J. Org. Chem. 1990, 55, 4880–4883

New Syntheses for Dilithiotrimethylenemethane: An Ionic Energy Minimum

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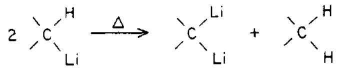
Received December 8, 1989

Thermal rearrangement of monolithium organic compounds, without β-hydrogens, have been shown to produce *gem*-dilithio organic molecules. The synthesis of three monolithium olefins, namely, 1-lithio-2-methylpropene (**1**), 3-lithio-2-methylpropene (**2**), and 1-lithio-2,2-diphenylethylene (**3**), from their respective tin derivatives are described. The thermal rearrangement of these lithiated olefins did not produce the expected geminal compounds. Both **1** and **2** produced the thermodynamically stable dilithiotrimethylenemethane, while **3** produced as the major product the coupled organic moiety 1,1,4,4-tetraphenyl-1,3-butadiene.

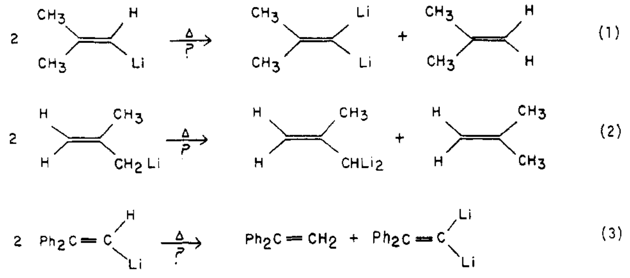
A new synthesis of *gem*-dilithio organic compounds by thermal rearrangement of monolithium organic compounds has been developed in our laboratory.[1-4] The reaction



mechanism for this type of reaction has not yet been established; however, dilithiomethanes are formed when no β-hydrogens are present in the reactant molecule. Monolithium molecules containing β-hydrogen undergo lithium hydride elimination to produce olefins.

The Kawa–Lagow-modified Ziegler rearrangement[4] produces highly pure *gem*-dilithio organic compounds, which are currently of great interest. These compounds may have unusual structural features (i.e., planar carbon) as predicted from ab initio molecular orbital calculations.[5] We are interested in applying this synthetic route in the preparation of *gem*-dilithio olefins. Here we report the synthesis of three monolithium olefin complexes from various organotin compounds. The choice of organotin reagent and solvent play an important role in the substitution of lithium for the trialkyl(aryl)tin group. The organotin reagents are used to avoid lithium halide contamination.

The monolithium olefins synthesized are 1-lithio-2-methylpropene (**1**), 3-lithio-2-methylpropene (**2**), and 1-lithio-2,2-diphenylethylene (**3**). These compounds do not contain β-hydrogens; therefore, the thermolysis reactions might be expected to take the following routes:



We will demonstrate that unfortunately this is not the route taken. The thermolyses of both **1** and **2** yield dilithiotrimethylenemethane, while the thermolysis of **3** gives the coupled organic moiety ($Ph_2C{=}CHCH{=}CPh_2$) as the major product.

Experimental Section

General Conditions. All solvents were dried and distilled under argon before use. Standard Schlenk and vacuum line techniques were used. ^1H and ^{13}C NMR spectra were taken on either a Nicolet NT-200 or NT-360 instrument. GC/MS were run on a Finnigan 4000 using a 30 m × 0.25 mm Heliflex bonded phase RSL-200 1.0 μm capillary column, with an injector temperature of 250 °C and an ionizer temperature of 150 °C. All thermolysis reactions were carried out in a Pyrex flask, equipped with a glass-covered stir bar, attached to a vacuum line through a liquid nitrogen trap. The solid remaining after heating was then derivatized (vide infra), and the organics were analyzed through GC/MS and NMR. The electron spin resonance spectrometer used was an IBM ER300.

Preparations. To a suspension of methyllithium (0.0348 mol) in hexanes was added isobutenyltrimethyltin[6] (0.0456 mol), and the solution was refluxed for 18 h. The solvent was vacuum distilled off, and the remaining cream-colored solids **1** were washed with pentane (yield 60%). Characterization was achieved through spectral analysis of the hydrolyzed organolithium product (vide infra).

2 can be synthesized via two routes. The first route follows Seyferth's preparation.[7] While Seyferth did not isolate the organolithium compound (it was only used in situ), we isolated **2** through the use of mixed solvent recrystallization (ether, benzene, and pentane) (yield 75%). The second route reacts methyllithium (0.0233 mol) with methallyltrimethyltin (0.0216 mol) in refluxing ether. The product was recrystallized from pentane, yielding a cream-colored powder (45% yield). Characterization was again achieved through spectral analysis of the hydrolyzed organolithium compound (vide infra).

The best procedure in synthesizing **3** was through the reaction of methyllithium with (2,2-diphenylethenyl)trimethyltin[8] in THF. The choice of solvent was critical in allowing the reaction to proceed (vide infra). A typical reaction was run in the following manner: to a stirred THF solution of (2,2-diphenylethenyl)trimethyltin (0.00566 mol) was added an ether solution of methyllithium (0.00570 mol). After 2 h, the resulting purple-blue solution was reduced in volume and pentane was added. The dark blue solids were collected and dried under vacuum (90% yield). Characterization was done through the spectral analysis of products derived from hydrolysis (vide infra).

Attempts to make **3** using (2,2-diphenylethenyl)triphenyltin and phenyllithium, in various solvents, lead instead to the for-

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[1] Chinn, J. W., Jr.; Gurak, J. A.; Lagow, R. J. *J. Am. Chem. Soc.* **1982**, *104*, 2637.

[2] Kawa, H.; Manley, B. C.; Lagow, R. J. *J. Am. Chem. Soc.* **1985**, *107*, 5313.

[3] Chauhan, H. P. S.; Kawa, H.; Lagow, R. J. *J. Org. Chem.* **1986**, *51*, 1632.

[4] Kawa, H.; Manley, B. C.; Lagow, R. J. *Polyhedron* **1988**, *7*, 2023.

[5] (a) Schleyer, P. v. R.; Seeger, R.; Pople, J. A. *J. Am. Chem. Soc.* **1976**, *98*, 5419. (b) Apeloig, Y.; Schleyer, P. v. R.; Binkley, J. S.; Pople, J. A. *J. Am. Chem. Soc.* **1976**, *98*, 4332.

[6] Synthesized according to Sheffy et al. Sheffy, F. K.; Godschalx, J. P.; Stille, J. K. *J. Am. Chem. Soc.* **1984**, *106*, 4833.

[7] Seyferth, D.; Weiner, M. A. *J. Org. Chem.* **1961**, *26*, 4797.

[8] Synthesized in an analogous manner as the other tin olefin compounds: bp 130–133 °C (1–2 mm); ^1H NMR (CCl_4) phenyls 7.28 (m), =CH 6.58 (s), $SnMe_3$ −0.09 ppm.

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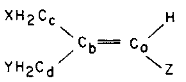
Table I. $^{13}C\{^1H\}$ **NMR Data for Deuterated 2-Methylpropene Derivatives**



	x, y, z		
C_x	H, H, D	D, H, H	D, D, H
C_a	110.17[a]	110.45	110.46
C_b	142.16	142.10	142.17
C_c } C_d	23.95	23.62[b] } 23.90	23.68[c]

[a] $J(^{13}C-^2H) = 23.7$. [b] $J(^{13}C-^2H) = 20.0$. [c] $J(^{13}C-^2H) = 19.7$.

mation of lithiotriphenyltin and triphenylethylene.

Results and Discussion

Characterizations of the monolithium olefins were done through derivatization in order to be certain of the lithium location. Derivatization was accomplished via hydrolysis (D_2O) and/or methylation using iodomethane. The organic products obtained were analyzed using GC/MS and NMR spectroscopy. The hydrolysis (D_2O) of **1** yielded >95% 2-methylpropene-*d* via GC/MS, mass units (%): 58 (2.8), 57 (60.2), 55 (22.1), 55 (5.1), 54 (5.1), base peak 42. GC/MS results for 2-methylpropene, mass units (%): 57 (2.60), 56 (52.03), 55 (20.94), 54 (2.11), base peak 41. The location of the deuterium was determined, via NMR, to be in the vinyl position. ^1H NMR: 4.65 (s, 1.0), 1.73 (s, 5.5). $^{13}C\{^1H\}$ NMR: 142.16, 110.17 (t, $J(^{13}C-^2H) = 23.7$ Hz), 23.95. The reaction of **1** with iodomethane yielded 2-methylbut-2-ene in 45% yield, as determined through GC/MS. The hydrolysis (D_2O) of **2** again yields 2-methylpropene-*d*. Through ^{13}C NMR spectroscopy the deuterium was determined to be located on a methyl group. The relevant data are in Table I. The reaction of **2** with methyl iodide yielded (47%) 2-methylbut-1-ene. The reaction of D_2O with **3** yielded >96% 1,1-diphenylethene-*d* as determined from GC/MS, mass units (%): 182 (8.3), 181 (100), 180 (63.6), 179 (50.6), 178 (4.8). GC/MS results for 1,1-diphenylethene, mass units (%): 181 (16.7), 180 (100), 179 (74.7), 178 (61.0), 177 (8.63). NMR spectroscopy showed that the deuterium was located in the vinyl position. The ^1H NMR signals were at 7.1 (m) for the phenyls and at 5.7 (s) for the vinyl hydrogen. The ^2H NMR showed only one peak at 5.56, which is indicative of deuterium in a vinyl position. This is also supported by $^{13}C\{^1H\}$ NMR data. The $^{13}C\{^1H\}$ NMR signals were found at 113.97 (t, $J(^{13}C-^2H) = 23.9$ Hz), 141.43 (s), and complex phenyl carbons centered near 128.1.

The thermal rearrangement, or disproportionation, of **1** and **2** will be discussed first. **1** was heated at 140 °C for 24 h. The volatile products that were collected in the liquid nitrogen trap were analyzed by GC/MS to be 2-methylpropene (0.1798 g, 86%). The resulting red-purple to red-brown solid (0.245 g) was found to be insoluble in benzene, ether, tetrahydrofuran, and tetramethylethylenediamine. Characterization of the solid remaining after heating was accomplished through the analysis of the products obtained after derivatization. Derivatization was accomplished either by slow hydrolysis with deuterium oxide or by methylation with iodomethane in tetrahydrofuran. The resulting products were analyzed by GC, GC/MS, and NMR (^1H, $^{13}C\{^1H\}$) spectroscopy. The reaction with deuterium oxide yielded dideuterio-2-methylpropene (86.3%) along with polydeuterated propene (12.7%), propyne (0.2%), and acetylene (0.7%). GC/MS data for dideuterio-2-methylpropene, mass units (%): 59 (4.8), 58 (59.3), 57 (23.9), 56 (6.3), 55 (3.10), 54 (3.0). The ^1H NMR spectrum of the product from this hydrolysis contained signals at 4.66 (br, s, 2 H) and 1.72 ppm (br, m, 4 H), while the $^{13}C\{^1H\}$ NMR contained peaks at 142.17 (s), 110.46 (s), and 23.68 ppm (t, $J(^{13}C-^2H) = 19.74$ Hz). These results established the location of both deuterium atoms on the methyl groups (one on each) of 2-methylpropene. The reaction with methyl iodide in tetrahydrofuran gave 2-ethyl-1-butene in 41% yield (see eq 4).

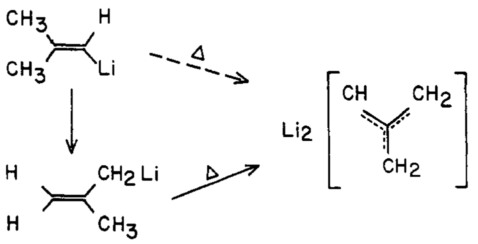
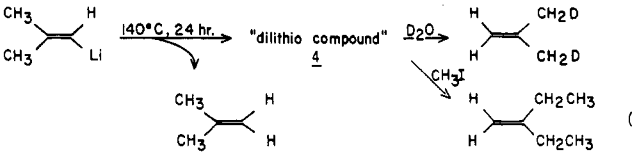
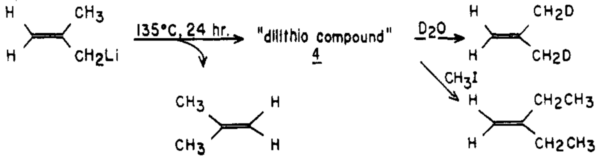


Figure 1. Dilithiotrimethylenemethane = dilithio-2-methylpropene.



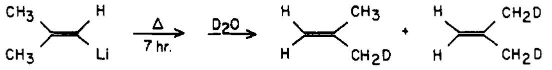
(4)

Similarly, thermolysis of 3-lithio-2-methylpropene (**2**) (0.530 g), at slightly lower temperatures (135 °C), resulted in producing the same product as above. After 24 h of heating, 2-methylpropene (0.234 g, 97%) was obtained in the cold trap and a red-brown solid (0.258 g, 89%) remained in the reaction flask. The deuterolysis of the solid again gave dideuterio-2-methylpropene in which the two deuteriums were also found to be located on the methyl groups by ^1H and $^{13}C\{^1H\}$ NMR analysis. The reaction with iodomethane in tetrahydrofuran gave 2-ethyl-1-butene in 40% yield (see eq 5).



(5)

In order to determine if there was a common pathway that both **1** and **2** used, in order to arrive at **4**, the thermolysis of **1** was stopped after 7 h. The resulting solid was hydrolyzed with deuterium oxide, and through GC/MS and $^{13}C\{^1H\}$ NMR only 3-deuterio-2-methylpropene and dideuterio-2-methylpropene were formed (see eq 6). This result implies that 1-lithio-2-methylpropene first isomerizes into 3-lithio-2-methylpropene and then disproportionates into 2-methylpropene and dilithio-2-methylpropene (**4**) (see Figure 1).



(6)

Dilithio-2-methylpropene has been synthesized before; however it is solvated by two molecules of tetramethylethylenediamine. Klein reacted 2-methylpropene with 2 equiv of *n*-butyllithium, in the presence of 2 equiv of tetramethylethylenediamine, in hexanes.[9] The product obtained was analyzed through ^1H NMR and its derivatization products and was formulated as the trimethylenemethane dianion. This 6-π-electron system has been said to be stabilized through Y aromaticity.[10] This

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[9] Klein, J.; Medlik, A. *J. Chem. Soc., Chem. Commun.* **1973**, 275.

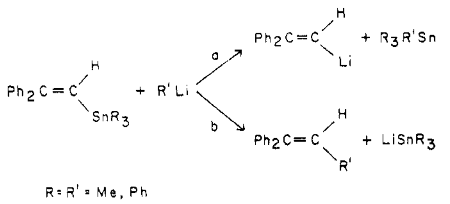

Figure 2. Reaction pathways for the reaction of phenyl- or methyllithium with (1,1-diphenylethenyl)triphenyltin or (1,1-diphenylethenyl)trimethyltin in THF.

extra stabilization is more than likely the driving force, i.e., thermodynamic sink, in yielding dilithiotrimethylenemethane from the thermolysis of the lithio-2-methylpropene isomers 1 and 2.

Two independent routes to the formation of 1,1-dilithio-2-methylpropene have been claimed. The first was by Lagow and co-workers via the reaction of lithium vapor with 2-methylpropene.[11] Hydrolysis of the products showed that substitution was preferentially achieved at the vinyl positions, i.e., products formed were 1-lithio-2-methylpropene (28%) and 1,1-dilithio-2-methylpropene (4%). The second route involved the cleavage of 2,3-dilithio-1,3-butadiene with $HgCl_2$ followed by reaction with lithium powder to produce 1,1-dilithio-2-methylpropene.[12] These two methods contrast with our present work in that the thermolysis produced the thermodynamically more stable dilithiotrimethylenemethane molecule in contrast to the geminal 1,1-dilithio-2-methylpropene formed by Lagow's and Maercker's methods.

In order to prevent the formation of this stable Y delocalization from occurring, substitution of the methyl groups in 2-methylpropene was explored. The parent compound chosen to work with was 1,1-diphenylethylene. The first reported in situ synthesis of 1-lithio-2,2-diphenylethylene was by Curtin and Flynn.[13] They reacted 1-bromo-2,2-diphenylethylene with *n*-butyllithium and obtained a 30% yield of β-phenylcinnamic acid after the solution was treated with carbon dioxide and acidified. Köbrick and Stöber further studied this reaction, achieving slightly higher yields of acids.[14] However, isolation of 1-lithio-2,2-diphenylethylene was not possible using this method. Thus the same synthetic route as for the lithio-2-methylpropene was chosen, i.e., synthesis via organotin compounds.

Initial work was with (1,1-diphenylethenyl)triphenyltin. When this was reacted with phenyllithium in ether, very little tetraphenyltin precipitated from solution. Analysis of the products after hydrolysis showed that not more than 8% of the organotin reacted. The reaction was repeated in tetrahydrofuran, expecting that the more polar solvent would drive the reaction. It did indeed drive the reaction; after derivatization with iodomethane, less than 15% of the organotin compound was present. The majority of the material was, however, found to be triphenylethylene and methyltriphenyltin. Smaller amounts of 1,1,4,4-tetraphenylbutadiene were also found. Therefore, the reaction pathway was found to follow path b and not path a as we expected (see Figure 2).

We then decided to make the analogous trimethyltin compound (see Experimental Section) and test its reactivity with methyllithium. When the reaction was carried out in either hexanes or ether, no or very little (<10%) reaction occurred, respectively. When tetrahydrofuran was used as the solvent, the reaction turned deep blue-purple in a couple of hours. Hydrolysis (D_2O) of the solution gave quantitative yields of 1-deuterio-2,2-diphenylethylene via GC/MS, mass units (%): 182 (14.7), 181 (100), 180 (67.1), 179 (51.9), 178 (11.6), 177 (8.1), 176 (1.4). GC/MS results for 1,1-diphenylethene, mass units (%): 181 (12.6), 180 (100), 179 (67.0), 178 (51.4), 177 (4.4). The location of the deuterium was confirmed to be in the vinyl position by $^{13}C\{^1H\}$ and ^2H NMR analysis. The $^{13}C\{^1H\}$ resonances were located at 141.43 (s) and 113.97 (t, $J\{^{13}C-^2H\}$ = 23.93 Hz), and 127.68, 128.13, 128.24, and 128.37 for the phenyl carbons. The ^2H NMR had one peak at 5.56 ppm, indicative of a vinylic deuterium. Derivatization of the blue solution with trimethylsilyl chloride yielded (1,1-diphenylethenyl)trimethylsilane. Therefore, the reaction in Figure 2 follows path a when R = Me, R′ = Me and when the solvent is THF.

Table II. Pyrolysis Conditions and Products of 1-Lithio-2,2-diphenylethylene (3)

time (h), temp (°C)	derivatizing agent	products
5, 95	D_2O	$Ph_2C{=}CHD$
24, 125	MeI	$Ph_2C{=}CHMe$, $(Ph_2C{=}CH)_2$
24, 130	MeI	$Ph_2C{=}CHMe$, $(Ph_2C{=}CH)_2$
24, 150	D_2O	Ph_2CHD, $Ph_2C{=}CHD$, $(Ph_2C{=}CH)_2$

Now that compound 3 can be isolated and purified, thermolysis of 3 can be examined. The first reaction started with short heating times (5 h) at low temperatures (95 °C), followed by reactions at higher temperatures and longer heating times (24 h) (see Table II). After thermolysis at 95 °C for 5 h, hydrolysis (D_2O) showed that only starting material was present. When the temperature of pyrolysis was increased to 125 °C and left at the temperature for 24 h, there was only a trace amount of 3 left as determined through the derivatization of the remaining solid with methyl iodide. The majority of the product formed as 1,1,4,4-tetraphenylbutadiene. Increasing the temperature further only lead to further decomposition of the starting material, i.e., fragmentation into lithiodiphenylmethane. Since the pyrolysis of 3 produced the coupled organic product in high yields, the query as to why arose. Going back and looking at 3 in more depth showed that 3 is quite unique. The ^1H NMR spectrum was difficult to obtain and produced only broad peaks in the phenyl and vinyl proton areas. Therefore, the paramagnetic properties were checked. 3 indeed has an unpaired electron as evidenced by a signal observed at 3364.83 G at room temperature (g = 2.123). Because of its radical property, the thermolysis of 3 probably favored formation of the dimer over any possible disproportionation products.

Conclusion

We have demonstrated that the thermal disproportionation of two structural isomers, namely, 1-lithio-2-methylpropene and 3-lithio-2-methylpropene, yields the same final product. This product was determined to be dilithiotrimethylenemethane. Its formation showed that it is indeed a stabilized molecule and therefore the thermodynamic sink for this thermal rearrangement reaction. Since no bases are attached, our dilithiotrimethylenemethane differs from Klein's quite dramatically. The thermal rearrangement of 1-lithio-2,2-diphenylethylene (3) gave a very interesting result. The product formed was that of dimerization (1,1,4,4-tetraphenylethene). Since 3 was found to have radical character, this might be the

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(12) Maercker, A.; Dujardin, R. *Angew. Chem., Int. Ed. Engl.* **1985**, *24*, 571.

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driving force in forming the dimer over any possible disproportionation products. Further work is in progress in the direction of the synthesis of 1-lithio-2,2-di-*tert*-butylethene and its thermal rearrangements.

It is very interesting to note that two completely different synthetic routes lead to the same dilithiotrimethylenemethane compound. This ionic form therefore definitely represents an energy minimum. This finding would add support for the thesis of Streitwieser and coworkers that many polylithium organic compounds are of a highly ionic nature.[15]

Acknowledgment. We are grateful to the National Science Foundation (CHE-8521390) and the Robert A. Welch Foundation (F-700) for support of this work.

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Radical Cyclization Strategies to Bridged Systems. Synthesis of Bicyclo[3.2.1]octan-3-ones from (*S*)-Carvone[1]

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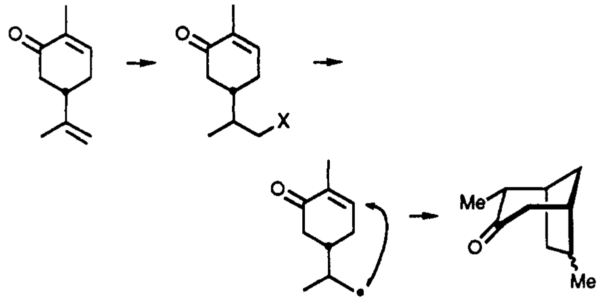
Received December 12, 1989

Radical cyclization of the bromo enones **2a–e**, obtained by regiospecific bromoetherification reaction on the electron-rich double bond of (*S*)-carvone (**1**), furnished regio- and stereospecifically bicyclo[3.2.1]octan-3-ones **3a–e** and **4a–e**. Analogously, radical cyclizations of the alcohols **6** and **7** gave bicyclo[3.2.1]octan-3-ols **8** and **9**, and the bromo enones **11a,b** gave the bridgehead-substituted bicyclo[3.2.1]octan-3-ones **12a,b** and **13a,b**.

The development of synthetic methods for the preparation of bridged systems has been stimulated by the discovery of polycyclic natural products that incorporate bridged systems as part structures. The bicyclo[3.2.1]octane system has received a relatively large amount of attention[2] due to its frequent presence in various sesqui- and diterpenoids. In the last decade there has been an upsurge of interest in the application of free-radical cyclization for the synthesis of fused carbo- and heterocyclic systems.[3] Even though the first report of formation of a bridged system by a radical cyclization reaction appeared in 1983, until recently, relatively little attention was given to the synthesis of bridged[4] systems by transannular radical cyclizations.[5]

In our quest for a simple method for the construction of chiral bicyclo[3.2.1]octanes, we conceived of a two-step sequence (eq 1) wherein carvone (**1**) is converted into a radical precursor, by manipulation of the electron-rich olefin, and regiospecific intramolecular addition of the radical to the enone moiety. This sequence has several attractive features, including the commercial availability of both the enantiomers of carvone (**1**), formation of two new stereocenters in a regio- and stereospecific way, and flexibility to provide more functionalized bicyclo[3.2.1]octanes. In this account, we report a general, efficient, and preparative route to chiral bicyclo[3.2.1]octan-3-ones and describe its extension to the bridgehead-substituted systems present in variety of natural products.



(1)

The sequence is depicted in the Scheme I. The radical derived from the bromide **2**, obtained from carvone by a bromoetherification reaction, on 5-*exo-trig* cyclization followed by abstraction of hydrogen from tin hydride furnishes the bicyclic systems. Thus, *N*-bromosuccinimide (NBS) bromination of the electron-rich olefin in (*S*)-carvone (**1**) in methanol–methylene chloride medium furnished an inseparable mixture (1:1 by ^{13}C NMR) of 4*S*,8*R* and 4*S*,8*S* diastereomeric methoxy bromides **2a** in a regiospecific manner. Refluxing a 0.02 M benzene solution of the bromide mixture **2a** with 1.1 equiv of tri-*n*-butyltin hydride (TBTH) in the presence of a catalytic amount of azobisisobutyronitrile (AIBN) for 3 h cleanly furnished, in 82% yield, a separable mixture of endo and exo bicyclic products **3a** and **4a**. The structures were delineated through their spectral data. Both **3a** and **4a** have bands at 1700 cm^{-1} (saturated carbonyl group) in their IR spectra and saturated carbonyl carbon resonances in their ^{13}C NMR spectra (δ 211.4 and 212.5). The absence of olefinic carbons in the ^{13}C NMR spectra, and olefinic proton, olefinic methyl, and bromomethylene resonances in the ^{1}H NMR spectra further support the cyclic structures. The presence of methyl doublets at δ 1.03 and 0.99 in the ^{1}H NMR spectra clearly establish not only the cyclization but

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[1] Part 4 in the series: Chiral synthons from carvone. Part 3: Srikrishna, A.; Hemamalini, P. *Indian J. Chem.* **1990**, *29B*, 201.

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[3] For recent reviews see: (a) Ramaiah, M. *Tetrahedron* **1987**, *43*, 3541. (b) Curran, D. P. *Synthesis* **1988**, 417 and 489.

[4] Some aspects of difficulty in bridge formations by the tin hydride method are discussed in ref 3b, sec. 5.2.2.

[5] For earlier reports, see: Stork, G.; Baine, N. H. *Tetrahedron Lett.* **1985**, *26*, 5927. Marinovic, N. N.; Ramanathan, H. *Tetrahedron Lett.* **1983**, *24*, 1871. Choi, J.-K.; Ha, D.-C.; Hart, D. J.; Lee, C.-S.; Ramesh, S.; Wu, S. *J. Org. Chem.* **1989**, *54*, 279. MacCorquodale, F.; Walton, J. C. *J. Chem. Soc., Perkin Trans. 1* **1989**, 347. Yadav, V.; Fallis, A. G. *Tetrahedron Lett.* **1989**, *30*, 3283. Vite, G. D.; Alonso, R.; Fraser-Reid, B. *J. Org. Chem.* **1989**, *54*, 2268. Rajamannar, T.; Balasubramanian, K. K. *Tetrahedron Lett.* **1988**, *29*, 5789. Vijayabhaskar, K.; Subba Rao, G. S. R. *Tetrahedron Lett.* **1989**, *30*, 225. Srikrishna, A.; Hemamalini, P. (part 2 of this series), *J. Chem. Soc., Perkin Trans. 1* **1989**, 2511.

0022-3263/90/1955-4883$02.50/0 © 1990 American Chemical Society