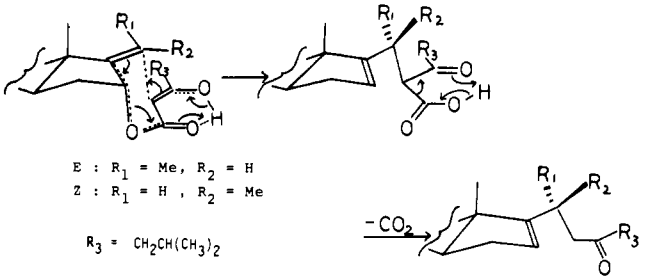
Scheme II




as an oil (**10**): $[\alpha]_D$ +38°; NMR 0.82 (s, 3 H, C-18 Me), 0.88 (d, $J$ = 7 Hz, 6 H, C-26,27 Me), 1.04 (s, 3 H, C-19 Me), 2.79 (t, $J$ = 2.5 Hz, 1 H, C-6$\alpha$ H), 3.33 (s, 3 H, C-6$\beta$ OMe), 5.31 (br s, 1 H, C-16 H); IR 1710 $cm^{-1}$. Catalytic hydrogenation of the 16-ene **10** with platinum black in ethyl acetate from the $\alpha$ side[12] fixes the C-17$\alpha$ H stereochemistry and yielded the crystalline dihydro compound **11** (96%): mp 72 °C; $[\alpha]_D$ +38°; NMR 0.75 (s, 3 H, C-18 Me), 0.90 (d, $J$ = 7 Hz, 6 H, C-26,27 Me), 1.01 (s, 3 H, C-19 Me), 2.76 (t, $J$ = 2.5 Hz, 1 H, C-6$\alpha$ H), 3.28 (s, 3 H, C-6$\beta$ OMe); IR 1710 $cm^{-1}$. Hydrolysis of the cyclo protecting group with dilute sulfuric acid yielded the known 23-ketocholesterol[13] **12** (84%): mp 145–146 °C; $[\alpha]_D$ −43°; NMR 0.72 (s, 3 H, C-18 Me), 0.92 (d, $J$ = 6 Hz, 6 H, C-26,27 Me), 1.02 (s, 3 H, C-19 Me), 3.50 (br m, C-3$\alpha$ H), 5.39 (br s, 1 H, C-6 H); IR 3350, 1710 $cm^{-1}$. Wolff-Kishner reduction of **12** gave cholesterol (**1**) in 97% yield, that was identical in all respects ($^1H$ NMR, $^{13}C$ NMR MS, IR, and GLC retention time) with an authentic sample.

20-Isocholesterol (**2**) was synthesized in a similar way from the isomeric $Z$-allylic acetoacetate (**8**). Carroll reaction of the $Z$-olefinic ester **8** (oil) [$[\alpha]_D$ −30°; NMR 0.80 (s, 3 H, C-18 Me), 0.89 (d, $J$ = 7 Hz, 6 H, ester dimethyl), 1.03 (s, 3 H, C-19 Me), 1.59 (d, $J$ = 8 Hz, 3 H, C-21 Me), 5.48 (d, q, $J$ = 2 and 8 Hz, 1 H, C-20 H), 5.83 (br s, 1 H, C-16 $\beta$H)] in refluxing xylenes for 4 h yielded the rearranged product **13** (62% yield, oil) [$[\alpha]_D$ +32°; NMR 0.87 (s, 3 H, C-18 Me), 0.92 (d, $J$ = 6 Hz, 6 H, C-26,27 Me), 1.07 (s, 3 H, C-19 Me), 1.07 (d, $J$ = 6 Hz, 3 H, C-21 Me), 5.41 (br s, 1 H, C-16 H); IR 3040, 1710 $cm^{-1}$] with 33% recovery of **8**[14] (see Scheme II). After catalytic hydrogenation of **13**, the dihydro compound **14** (an oil) [$[\alpha]_D$ +38°; NMR 0.73 (s, 3 H, C-18 Me), 0.90 (d, $J$ = 6 Hz, 6 H, C-26,27 Me), 1.02 (s, 3 H, C-19 Me); IR 3030, 1710 $cm^{-1}$] was converted into 23-keto-20-isocholesterol (**15**, 82%) [mp 143–145 °C; $[\alpha]_D$ −45°; NMR 0.71 (s, 3 H, C-18 Me), 0.75 (d, $J$ = 7 Hz, 3H, C-21 Me), 0.91 (d, $J$ = 6 Hz, 6 H, C-26,27 Me), 1.00 (s, 3 H, C-19 Me), 3.50 (br m, 1 H, C-3 H), 5.39 (m, 1 H, C-6 H); IR 3250, 1710, 1080 $cm^{-1}$] from **14** by treatment with dilute sulfuric acid. Wolff-Kishner reduction of **15** gave 20-isocholesterol (**2**) in quantitative yield [mp 149–151 °C; $[\alpha]_D$ −55° (lit.[15] mp 152–154 °C; $[\alpha]_D$ −42 °); NMR 0.69 (s, 3 H, C-18 Me), 0.82 (d, $J$ = 6 Hz, 3 H, C-21 Me), 0.88 (d, $J$ = 6 Hz, 6 H, C-26,27 Me), 1.02 (s, 3 H, C-19 Me), 3.49 (br m, 1 H, C-3 H), 5.35 (m, 1 H, C-6H)], which showed a depression of the melting point on admixture with authentic cholesterol. The retention time of **2** on GLC is shorter than that of cholesterol (**1**). Thus, these results provide a useful method for stereocontrolled introduction of the desired C-20 stereochemistry in steroid side-chain synthesis via Claisen rearrangement and related reactions.

**Acknowledgment.** This work was supported by the National Institute of Health, Grant No. GM 27058-01.

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## An Efficient and Versatile Generation of *o*-Xylylenes by Fluoride Anion Induced 1,4 Elimination of *o*-($\alpha$-Trimethylsilylalkyl)benzyltrimethylammonium Halides

*Sir:*

Cycloaddition of olefins to *o*-xylylenes provides a convenient synthetic method for the preparation of tetrahydronaphthalene derivatives. The *o*-xylylene moiety is generated in situ by the metal induced[1] or thermal[2] 1,4 elimination reactions of the corresponding *o*-xylylene derivatives such as *o*-xylylene dihalides and *o*-methylbenzyltrimethylammonium hydroxides. Intramolecular cycloaddition of *o*-xylylenes generated by electrocyclic ring opening of substituted benzocyclobutenes was reported recently,[3] which constitutes a new approach to the synthesis of polycyclic ring systems including natural products.

Herein we report an efficient and versatile method for the generation of *o*-xylylene intermediates (**2**) by fluoride anion induced 1,4 elimination[4] of *o*-($\alpha$-trimethylsilylalkyl)benzyltrimethylammonium halides (**1**). A simple and mild gen-

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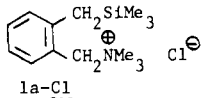
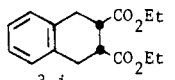
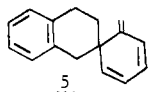
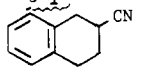
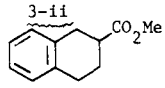
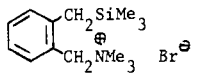
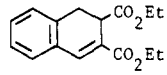
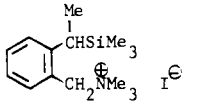
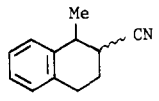
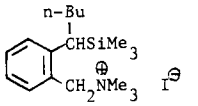
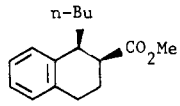
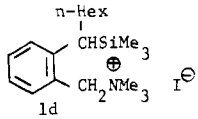
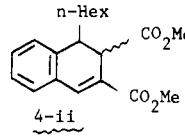
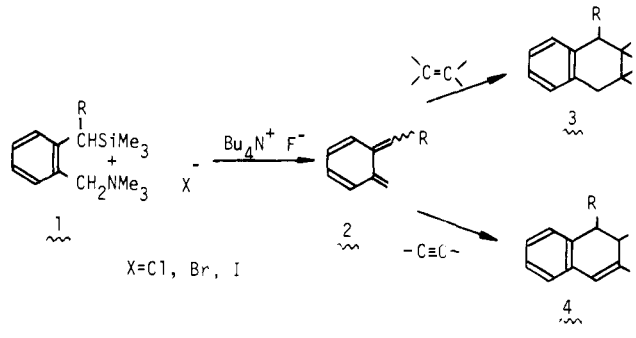
**Table I.** Cycloadditions of *o*-Xylylenes with Olefins and Acetylenes

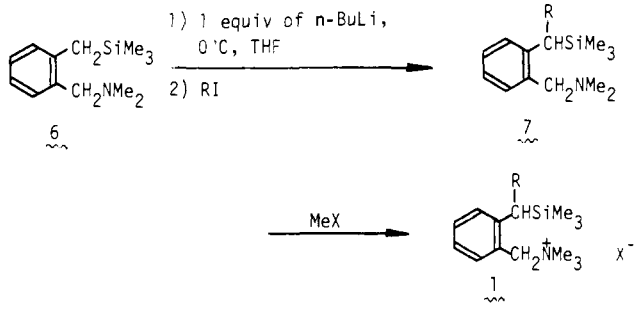
| 1 | Olefins or Acetylenes (Molar equiv) | Products (Isolated %)[a] |
|---|---|---|
|  1a-Cl | cis-$EtO_2CCH{=}CHCO_2Et$[b] (3) |  3-i (46)[d]  5 (38)[e] |
| | $CH_2{=}CHCN$[b] (3) |  3-ii (82)[f] |
| | $CH_2{=}CHCO_2Me$[b] (3) |  3-iii (90)[f] |
|  1a-Br | $EtO_2CC{\equiv}CCO_2Et$[c] (1.3) |  4-i (76)[f] |
|  1b | $CH_2{=}CHCN$[c] (3) |  3-iv (80)[g, h, i] |
|  1c | $CH_2{=}CHCO_2Me$[c] (1.1) |  3-v (88)[g] |
|  1d | $MeO_2CC{\equiv}CCO_2Me$[c] (1.1) |  4-ii (79)[h, j, k] |

[a] Yields are based on **1** used and not optimized. [b] $CH_2Cl_2$ solvent. [c] $CH_3CN$ solvent. [d] Reference 14. [e] Reference 2. [f] Reference 1e. [g] Contaminated with another regioadduct (<20%). [h] A cis and trans mixture. [i] Reference 15. [j] Contaminated with olefinic isomerization products (<10%). [k] Reference 16.




eration of *o*-xylylene intermediates followed by their intermolecular trappings with electron-deficient olefins or acetylenes is illustrated as follows. To a stirring solution of 172 mg (0.63 mmol) of *o*-(trimethylsilylmethyl)benzyltrimethylammonium chloride (**1a**-Cl, R = H)[5] and 0.16 g (1.9 mmol) of methyl acrylate in 5 mL of methylene chloride, a solution of 215 mg (0.82 mmol) of tetrabutylammonium fluoride in 5 mL of methylene chloride was added dropwise at room temperature over 0.5 h. After the mixture was stirred for 1 h at room temperature, ether was added and the mixture was washed with water and dried over anhydrous $Na_2SO_4$. The ether solution was evaporated in vacuo and chromatographed on silica gel to produce 1,2,3,4-tetrahydro-2-carbomethoxynaphthalene (**3**-iii)[1e] [TLC (1:1 chloroform–benzene), $R_f$ = 0.52] in a 90% yield. When **1a**-Cl was reacted with tetrabutylammonium fluoride in the absence of methyl acrylate, spiro[di-*o*-xylylene] (**5**)[6] was produced in a 64% isolated yield. Use of *o*-(trimethylsilylmethyl)benzyl chloride instead of **1a** in the above procedure furnished a similar result but in a slightly decreased yield.

The most attractive feature of the present method for generation of *o*-xylylene intermediates is that α-substituted *o*-xylylenes are generated in situ from *o*-(α-trimethylsilylalkyl)benzyltrimethylammonium halides (**1b–e**), which are

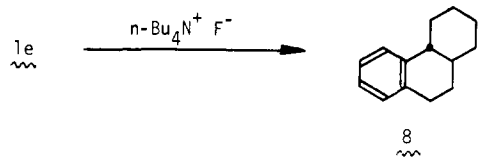



| | Overall Yield (%) |
|---|---|
| b: R=$CH_3$, X=I | 56 |
| c: R=$CH_2(CH_2)_2CH_3$, X=I | 75 |
| d: R=$CH_2(CH_2)_4CH_3$, X=I | 72 |
| e: R=$CH_2(CH_2)_3CH{=}CH_2$, X=I | 75 |

readily derived via alkylation of the silyl-stabilized anion of *o*-(trimethylsilylmethyl)benzyldimethylamine (**6**)[7] and the subsequent quaternalization with methyl halides.

Reaction of *o*-(α-trimethylsilylpentyl)benzyltrimethylammonium iodide (**1c**) with methyl acrylate was similarly caused by tetrabutylammonium fluoride to afford 1,2,3,4-tetrahydro-*cis*-1-butyl-2-carbomethoxynaphthalene (**3**-v)[10] as a major product in 88% yield. Some examples of cycloadditions of *o*-xylylene intermediates with olefins and acetylenes are summarized in Table I.

The present method for generation of *o*-xylylenes and their trappings with olefins can be extended to intramolecular cycloaddition of *o*-xylylenes leading to polycycles. When a solution of 145 mg (0.55 mmol) of tetrabutylammonium fluoride in 10 mL of acetonitrile was added dropwise over 1 h to a refluxing solution of 225 mg (0.44 mmol) of *o*-(1-trimethylsilylhept-6-enyl)benzyltrimethylammonium iodide (**1e**)[11] in 5 mL of acetonitrile, *trans*-octahydrophenanthrene (**8**)[12] was




produced in 70% yield together with an 8% yield of the corresponding spiro[di-*o*-xylylene] derivative (**9**).[13] We plan to report further studies on this reaction and its application to the synthesis of steroidal structure in the near future.

**Acknowledgment.** We thank Dr. T. Suzuki of Kyoto University for the $^{13}C$ NMR measurement. We are grateful to Professor K. P. C. Vollhardt for providing us with $^{13}C$ NMR and 360-MHz NMR spectra of *trans*-octahydrophenanthrene.

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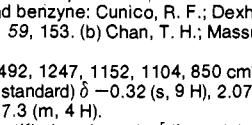
(5) **1a**-Cl: IR (KBr disk) 1492, 1247, 1152, 1104, 850 $cm^{-1}$; NMR ($CD_3CN$ with $Me_4Si$ as an external standard) δ −0.32 (s, 9 H), 2.07 (s, 2 H), 2.85 (s, 9 H), 4.36 (br s, 2 H), 6.6–7.3 (m, 4 H).
(6) Compound **5** was identified as the spiro[di-*o*-xylylene] **5** by comparison of its NMR and IR spectra with those reported by Errede.[2]




(7) **6** was prepared in 85% overall yield via Sommelet rearrangement of benzyltrimethylammonium iodide[8] and silylation[9] of the resulting *o*-methylbenzyldimethylamine. **6**: IR (neat) 1249, 840 $cm^{-1}$; NMR ($CCl_4$ with $Me_4Si$ as an external standard) δ 0.00 (s, 9 H), 2.06 (s, 6 H), 2.12 (s, 2 H), 3.13 (s, 2 H), 6.6–7.1 (m, 4 H).
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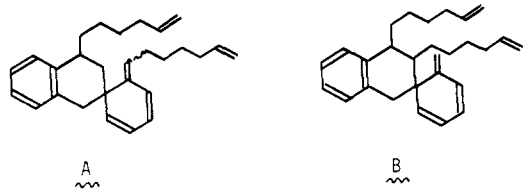
(10) **3**-v: IR (neat) 1738, 1150, 1200, 768 $cm^{-1}$; NMR (100 MHz) ($CCl_4$ with $Me_4Si$) δ 0.8–1.2 (m, 3 H), 1.2–1.8 (m, 6 H), 2.1 (quasi q, 2 H), 2.6–3.3 (m, 4 H), 3.77 (s, 3 H), 7.10 (s, 4 H). Cis configuration of **3**-v was established by decoupling technique which revealed a sharp doublet ($J_{H_1-H_2}$ = 4.5 Hz) at δ 3.15 ascribed to a hydrogen on $C_1$.
(11) **1e**: IR (KBr disk) 1641, 1246, 990, 910, 842 $cm^{-1}$; NMR ($CD_3CN$ with $Me_4Si$ as an external standard) δ −0.36 (s, 9 H), 1.2–2.1 (m, 8 H), 2.30 (t, 1 H), 2.76 (s, 9 H), 4.18 (dd, 2 H), 4.3–5.8 (m, 3 H), 6.6–7.3 (m, 4 H).
(12) **8**: $^{13}C$ NMR ($CDCl_3$ with $Me_4Si$) δ 26.31, 27.00, 29.92, 30.68, 30.97, 34.40, 40.62, 43.81, 125.36 (2 C), 125.43, 128.94, 137.00, 140.54 ppm. Trans stereochemistry of **8** was convincingly confirmed by comparison of its $^{13}C$ NMR spectrum with that of *trans*-octahydrophenanthrene, which was provided by Professor Vollhardt.
(13) Structure A was assigned to compound **9** on the basis of its IR and NMR spectra: IR (neat) 1640, 995, 909, 755 $cm^{-1}$; NMR ($CDCl_3$ with $Me_4Si$) δ 1.0–3.0 (m, 21 H), 4.6–6.5 (m, 11 H), 6.9–7.2 (m, 4 H). A possibility of the regioisomeric structure (B) for **9** was excluded by lack of IR absorption band at 890 $cm^{-1}$ characteric of the *exo*-methylene structure.




(14) **3**-i: NMR (100 MHz) ($CDCl_3$ with $Me_4Si$) δ 1.28 (t, 6 H), 3.01 (br t, 2 H), 3.17 (br d, 4 H), 4.21 (t, 4 H), 7.05 (s, 4 H).
(15) Dehydrogenation of **3**-iv by palladium on charcoal gave 1-methyl-2-cyanonaphthalene. **3**-iv: NMR ($CDCl_3$ with $Me_4Si$) δ 1.45 (d, 3 H), 1.8–2.3 (m, 2 H), 2.6–3.3 (m, 4 H), 7.05 (br s, 4 H).
(16) **4**-ii: NMR (100 MHz) ($CDCl_3$ with $Me_4Si$) δ 0.7–1.1 (m, 3 H), 1.1–1.2 (m, 10 H), 2.9–3.7 (m, 2 H), 3.52–3.82 (4s, 6 H), 7.0–7.4 (m, 4 H), 7.56 and 7.61 (2s, 1 H, olefinic proton).

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## Palladium(II) Chloride Catalyzed Cope Rearrangements of Acyclic 1,5-Dienes[1]

*Sir:*

The Cope rearrangement of 1,5-dienes typically requires elevated temperatures.[2] Catalytic methods for effecting this carbon–carbon-bond-forming transformation enhance its synthetic utility, and in recent years impressive accomplishments have been recorded in catalyzing Cope rearrangements of functionalized 1,5-dienes.[3] The development of more general methods for catalyzing the rearrangement of simple 1,5-dienes remains, however, a challenging problem.[4] In 1966 Jonassen and co-workers[7a] reported that treatment of excess *cis,trans*-1,5-cyclodecadiene at room temperature with bis(benzonitrile)palladium(II) chloride gave the crystalline palladium(II) dichloride complex of *cis*-1,2-divinylcyclohexane in 82% yield.[7,8] The similar rearrangement of *cis*-1,2-divinylcyclobutanes to give palladium(II) dichloride complexes of 1,5-cyclooctadienes has been extensively studied by Heimbach and co-workers.[9] These studies,[7-9] while clearly demonstrating that stoichiometric amounts of palladium(II) chloride can promote the Cope rearrangement of strained cyclic 1,5-dienes, leave unanswered questions of the generality or potential catalytic nature of this reaction. In this communication we report for the first time that palladium(II) promoted Cope rearrangements can be conducted in a *catalytic* fashion to produce the rearranged diene, rather than the diene–palladium(II) dichloride complex. We moreover report *that Cope rearrangements of many unstrained, conformationally flexible, acyclic 1,5-dienes are dramatically catalyzed by palladium(II) chloride salts and occur readily at room temperature.*

Treatment of 2-methyl-3-phenyl-1,5-hexadiene (**1**)[10] with 0.06 equiv of $PdCl_2(PhCN)_2$ in tetrahydrofuran (THF) at room temperature for 24 h produced dienes **2**[11,12a] and **3**[11] in a 93:7 ratio (87% yield after bulb-to-bulb distillation). In contrast, thermal Cope rearrangement of diene **1** required elevated temperatures (half-life, 13 h; 177 °C; $C_6D_6$ solvent) and proceeded less stereoselectively, to yield **2** and **3** in a kinetically controlled[13] 3:1 ratio. Although the $^1H$ NMR, IR, and mass spectra for stereoisomers **2** and **3** are nearly identical, stereochemical assignments follow unambiguously from $^{13}C$

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