tonitrile, reflux 42 h) afforded the required azalactone 7[9] in 73% yield after chromatography on silica gel. Subjection of 7 to the usual silylation conditions[1] (1.2 equiv of LDA, 1.2 equiv of t-BuMe$_2$SiCl, THF, −70 °C) produced the expected rearrangement product 8 in 93% yield. Hydrolysis of the silyl ester (2 equiv of HF, 3 N in acetonitrile, 0 °C) gave a single carboxylic acid 9[9] in 92% yield (86% based on 7). None of the trans-3,4-disubstituted isomer of 9 could be detected.[10] Completion of the synthesis by means of the Wolff rearrangement proved uneventful. Treatment of 9 sequentially with sodium hydride and oxalyl chloride (1.0 equiv of each, ether, 0 °C) followed by the addition of this reaction mixture to excess ethereal diazomethane (0 °C, 1 h)[11] gave the diazo ketone 10 as an oil in 83% yield. Without purification, 10 was treated with silver benzoate and triethylamine in methanol[12] (0 °C, 6 h) to afford N-benzoylmeroquinene methyl ester 12 in 77% yield. Ester 12 obtained in this manner was identical in all respects with an authentic sample[13] of 12 kindly provided by Dr. M. R. Uskokovič of Hoffmann-La Roche, Inc. The overall yield of 12 obtained by this route was 25% based on 3.

The methodology reported herein represents a new strategy for heterocycle synthesis. We are currently investigating the application of this approach in the synthesis of the pyrrolidine α-kainic acid and other heterocycles.

Acknowledgment. We appreciate the financial and material support provided by the National Institutes of Health (Grant No. GM2866301). High-field (360 MHz) ^{1}H and ^{13}C NMR spectra were obtained on a spectrometer purchased with funds provided, in part, by the National Science Foundation (Grant No. CHE-80-24328). Mass spectra were obtained through the National Science Foundation Regional Mass Spectroscopy Center at the University of Nebraska (Grant CHE-82-11164).

Registry No. 3, 35340-63-7; (±)-4, 92127-12-3; (±)-5, 92127-13-4; 6, 92127-14-5; 7, 92127-15-6; (±)-8, 92127-16-7; (±)-9, 92127-17-8; (±)-9 (R = Cl), 92127-20-3; (±)10, 92127-18-9; (±)-11, 92127-19-0; (±)-12, 26013-17-2; quinine, 130-95-0.

(9) Lactone 7: IR (NaCl, neat) 3055, 3025, 2930, 2870, 1748, 1635, 1572, 1490, 1443, 1428, 1395, 1375, 1325, 1280, 1240, 1195, 1157, 1135, 1080, 1055, 1031, 1024, 790, 718, 695, 655 cm^{-1}; ^{1}H NMR (90 MHz, $CDCl_3$, 25 °C) δ 7.33 (br s, 5 H), 5.90–5.43 (m, 2 H), 4.67 (m, 2 H), 4.11 (br d, 2 H, J = 7.5 Hz), 3.26 (br t, 2 H, J = 5.0 Hz), 2.50–2.27 (m, 2 H), 2.13–1.83 (m, 2 H); exact mass calcd for $C_{15}H_{17}O_3N$ 259.1209, found 259.1200; m/e (relative intensity) 259 ($M^{+\cdot}$, 0.21), 216 (1.74), 215 (11.27), 214 (2.10), 187 (3.81), 186 (3.54), 159 (1.00), 158 (2.01), 105 (100.0), 82 (1.28), 78 (2.38), 77 (25.48), 74 (6.32), 69 (1.22), 59 (9.42), 55 (1.27), 54 (1.23), 51 (3.80). Anal. Calcd for $C_{15}H_{17}O_3N$: C, 69.48; H, 6.61; N, 5.41. Found: C, 69.36, H, 6.79; N, 5.49. Carboxylic acid 9: mp 48–9 °C; IR (NaCl, neat) 3650–2850, 3040, 3025, 2995, 2925, 2855, 1720, 1595, 1575, 1465, 1450, 1305, 1255, 1180, 1018, 915, 785, 730 cm^{-1}; ^{1}H NMR (360 MHz, $CDCl_3$, 60 °C) δ 7.39 (br s, 5 H), 5.85 (ddd, 1 H, J = 17.21, 10.38, 7.65 Hz), 5.25–5.07 (m, 2 H), 4.44–3.73 (m, 2 H), 3.41 (dd, 1 H, J = 13.12, 2.56 Hz), 3.26 (br t, 1 H, J = 8.81 Hz), 2.90–2.75 (m, 2 H), 2.04–1.89 (m, 1 H), 1.88–1.74 (m, 1 H); ^{13}C NMR (50.31 MHz, $CDCl_3$, 50 °C) δ 177.11, 171.11, 135.86, 134.59, 129.66, 128.41, 126.96, 118.04, 44.46, 40.90, 24.33; exact mass calcd for $C_{15}H_{17}O_3N$ 259.1209, found 259.1216; m/e (relative intensity) 259 ($M^{+\cdot}$, 2.69), 218 (2.39), 154 (2.73), 122 (2.93), 106 (4.11), 105 (60.22), 77 (17.87), 51 (2.89), 40 (7.89). Anal. Calcd for $C_{15}H_{17}O_3N$: C, 69.48; H, 6.61; N, 5.41. Found: C, 69.40; H, 6.74; N, 5.27.

(10) Product stereochemical integrity was confirmed by HPLC and ^{1}H NMR (360 MHz) analysis of the methyl ester 11 (CH_2N_2, ether, 0 °C) derived from the crude product 9. A single methyl ester peak was observed at δ 3.63, and HPLC analysis showed no other isomers. The presumed stereochemical assignment of 9 was confirmed by its successful conversion to 2.

(11) Clark, R. D. *Synth. Commun.* **1979**, *9*, 325.

(12) (a) Newman, M. S.; Beal, P. F., III *J. Am. Chem. Soc.* **1950**, *72*, 5163. (b) Hudlicky, T.; Sheth, J. P. *Tetrahedron Lett.* **1979**, 2667.

(13) The ^{1}H NMR (90 and 360 MHz), ^{13}C NMR (50.31 MHz), IR, and MS spectra and HPLC and TLC (three solvent systems) chromatograms were compared.

(14) Maude Hammond Fling Fellow, 1982–84, from the University of Nebraska.

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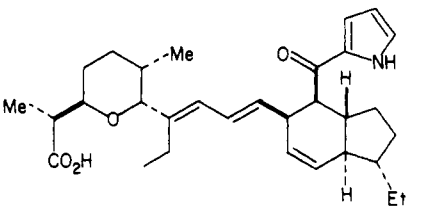
Polysubstituted Dihydropyrans via the Enolate Claisen Rearrangement. A Stereocontrolled Route to *C*-Pyranosides

Summary: A new method for the stereoselective synthesis of dihydropyrans of a variety of substitution patterns is described, involving [3,3]-sigmatropic reorganizations of 6-alkenyl-4-oxapyran-2-ones of general structure 1 or 3 to the product dihydropyrans (2 or 4, respectively).

Sir: The polysubstituted hydropyran nucleus is a structural subunit in a number of recently isolated natural products of chemical and biological interest. Selected examples include the ionophore antibiotic X-14547A,[2] the



X-14547A

antifungal agent ambruticin,[3] and the structurally daunting marine natural product palytoxin.[4] In each of these there exist one or more hydropyran units, formally classified as *C*-pyranosides, wherein the C(2) and C(6) positions flanking the ring oxygen have carbon side-chain substituents. We report herein a stereocontrolled route to the *C*-pyranoside system.[5]

(1) (a) Research Fellow of the Alfred P. Sloan Foundation. (b) Recipient of an NSF Presidential Young Investigator Award.

(2) (a) Westley, J. W.; Evans, R. H., Jr.; Liu, C.-M., Hermann, T.; Blount, J. F. *J. Am. Chem. Soc.* **1978**, *100*, 6786. (b) Liu, C.-M.; Hermann, T. E.; Liu, M.; Bull, D. N.; Palleroni, N. J.; Prosser, B. L. T.; Westley, J. W.; Miller, P. A. *J. Antibiot.* **1979**, *32*, 95. (c) Westley, J. W.; Evans, R. H., Jr.; Sello, L. H.; Troupe, N.; Liu, C.-M.; Blount, J. F. *Ibid.* **1979**, *32*, 100. (d) Westley, J. W.; Liu, C.-M. U.S. Patent 4 100 171, **1978**. (e) Nicolaou, K. C.; Magolda, R. L. *J. Org. Chem.* **1981**, *46*, 1506. (f) Roush, W. R.; Myers, A. G. *J. Org. Chem.* **1981**, *46*, 1509. (g) Edwards, M. P.; Ley, S. V.; Lister, S. G. *Tetrahedron Lett.* **1981**, *22*, 361. (h) Nicolaou, K. C.; Papahatjis, D. P.; Claremon, D. A.; Dolle, R. E., III. *J. Am. Chem. Soc.* **1981**, *103*, 6967. (i) Nicolaou, K. C.; Claremon, D. A.; Papahatjis, D. P.; Magolda, R. L. *Ibid.* **1981**, *103*, 6969. (j) Ho, P. *Can. J. Chem.* **1982**, *60*, 90. (k) Roush, W. R.; Peseckis, S. M. *Tetrahedron Lett.* **1982**, *23*, 4879. (l) Edwards, M. P.; Ley, S. V.; Lister, S. G.; Palmer, B. D. *J. Chem. Soc., Chem. Commun.* **1983**, 630.

(3) (a) Barnes, N. J.; Davidson, A. H.; Hughes, L. R.; Procter, G.; Rajcoomar, V. *Tetrahedron Lett.* **1981**, *22*, 1751. (b) Conner, D. T.; Greenough, R. C.; von Strandtmann, M. *J. Org. Chem.* **1977**, *42*, 3664. (c) Ringel, S. M.; Greenough, R. C.; Roemer, S.; Connor, D.; Gutt, A. L.; Blair, B.; Kanter, G.; von Strandtmann, M. *J. Antibiot.* **1977**, *30*, 371. (d) Connor, D. T.; von Strandtmann, M. *J. Org. Chem.* **1978**, *43*, 4606.

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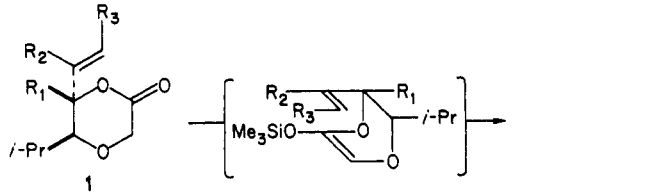
0022-3263/84/1949-4320$01.50/0

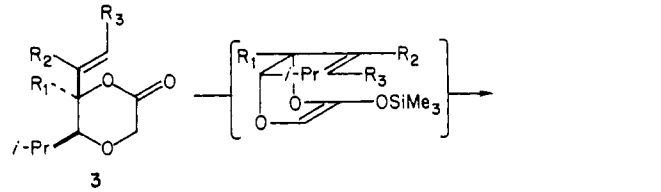
Table I[9]

entry	oxapyranone substrates	dihydropyran products	yield, %
1	**1a**, $R_1 = R_2 = R_3 = H$	**2a**, $R_1 = R_2 = R_3 = H$	67
2	**1b**, $R_1 = R_3 = H$; $R_2 = Me$	**2b**, $R_1 = R_3 = H$; $R_2 = Me$	75
3	**1c**, $R_1 = R_2 = H$; $R_3 = SiMe_3$	**2c**, $R_1 = R_2 = H$; $R_3 = SiMe_3$	52
4	**1d**, $R_1 = R_2 = H$; $R_3 = Me$	**2d**, $R_1 = R_2 = H$; $R_3 = Me$	70
5	**1e**, $R_1 = Me$; $R_2 = R_3 = H$	**2e**, $R_1 = Me$; $R_2 = R_3 = H$	90
6	**1f**, $R_1 = R_2 = Me$; $R_3 = H$	**2f**, $R_1 = R_2 = Me$; $R_3 = H$	80
7	**3a**, $R_1 = R_2 = R_3 = H$	**4a**, $R_1 = R_2 = R_3 = H$	69
8	**3b**, $R_1 = R_3 = H$; $R_2 = Me$	**4b**, $R_1 = R_3 = H$; $R_2 = Me$	78
9	**3c**, $R_1 = R_2 = H$; $R_3 = SiMe_3$	**4c**, $R_1 = R_2 = H$; $R_3 = SiMe_3$	61
10	**3d**, $R_1 = R_2 = H$; $R_3 = Me$	**4d**, $R_1 = R_2 = H$; $R_3 = Me$	81
11	**3e**, $R_1 = Me$; $R_2 = R_3 = H$	**4e**, $R_1 = Me$; $R_2 = R_3 = H$	91

In the interest of flexibility and generality, we chose to develop a method which did not depend upon the modification of a preexisting carbohydrate framework.[6] There is illustrated in eq 1 and 2 the key reaction by which a



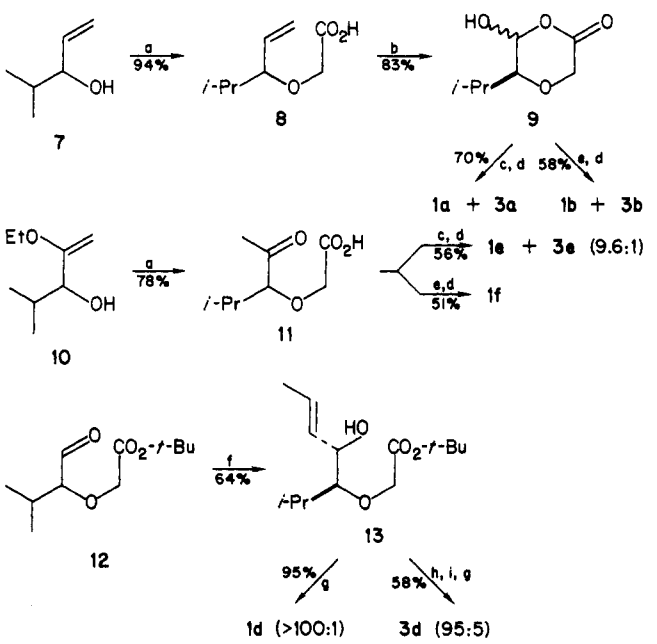
(1)



(2)

generalized 6-alkenyl-4-oxapyran-2-one (**1** or **3**) is transformed into a substituted dihydropyran (**2** or **4**, respectively). Following the precedents of Ireland[7] and Danishefsky,[8] the plan involved the conversion of the pyranone substrates **1** and **3** to the corresponding trimethylsilyl ketene acetals. Claisen[3,3]-sigmatropic rearrangement via the bracketed intermediates would then yield, after hydrolysis and esterification, the product dihydropyrans of general structures **2** and **4**.

Scheme I[9,a]



[a] (a) NaH, THF, 0–25 °C; $BrCH_2CO_2Na$, reflux; H_3O^+ quench. (b) O_3, CH_2Cl_2, −78 °C; Me_2S. (c) $H_2C{=}CHMgBr$, THF, −78 °C; H_3O^+ quench. (d) camphorsulfonic acid, PhH, reflux. (e) $H_2C{=}C(CH_3)MgBr$, THF, −78 °C; H_3O^+ quench. (f) *trans*-$LiCu(HC{=}CHCH_3)_2$, Et_2O, −78 °C. (g) F_3CCO_2H, CH_2Cl_2, 25 °C. (h) Jones reagent, 0 °C. (i) $Zn(BH_4)_2$, Et_2O, 0 °C.

The results of such rearrangements for substrates **1a–f** and **3a–e** are summarized in Table I.[9] The isolated yields of purified products range from good to excellent (52 → 91%). Typically, the substrate lactones were deprotonated at −78 °C in tetrahydrofuran (THF) with lithium diisopropylamide (LDA) and the resultant enolates were trapped with chlorotrimethylsilane (Me_3SiCl) in Et_3N (−78 °C → 25 °C). The volatiles were removed in vacuo and were replaced with dry toluene. After the resulting solutions had been heated for 3–4 h at 105–110 °C (bath temperature), the product trimethylsilyl esters were hydrolyzed with 5% aqueous HCl in Et_2O, and the crude acids were esterified with ethereal CH_2N_2. The product dihydropyrans **2a–f** and **4a–e** were produced in the cited yields

(5) For selected recent efforts directs at *C*-pyranoside synthesis, see: (a) Keck, G. F.; Enholm, E. J.; Kachensky, D. F. *Tetrahedron Lett.* **1984**, *25*, 1867. (b) Hosomi, A.; Sakata, Y.; Sakurai, H. *Ibid.* **1984**, *25*, 2383. (c) Wilcox, C. S.; Long, G. W.; Suh, H. *Ibid.* **1984**, *25*, 395. (d) Williams, R. M.; Stewart, A. O. *Ibid.* **1983**, *24*, 2715. (e) Kozikowski, A. P.; Sorgi, K. L.; Wang, B. C.; Xu, Z.-b. *Ibid.* **1983**, *24*, 1563. (f) Lancelin, J.-M.; Zollo, P. H. A.; Sinaÿ, P. *Ibid.* **1983**, *24*, 4833. (g) Lewis, M. D.; Cha, J. K.; Kishi, Y. *J. Am. Chem. Soc.* **1982**, *104*, 4976. (h) Danishefsky, S.; Kerwin, J. F., Jr.; Kobayashi, S. *Ibid.* **1982**, *104*, 358. (h) Dawe, R. D.; Fraser-Reid, B. *J. Org. Chem.* **1984**, *49*, 522. (j) Tulshian, D. B.; Fraser-Reid, R. *Ibid.* **1984**, *49*, 518. (k) Dunkerton, L. V.; Serino, A. J. *Ibid.* **1982**, *47*, 2812.

(6) Most of the *C*-pyranoside syntheses cited in ref 5 involve substitution at the anomeric site of carbohydrates (or derivative thereof) by carbon nucleophiles. Such an approach is attended by obvious advantages and disadvantages. The method described herein offers a complimentary solution.

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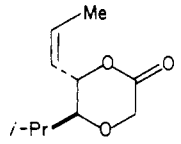
(8) Danishefsky, S.; Funk, R. L.; Kerwin, J. F., Jr. *J. Am. Chem. Soc.* **1980**, *102*, 6889.

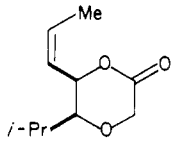
(9) In the table, scheme, and equations, all chiral substances were produced as racemates; a single enantiomer is shown for simplicity. All structural assignments are supported by IR, 400-MHz ^1H NMR, ^{13}C NMR, and mass spectrometric and elemental analyses. Yields cited are for chromatographically and spectroscopically pure substances.

after chromatographic purification on silica gel.

It should be noted that the projection of the isopropyl residue into the presumed pericyclic transition state leading from **3** to **4** seems to offer no impediment to rearrangement. However, cis-terminal substitution on the alkenyl residue prevents rearrangement. For example, substrates **5** and **6** are recovered unchanged after subjection to the rearrangement conditions and subsequent hydrolysis.



5

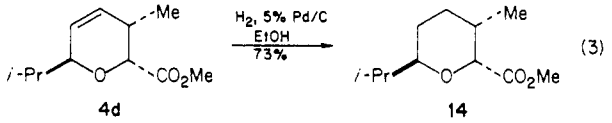


6

The preparations of the substrate oxapyranones **1a–f** and **3a–e** were accomplished by straightforward vinylmetallic 1,2-additions to carbonyls, as exemplified in Scheme I. *O*-Alkylation of 4-methyl-1-penten-3-ol (**7**)[10] with the sodium salt of bromoacetic acid gave **8** in 94% yield. Ozonolytic cleavage of the olefin linkage in **8** and Kugelrohr distillation provided the lactol **9** (83%) which, when reacted with excess vinylmagnesium bromide in THF at −78 °C, followed by lactonization of the hydroxy acids (camphorsulfonic acid in refluxing benzene), gave **1a** and **3a** (70%, 1.53:1). Similarly, reaction of **9** with the Grignard reagent derived from 2-bromopropene followed by acid-catalyzed lactonization gave substrate oxapyranones **1b** and **3b** (58%, 2.54:1). In every case the diastereomeric pairs produced were easily separated by flash chromatography.[11] As illustrated in Scheme I, the keto acid **11**, derived from 2-ethoxy-4-methyl-1-penten-3-ol (**10**),[12] exhibited greater stereoselectivity in the vinyl Grignard addition reactions, providing **1e** and **3e** in a 9.6:1 ratio (56%), and yielding **1f** (51%) free of the corresponding diastereomer.[13] The substrates for entries 3 and 9 were prepared by the addition of [*trans*-β-(trimethylsilyl)vinyl]lithium[14] to **9**. Note that the products of these entries incorporate an allylsilane residue[15] for further manipulation.

Also illustrated in Scheme I is a sequence by which the *trans*- or *cis*-5,6-disubstituted oxapyranones (exemplified by **1d** and **3d**, respectively) can be generated stereoselectively. Addition of the cuprate derived from *trans*-1-propenyllithium to the aldehyde **12** proceeded with >100:1 Cram-cyclic stereoselectivity[13,16] to give the hydroxy ester **13**. Acid-catalyzed lactonization gave the lactone **1d** in very high diastereomeric purity. The epimeric oxapyranone **3d** was prepared from **13** by oxidation to the enone and reduction with $Zn(BH_4)_2$[17] to provide the hydroxy ester epimer of **13**. Acid-catalyzed lactonization proceeded (albeit more sluggishly than for **1d**) to give the *cis*-5,6-disubstituted oxapyranone **3d** in 95:5 diastereomeric purity.[13]

Finally, catalytic hydrogenation of the product dihydropyrans proceeds without complication. For example (eq 3), the product **4d** is transformed to **14** in good yield,



4d **14** (3)

as shown. A comparison of **14** and the "left-wing" of the antibiotic X-14547A suggests an application of this method. Reports detailing the use of this versatile method in the synthesis of *C*-pyranosidic natural products will be forthcoming.

Acknowledgment. We gratefully acknowledge the National Institutes of Health for generously supporting this research. High-field NMR spectra were obtained through the NSF Regional NMR Center at the University of South Carolina (CHE 82-07445).

Registry No. **1a**, 92420-30-9; **1b**, 92420-31-0; **1c**, 92420-32-1; **1d**, 92420-33-2; **1e**, 92420-34-3; **1f**, 92420-35-4; **2a**, 92420-39-8; **2b**, 92420-40-1; **2c**, 92420-41-2; **2d**, 92420-42-3; **2e**, 92420-43-4; **2f**, 92420-44-5; **3a**, 92420-36-5; **3b**, 92420-37-6; **3c**, 92471-18-6; **3d**, 92471-19-7; **3e**, 92420-38-7; **4a**, 92420-45-6; **4b**, 92420-46-7; **4c**, 92420-47-8; **4d**, 92420-48-9; **4e**, 92420-49-0; **7**, 4798-45-2; **8**, 92420-50-3; **9**, 92420-51-4; **10**, 92420-53-6; **11**, 92420-52-5; **12**, 92420-54-7; **13** (epimer 1), 92456-09-2; **13** (epimer 2), 92420-55-8; **14**, 92420-56-9; bromoacetic acid sodium salt, 1068-52-6; vinyl bromide, 593-60-2; 2-bromopropene, 557-93-7; [*trans*-β-(trimethylsilyl)vinyl]lithium, 55339-31-6.

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(10) 4-Methyl-1-penten-3-ol is commercially available from Wiley Organics, Inc., Columbus, OH.
(11) Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.
(12) Prepared by the reaction of (α-ethoxyvinyl)lithium with isobutyraldehyde in THF at −78 °C. See: Baldwin, J. W.; Höfle, G. A.; Lever, O. W., Jr. *J. Am. Chem. Soc.* **1974**, *96*, 7125.
(13) The diastereomer ratios were determined by glass capillary GLC using 25-m columns coated with either SE-54 or Superox-4.
(14) (a) Cunico, R. F.; Clayton, F. J. *J. Org. Chem.* **1976**, *41*, 1380. (b) Burke, S. D.; Murtiashaw, C. W.; Dike, M. S.; Strickland, S. M. S.; Saunders, J. O. *Ibid.* **1981**, *46*, 2400.
(15) For reviews of allylsilane chemistry, see: (a) Chan, T. H.; Fleming, I. *Synthesis* **1979**, 761. (b) Colvin, E. W. "Silicon in Organic Synthesis"; Butterworths: London, 1981; p 97.
(16) Still, W. C.; Schneider, J. A. *Tetrahedron Lett.* **1980**, *21*, 1035.
(17) (a) Nakata, T.; Tanaka, T.; Oishi, T. *Tetrahedron Lett.* **1981**, *22*, 4723. (b) McGarvey, G. J.; Kimura, M. *J. Org. Chem.* **1982**, *47*, 5420.

Synthesis of Bicyclic Vinylcyclobutanes via Copper(I)-Catalyzed Intramolecular 2π + 2π Photocycloadditions of Conjugated Dienes to Alkenes[1]

Summary: Copper(I) trifluoromethanesulfonate catalyzes photobicyclization of myrcene to afford 6,6-dimethyl-2-methylenebicyclo[3.2.0]heptane, demonstrating the ability of catalysis to promote novel photochemistry.

Sir: We are intrigued by the potential for homogeneous metal catalysis in organic photochemistry[2] to provide novel and synthetically useful organic reactions. The possibility that vinyl substituents might allow useful transformations of the photoproducts led us to explore the suitability of 1,6-dienes bearing conjugated vinyl substituents as substrates for copper(I)-catalyzed intramolecular 2π + 2π photocycloadditions. We now report that copper(I) trifluoromethanesulfonate[3] (CuOTf) catalyzes a novel intramolecular 2π + 2π photocycloaddition of myrcene (**1**), affording 6,6-dimethyl-2-methylenebicyclo[3.2.0]heptane (**2**), a ring system not obtained previously from this triene upon direct[4] or triplet sensitized[5] ultraviolet irradiation.

(1) Copper(I) Catalysis of Olefin Photoreactions. 13. For paper 12 in this series, see: Salomon, R. G.; Ghosh, S.; Raychaudhuri, S. R.; Miranti, T. S. *Tetrahedron Lett.* **1984**, *25*, 3167.
(2) For a recent review, see: Salomon, R. G. *Tetrahedron* **1983**, *39*, 485.
(3) Salomon, R. G.; Kochi, J. K. *J. Am. Chem. Soc.* **1973**, *95*, 1889.

0022-3263/84/1949-4322$01.50/0