieser and Fieser.[10] The mixture was stirred at room temperature until complete dissolution of the metal. Saturated NH_4Cl was added, the organic layer decanted, and the aluminum hydroxide slurry further extracted with ether. The combined extracts were washed with saturated NH_4Cl, dried (Na_2SO_4), and evaporated. Preparative TLC (eluting solvent: ether–petroleum ether, 2:8) afforded *dl*-methyl dehydrojasmonate (**4**, 100 mg, 92%). Bulb-to-bulb distillation at 0.01 mm (90 °C bath temperature) gave an analytical sample: IR (film) 1740 cm^{-1}; ^1H NMR 1.08 (t, 3 H, J = 7 Hz, CH_3CH_2), 1.68–2.97 (m, 12 H), 3.63 ppm (s, 3 H, CH_3O). Semicarbazone: mp 167–168 °C (from MeOH) (lit. 169–171,[9a] 168–169 °C[9c]).

Acknowledgment. The author wishes to thank Dr. Paul Baker of Centro de Pesquisas de Produtos Naturais, Rio de Janeiro, and Dr. Herbert Budzikiewicz of Institut für Organische Chemie, Köln, for the mass spectral data, and The Conselho de Desenvolvimento Científico e Tecnológico (CNPq) for financial assistance.

Registry No.—**1**, 34780-08-0; *cis*-**2**, 62067-30-5; *trans*-**2**, 62067-31-6; **4**, 29119-47-9; **5**, 62067-32-7; **5** semicarbazone, 62067-33-8; *cis*-**6**, 62067-34-9; *trans*-**6**, 62067-35-0; phenylsulfenyl chloride, 931-59-9; cyclopentanone, 120-92-3; 2-pentynyl bromide, 16400-32-1; dimethyl malonate, 108-59-8.

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A New Synthesis of *dl*-Muscone

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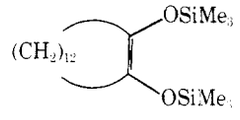
Received November 22, 1976

We wish to report a convenient synthesis of *dl*-muscone (**7**),[1] employing as its key step the $FeCl_3$ induced ring expansion of bis(trimethylsilyloxy)bicyclo[*n*.1.0]alkanes (e.g., **3** → **4**) which was found by us.[2] Our synthesis started from electrolysis of methyl hydrogen suberate producing dimethyl tetradecanedioate (**1**),[3] of which the silyl acyl condensation according to the Rühlmann procedure[4] afforded 1,2-bis(trimethylsilyloxy)cyclotetradecene (**2**). Cyclopropanation of **2**



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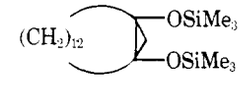

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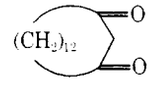

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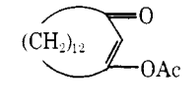

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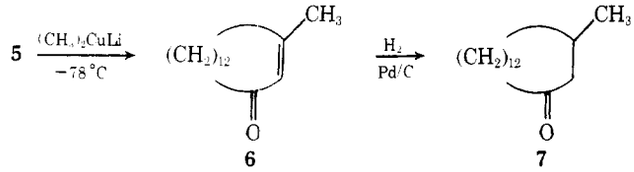
with diethylzinc and methylene diiodide[5] furnished a high yield of 1,14-bis(trimethylsilyloxy)bicyclo[12.1.0]pentadecane (**3**). But use of the conventional Simmons–Smith reagent, zinc–copper couple and methylene diiodide,[6] for cyclopropanation of **2** produced a complex mixture including **3**. A solution of **3** (10 mmol) in DMF (5 mL) was added to a stirring solution of anhydrous $FeCl_3$ (20 mmol) in DMF (25 mL) at room temperature, and the mixture was heated at 60 °C for 3 h. Acid workup and column chromatography on silica gel (benzene eluent) furnished cyclopentadecane-1,3-dione (**4**) as a keto–enol tautomer mixture in 88% yield. Conjugate addition[7] of lithium dimethylcopper to the enol acetate (**5**),



6 **7**

which was readily derived by the reaction of **4** with isopropenyl acetate in the presence of *p*-toluenesulfonic acid,[8] followed by the workup with aqueous NH_4Cl and the subsequent hydrogenation with Pd/C of the resulting 3-methyl-2-cyclopentadecenone (**6**), yielded *dl*-muscone (**7**) as a light yellow oil (78%). The overall yield of *dl*-muscone from the readily available methyl hydrogen suberate is about 23%.

Experimental Section

Materials. Anhydrous $FeCl_3$ was prepared by treating $FeCl_3 \cdot 6H_2O$ with thionyl chloride. Diethylzinc[9] was prepared by the reaction of zinc–copper couple with ethyl iodide and ethyl bromide. Dimethyl tetradecanedioate (**1**) was synthesized in 65% yield by electrolysis of methyl hydrogen suberate according to the procedure[3] employed for decarboxylative dimerization of methyl hydrogen sebacate producing dimethyl octadecanedioate.

1,2-Bis(trimethylsilyloxy)cyclotetradecene (2). Under a nitrogen atmosphere, a solution of 13.7 g (48 mmol) of dimethyl tetradecanedioate (**1**) in 50 mL of xylene was added dropwise into a stirring mixture of a fine dispersion of molten sodium (5.75 g, 0.25 g-atom) and 28.2 g (260 mmol) of trimethylchlorosilane in 200 mL of xylene at 40–50 °C over 3 h, and then the reaction mixture was heated at reflux for 5 h. After cooling, the reaction mixture was filtered to remove sodium chloride and concentrated in vacuo. The residue was subjected to Kugelrohr distillation to afford 11.2 g (63% yield) of 1,2-bis(trimethylsilyloxy)cyclotetradecene (**2**): bp 100–110 °C (1 mm); IR (neat) 1672, 1250, 1210, 850 cm^{-1}; NMR (CCl_4) δ 0.14 (s, 18 H), 1.20–1.70 (m, 20 H), 1.80–2.20 (m, 4 H).

Anal. Calcd for $C_{20}H_{42}O_2Si_2$: C, 64.80; H, 11.42. Found C, 64.92; H, 11.38.

Cyclopentadecane-1,3-dione (4). Under a nitrogen atmosphere, 8.61 g (32 mmol) of methylene diiodide was added dropwise to a stirring mixture of 3.95 g (32 mmol) of diethylzinc[9] and 5.92 g (16 mmol) of 1,2-bis(trimethylsilyloxy)cyclotetradecene (**2**) in 50 mL of benzene at room temperature and then the mixture was heated at reflux for 3 h. The reaction mixture was worked up with aqueous NH_4Cl and extracted with benzene. The benzene extract was dried over anhydrous Na_2SO_4 and concentrated in vacuo. Kugelrohr distillation of the residue afforded 4.98 g (81% yield) of 1,14-bis(trimethylsilyloxy)bicyclo[12.1.0]pentadecane (**3**): bp 110–120 °C (1 mm); IR (neat) 3070, 1250, 1220, 840 cm^{-1}; NMR (CCl_4) δ 0.12 (s, 18 H), 0.30–0.70 (m, 2 H), 0.70–2.00 (m, 24 H).

A solution of 3.84 g (10 mmol) of **3** in 5 mL of DMF was added to a stirring solution of 3.25 g (20 mmol) of anhydrous $FeCl_3$ in 25 mL of DMF at room temperature, and the mixture was heated at 60 °C for 3 h. The reaction mixture was poured into 10% aqueous HCl and extracted with chloroform. The chloroform extract was washed with 10% aqueous HCl and with water and dried over anhydrous Na_2SO_4. After the chloroform solution was evaporated, the residue was subjected to column chromatography on silica gel (benzene eluent) to furnish 2.10 g (88% yield) of cyclopentadecane-1,3-dione (**4**) as a keto–enol tautomer mixture. **4**: IR (neat) 1700, 1600 cm^{-1} (broad); NMR ($CDCl_3$ with Me_4Si) δ 1.44–2.00 (broad s, 20 H), 2.32–2.62 (m, 4 H), 3.68 (s) + 5.67 (s) + 15.72 (broad s) (2 H); mass spectrum M^+ *m/e* 238.

Anal. Calcd for $C_{15}H_{26}O_2$: C, 75.58; H, 11.00. Found: C, 75.71; H, 11.18.

***dl*-Muscone (7).** A mixture of 2.38 g (10 mmol) of cyclopentadecane-1,3-dione (**4**) and 5 mL of isopropenyl acetate was heated at reflux for 3 h with 100 mg of *p*-toluenesulfonic acid.[8] The reaction mixture was poured into ice-cold water and extracted with chloroform. The chloroform extract was washed with aqueous NaCl and dried over Na_2SO_4. After the chloroform solution was evaporated, the residue was subjected to column chromatography (silica gel–chloroform) to produce 2.75 g of 3-acetoxy-2-cyclopentadecenone (**5**): IR (neat) 1770, 1700, 1630 cm^{-1}; NMR ($CDCl_3$ with Me_4Si) δ 1.10–1.80 (broad s, 16 H), 2.25 (s, 3 H), 2.10–2.50 (broad m, 4 H), 5.85 (s, 1 H). Lithium dimethylcopper,[7] prepared from 415 mg (2.3 mmol) of cuprous iodide and 4.6 mmol of methyllithium in 10 mL of ether at 0 °C, was added dropwise to a stirring solution of 560 mg (2.0 mmol) of **5** in 10 mL of ether, which was kept at −78 °C. The reaction mixture was stirred for an additional 30 min at −78 °C, and worked up with aqueous NH_4Cl, followed by extraction with ether. The ether extract was concentrated and subjected to the conventional hydrogenation with Pd/C. *dl*-Muscone (**7**, 372 mg, 78% yield) was isolated by preparative TLC on silica gel (chloroform solvent) as a light yellow oil, which was identified by comparison of its spectral data with those of an authentic sample.[10]

Registry No.—**1**, 5024-21-5; **2**, 62078-79-9; **3**, 62078-80-2; **4**, 21173-90-0; **5**, 62078-81-3; **7**, 956-82-1; trimethylchlorosilane, 75-77-4.