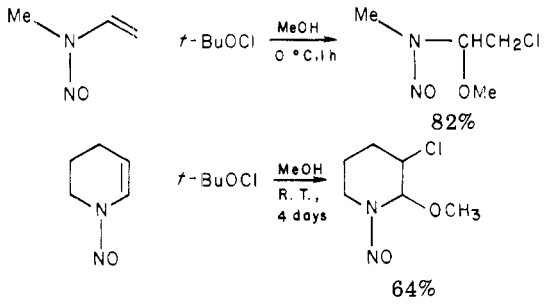
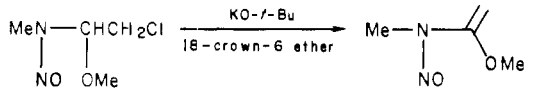
The reaction of *tert*-butyl hypochlorite with the nitrosoenamines in methanol gave good yields of β-chloro-α-methoxy derivatives. The chloromethoxy adducts allow



a convenient preparation of the heretofore unknown enol ethers of *N*-nitrosamides. Thus, treatment of methyl(1-methoxy-2-chloroethyl)nitrosamine with potassium *tert*-butoxide in ether in the presence of a catalytic quantity of 18-crown-6 ether resulted in a smooth dehydrohalogenation to the corresponding enol ether. Enol ethers of *N*-nitrosamides are potentially very interesting substances because they are readily hydrolyzed to *N*-nitrosamides in mild acid.



In forthcoming publications, applications of some of these reactions will be described.

Registry No. Methylvinylnitrosamine, 4549-40-0; methylhexylnitrosamine, 28538-70-7; cyclohexanone, 108-94-1; methyl(2-(cyclohexanon-2-yl)ethyl)nitrosamine, 73908-51-7; methyl(2-phenylethyl)nitrosamine, 13256-11-6; 2,3-dehydro-*N*-nitrosopyrrolidine, 70501-84-7; 3-methoxy-*N*-nitrosopyrrolidine, 61467-70-7; 3-*tert*-butoxy-*N*-nitrosopyrrolidine, 73908-52-8; methanol, 67-56-1; *tert*-butyl alcohol, 75-65-0; 2,3-dehydro-*N*-nitrosopiperidine, 70501-82-5; 3-methoxy-*N*-nitrosopiperidine, 73908-53-9; acetic acid, 64-19-7; methyl(1-acetoxyethyl)nitrosamine, 65986-79-0; methyl(1-methoxyethyl)nitrosamine, 61738-05-4; methyl(2-chloro-1-methoxyethyl)-nitrosamine, 73926-11-1; 3-chloro-2-methoxy-*N*-nitrosopiperidine, 73908-54-0; methyl(1-methoxyethenyl)nitrosamine, 73908-55-1.

Supplementary Material Available: Experimental detail for general reactions of selected *N*-nitrosoenamines (3 pages). Ordering information is given on any current masthead page.

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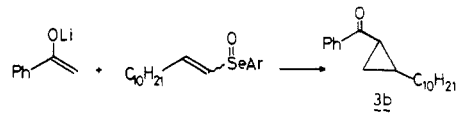
Aryl Vinyl Selenoxide as a Versatile Reagent for Transfer of an Ethylene Unit to Enolates. New Synthetic Method of Formation of Cyclopropyl Ketones

Summary: The reaction between an aryl vinyl selenoxide and the lithium enolate of a ketone or an ester gave a cyclopropyl carbonyl compound in good yield.

Sir: Recent developments in organoselenium chemistry have realized a variety of useful reactions for constructing versatile synthetic intermediates.[1]

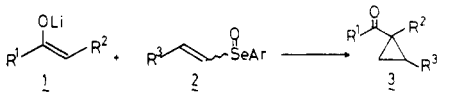
Table I. Effect of Aryl Substituents[a]



Ar	solvent	% yield of 3b[b]
C_6H_5	Et_2O	63
o-$CH_3C_6H_4$	THF	17
m-$CF_3C_6H_4$	Et_2O	73
o-$NO_2C_6H_4$	Et_2O	trace
p-ClC_6H_4	Et_2O	81
p-ClC_6H_4	THF	69
p-ClC_6H_4	THF[c]	91

[a] Reactions were carried out on 0.5-mmol scale with a reactant ratio, enolate-selenoxide = ~1.2-1.25:1.0. [b] Isolated yield. [c] Enolate (1.7 equiv) was used.

We examined the reactivity of aryl vinyl selenoxides[2,3] as 1,4-addition acceptors and found a novel cyclopropanation reaction between aryl vinyl selenoxides and ketone or ester enolates, as shown in eq 1, which consti-



(1)

tutes an efficient method for the transfer of an ethylene unit to ketones or esters to produce cyclopropyl carbonyl compounds.

The following example illustrates a typical procedure. To a solution of LDA (0.75 mmol) in ether (2 mL) was added a solution of acetophenone (66 mg, 0.55 mmol) in ether (3 mL) at −30 °C and the mixture was stirred at that temperature for 30 min. An ethereal (5 mL) solution of *p*-chlorophenyl 1-dodecenyl selenoxide[4] (164 mg, 0.44 mmol) was added to the solution of the enolate and the mixture was stirred at −30 °C for 1 h and then at room temperature for 5 h. The pale yellow mixture was washed with saturated aqueous NaCl and dried. Removal of the solvent followed by purification by preparative TLC gave 1-benzoyl-2-decylcyclopropane (102 mg, 81%) as a colorless oil.

As shown in Table I, significant effects of substituents on the aryl selenoxide moiety were observed in the present reaction. A *p*-chloro or *m*-trifluoromethyl substituent effectively enhanced the desired reaction. In contrast to their effects of enhancing the syn elimination reaction of alkyl aryl selenoxides,[5] *o*-nitrophenyl selenoxide gave poor results. Because *p*-chlorophenyl dodecenyl selenoxide afforded the corresponding cyclopropyl ketone in high

(1) For recent reviews, see: Sharpless, K. B.; Lauer, R. F.; Patrick, D. W.; Singer, S. P.; Young, M. W. *Chem. Scr.* **1975**, *8A*, 9; Clive, D. J. L. *Tetrahedron* **1978**, *34*, 1049; Reich, H. J. *Acc. Chem. Res.* **1979**, *12*, 22.

(2) The preparation of vinyl selenoxides and some of their reactions were reported: Sevrin, M.; Dumont, W.; Krief, A. *Tetrahedron Lett.* **1977**, 3835; see also footnote 7 of ref 6e.

(3) For the use of vinyl sulfoxide as conjugate addition acceptor, see: Tsuchihashi, G.; Mitamura, S.; Inoue, S.; Ogura, K. *Tetrahedron Lett.* **1973**, 323; Tsuchihashi, G.; Mitamura, S.; Ogura, K. *Ibid.* **1976**, 855; Koppel, G. A.; Kinnick, M. D. *J. Chem. Soc., Chem. Commun.* **1975**, 473.

(4) Aryl vinyl selenides were oxidized either by *m*-CPBA or $NaIO_4$. The yields of selenoxides were ~90–100%; see ref 2.

(5) An electron-withdrawing substituent on the aryl group enhances the selenoxide elimination reaction: Sharpless, K. B.; Young, M. W. *J. Org. Chem.* **1975**, *40*, 47; Grieco, P. A.; Masaki, Y.; Boxler, D. *J. Am. Chem. Soc.* **1975**, *97*, 1597; Grieco, P. A.; Noguez, J. A.; Masaki, Y. *Tetrahedron Lett.* **1975**, 4213; Reich, H. J.; Renga, J. M.; Reich, I. L. *J. Am. Chem. Soc.* **1975**, *97*, 5434.

(6) For the preparation of vinyl selenides, see: (a) Reich, H. J.; Chow, F. *J. Chem. Soc., Chem. Commun.* **1975**, *790*; (b) Raucher, S. *J. Org. Chem.* **1977**, *42*, 2950; (c) Raucher, S.; Hansen, M. R.; Colter, M. A. *Ibid.* **1978**, *43*, 4885; (d) Raucher, S.; Koolpe, G. A. *Ibid.* **1978**, *43*, 3794; (e) *Ibid.* **1978**, *43*, 4252; (f) ref 2; (g) Dumont, W.; Van Ende, D.; Krief, A. *Tetrahedron Lett.* **1979**, 485; (h) Sevrin, M.; Denis, J. N.; Krief, A. *Angew. Chem., Int. Ed. Engl.* **1978**, *17*, 526.

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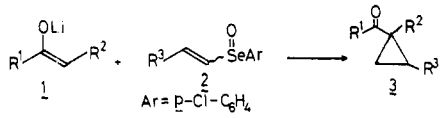
Table II. Reaction of Aryl Vinyl Selenoxide with Enolates[a]



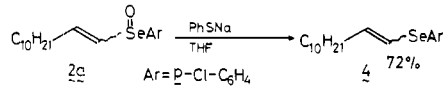
entry	R^1	R^2	R^3	% yield[b] of 3[c]
1	C_6H_5	H	H[d]	72[e]
2	C_6H_5	CH_3	H	59[e]
3	C_2H_5O	C_6H_5S	H	65[e]
4	C_6H_5	H	$C_6H_5CH_2OCH_2$[f]	70
5	C_6H_5	CH_3	$C_6H_5CH_2OCH_2$	84[e]
6	C_2H_5O	C_6H_5S	$C_6H_5CH_2OCH_2$	72
7	$(CH_2)_{10}$		$C_6H_5CH_2OCH_2$	55[e]
8	C_6H_5	CH_3	$C_{10}H_{21}$	62[e]
9	C_2H_5O	C_6H_5S	$C_{10}H_{21}$	71
10	$(CH_3)_3C$	H	$C_{10}H_{21}$	72
11	$(CH_3)_3CO$	H	$C_{10}H_{21}$	52
12	$(CH_2)_4$		$C_{10}H_{21}$	51[e]
13	$(CH_3)_2CH$	H	$C_{10}H_{21}$	56
14	C_5H_{11}	C_6H_5S	$C_{10}H_{21}$	47
15	C_2H_5O	C_6H_5SO	$C_{10}H_{21}$	35
16	C_2H_5O	$C_6H_5SO_2$	$C_{10}H_{21}$	13
17	C_2H_5O	C_2H_5O-CO	$C_{10}H_{21}$	23

[a] Reactions were carried out on 0.5-mmol scale with a reactant ratio, selenoxide-ketone-LDA = 1.0:1.20~1.25:1.5. [b] Isolated yield. [c] All products were fully characterized by their spectroscopic data and elemental analyses. [d] The starting selenide was prepared according to footnote 6 of ref 6e. [e] Selenoxide-ketone-LDA = 1.2:1.0:1.5. [f] For the preparation, see ref 17.

yield and because its analogues, various *p*-chlorophenyl vinyl selenoxides, were easily accessible from the corresponding olefins, they were most suitably employed for the present purpose.

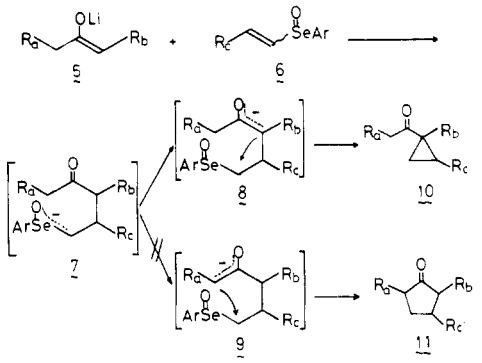
This reaction appears sensitive to the reaction solvent. For instance, a considerable amount (up to 15%) of α-arylselenoacetophenone was obtained in THF, whereas ether or DME prevented this side reaction.

Table II summarizes results for the syntheses of various cyclopropyl carbonyl compounds. As shown, in the present reaction relatively hard nucleophiles such as ketone or α-phenylsulfenyl ester enolates (entries 3, 6, and 9) serve the purpose, while (phenylthio)methyl ketone[7] enolate (entry 14) gave the expected product in moderate yield. Lithium enolates of diethyl malonate (entry 17) and α-phenylsulfonyl (entry 16) or α-phenylsulfinyl (entry 15) ester enolate afforded cyclopropyl esters in low yields. When vinyl selenoxide **2a** was treated with sodium phenylthiolate in THF, reduction of the selenoxide moiety took place to give the starting selenide **4**.[8]



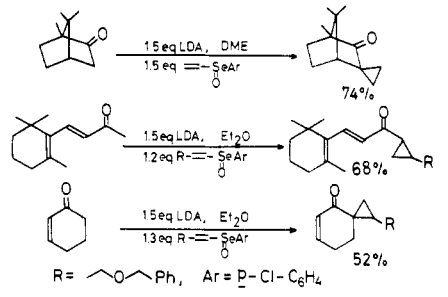
From the mechanistic point of view, the involvement of proton transfer appears to be reasonable and compatible with the observed results. The present reaction possibly proceeds through conjugate addition of nucleophile **5** to vinyl selenoxide **6** followed by proton transfer from **7** to **8** and displacement of the arylselenino group as outlined in Scheme I.[9] This type of intramolecular displacement

Scheme I



has closely related precedents in sulfone chemistry.[10] Although the formation of a cyclopentanone derivative **11** (via **7** to **9**) is possible where the ketone is enolizable on both sides (entries 7, 12, and 13), as indicated in Scheme I, such products were not obtained in any case.[11] A slight excess of either enolate or selenoxide and the use of 1.5 equiv of LDA were required for the optimum yield.

Further applications to cyclohexenone, β-ionone, and camphor[12] show the utility of this reaction.



Transition metal catalyzed reactions of diazomethyl ketones[13] have been used for one-step construction of cyclopropyl ketones and the use of vinylsulfonium[14] and/or oxosulfonium[15] salts also enables the transfer of an ethylene unit to active methylene compounds.[16] Being complementary to these methods, the present procedure

(7) Prepared as reported before: Kuwajima, I.; Kurata, Y. *Chem. Lett.* **197**, 291.

(8) Vinyl selenones appear to exhibit different reactivities. For example, oxidation of dodecenyl phenyl selenide with 2 equiv of *m*-CPBA followed by treatment with sodium hydroxide in methanol gave 1,2-dimethoxydodecane in 51% yield.

(9) Reaction of methyl phenyl selenoxide with the lithium enolate of cyclohexanone produced 2-methylcyclohexanone together with the parent ketone. This verifies the possibility of the proposed S_N2 displacement of the arylselenino group in the present reaction.

(10) For cyclopropane formation via an S_N2 type elmination of a phenylsulfonyl group, see: Martel, J.; Huynh, C. *Bull. Soc. Chim. Fr.* **1967**, 985; Velluz, L.; Martel, J.; Namine, G. *C. R. Hebd. Seances Acad. Sci., Ser. C* **1965**, *268*, 2199; Julia, M.; Guy-Rouault A. *Bull. Soc. Chim. Fr.* **1967**, 1411; Campbell, R. V. M.; Crombie, L.; Pattenden, G. *J. Chem. Soc., Chem. Commun.* **1971**, 218; Crombie, L.; Findley, D. A. R.; Whiting, D. A. *Ibid.* **1972**, 1045.

(11) In the case of 4-phenyl-2-butanone, the product was slightly contaminated with the selenenylated product.

(12) DME was used as a solvent. In THF, ca. 18% of α-selenenylated product was formed together with the desired cyclopropyl ketone.

(13) See, for example: ApSimon, J., Ed., "The Total Synthesis of Natural Products"; Wiley Interscience: New York, 1973; Vol. II, pp 197–558, and references cited therein; Nakamura, A.; Konishi, A.; Tatsuno, Y.; Otsuka, S. *J. Am. Chem. Soc.* **1978**, *100*, 3443, 3449, and references cited therein.

(14) Gosselck, J.; Ahlbrecht, H.; Dost, F.; Schenk, H.; Schmidt, G. *Tetrahedron Lett.* **1968**, 995; Schmidt, G.; Gosselck, J. *Ibid.* **1969**, 3445; Gosselck, J.; Beress, L.; Schenk, H. *Angew. Chem.* **1966**, *78*, 606; Takaki, K.; Agawa, T. *J. Org. Chem.* **1977**, *42*, 3303; Takaki, K.; Neguro, K.; Agawa, T. *J. Chem. Soc., Perkin Trans. 1* **1979**, 1490.

(15) Johnson, C. R.; Lockard, J. P. *Tetrahedron Lett.* **1971**, 4589.

(16) For the cyclopropyl ketone synthesis via methylene transfer to α,β-unsaturated ketones by Simmons–Smith type reaction, see: Simmons, H. E.; Cairns, T. L.; Vladuchick, S. A.; Hoiness, C. M. *Org. React.* **1973**, *20*, 1. By oxosulfonium ylide: Corey, E. J.; Chaykovsky, M. *J. Am. Chem. Soc.* **1965**, *87*, 1353. By selenonium ylide: Lotz, W. W.; Gosselck, J. *Tetrahedron* **1973**, *29*, 917.

(17) Prepared by treatment of 3-((*p*-chlorophenyl)seleno)-1-propene oxide with sodium hydride in THF followed by benzylation.

offers a simple and useful approach to cyclopropyl carbonyl compounds.

We are currently investigating the application of the present reaction to the synthesis of natural products possessing cyclopropane rings.

Acknowledgment. This work was supported in parts by grants from the Ministry of Education (45148) of the Japanese Government and from the Kurata Foundation.

Registry No. 1 (R^1 = Ph; R^2 = H), 54533-00-5; **i** (R^1 = Ph; R^2 = CH_3), 70887-62-6; **1** (R^1 = C_2H_5O; R^2 = C_6H_5S), 73971-36-5; **1** (R^1, R^2 = $(CH_2)_{10}$), 73971-37-6; **1** (R^1 = $(CH_3)_3C$; R^2 = H), 70367-67-8; **1** (R^1, R^2 = $(CH_2)_4$), 56528-89-3; **1** (R^1 = $(CH_3)_2CH$; R^2 = H), 70367-68-9; **1** (R^1 = C_5H_{11}; R^2 = C_6H_5S), 73971-38-7; **1** (R^1 = C_2H_5O; R^2 = C_6H_5SO), 73971-39-8; **1** (R^1 = C_2H_5O; R^2 = $C_6H_5SO_2$), 73971-40-1; **1** (R^1 = C_2H_5O; R^2 = C_2H_5O-CO), 34727-00-9; **2** (R^3 = $C_{10}H_{21}$; Ar = Ph), 73971-41-2; **2** (R^3 = $C_{10}H_{21}$; Ar = o-$CH_3C_6H_4$), 73971-42-3; **2** (R^3 = $C_{10}H_{21}$; Ar = m-$CF_3C_6H_4$), 73971-43-4; **2** (R^3 = $C_{10}H_{21}$; Ar = o-$NO_2C_6H_4$), 73971-44-5; **2** (R^3 = H; Ar = p-ClC_6H_4), 73971-45-6; **2** (R^3 = $C_6H_5CH_2OCH_2$; Ar = p-ClC_6H_4), 73971-46-7; **2a**, 73971-47-8; **3** (R^1 = Ph; R^2 = H; R^3 = $C_{10}H_{21}$), 73985-89-4; **3** (R^1 = Ph; R^2 = H; R^3 = H), 3481-02-5; **3** (R^1 = Ph; R^2 = CH_3; R^3 = H), 26921-44-8; **3** (R^1 = C_2H_5O; R^2 = C_6H_5S; R^3 = H), 73971-48-9; **3** (R^1 = Ph; R^2 = H; R^3 = $C_6H_5CH_2OCH_2$), 73985-90-7; **3** (R^1 = Ph; R^2 = CH_3; R^3 = $C_6H_5CH_2OCH_2$), 73971-49-0; **3** (R^1 = C_2H_5O; R^2 = C_6H_5S; R^3 = $C_6H_5CH_2OCH_2$), 73971-50-3; **3** (R^1, R^2 = $(CH_2)_{10}$; R^3 = $C_6H_5CH_2OCH_2$), 73971-51-4; **3** (R^1 = Ph; R^2 = CH_3; R^3 = $C_{10}H_{21}$), 73971-52-5; **3** (R^1 = C_2H_5O; R^2 = C_6H_5S; R^3 = $C_{10}H_{21}$), 73971-53-6; **3** (R^1 = $(CH_3)_3C$; R^2 = H; R^3 = $C_{10}H_{21}$), 73971-54-7; **3** (R^1, R^2 = $(CH_2)_4$; R^3 = $C_{10}H_{21}$), 73971-55-8; **3** (R^1 = $(CH_3)_2CH$; R^2 = H; R^3 = $C_{10}H_{21}$), 73971-56-9; **3** (R^1 = C_5H_{11}; R^2 = C_6H_5S; R^3 = $C_{10}H_{21}$), 73971-57-0; **3** (R^1 = C_2H_5O; R^2 = C_6H_5SO; R^3 = $C_{10}H_{21}$), 73971-58-1; **3** (R^1 = C_5H_5O; R^2 = $C_6H_5SO_2$; R^3 = $C_{10}H_{21}$), 73971-59-2; **3** (R^1 = C_2H_5O; R^2 = C_2H_5OCO; R^3 = $C_{10}H_{21}$), 73971-60-5; **4**, 73971-61-6; camphor, 76-22-2; β-ionone, 14901-07-6; cyclohexenone, 930-68-7; 4,7,7-trimethylspiro[bicyclo[2.2.1]heptane-2,1′-cyclopropan]-3-one, 57761-38-3; 1-[2-[(phenylmethoxy)methyl]cyclopropyl]-3-(2,6,6-trimethyl-1-cyclohexen-1-yl-2-propen-1-one, 73971-62-7; 1-[(phenylmethoxy)methyl]spiro[2.5]oct-5-en-4-one, 73971-63-8.

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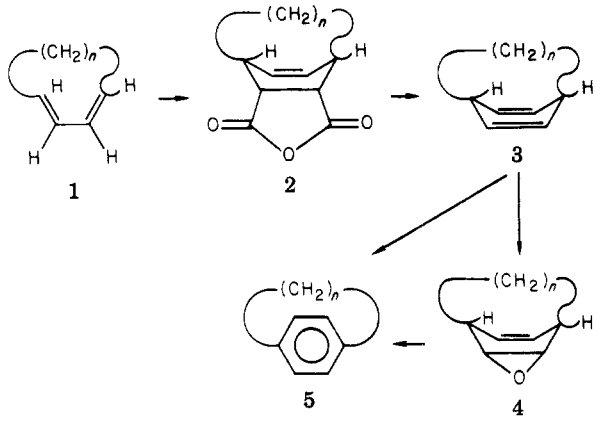
Received December 27, 1979

A General Approach to Small [*n*]Paracyclophanes

Summary: A general procedure has been developed for the synthesis of both substituted and unsubstituted [*n*]-paracyclophanes, where *n* equals 7 or 8.

Sir: There has been considerable recent interest in the synthesis[1] and properties[2] of [*n*]paracyclophanes where *n* equals 6,[1a] 7,[1b,c] and 8.[1d,2] Most of this interest has centered on the distortion of the aromatic ring and the effect of this distortion on both the physical and chemical properties of this ring. The currently available methods for the synthesis of the smaller of these systems do not readily lend themselves to the preparation of aryl-substituted versions of these compounds. We now report a general synthesis of both substituted and unsubstituted versions of [7]paracyclophane and [8]paracyclophane. Our synthetic process has its origin in our work on (i,o)-bicyclo[*n*.2.2]alkadienes.[3]

In analogy with our earlier work,[3] we added maleic anhydride to *cis,trans*-1,3-cyclododecadiene (**1a**) and to *cis,trans*-1,3-cycloundecadiene (**1b**) to give the corresponding Diels–Alder adducts, **2a** and **2b**, in 21% and 23% yields, respectively.[4] Refluxing of **2a** in 10% aqueous



a, n = 8; **b**, n = 7

tetrahydrofuran gave a 98% yield of the corresponding diacid which was subjected to lead tetraacetate oxidation in toluene–pyridine to yield 22% **3a**.[5] The direct electrochemical decarboxylation of **2a** to **3a** was accomplished in 39% yield. Treatment of **3a** with 1 equiv of *m*-chloroperbenzoic acid gave an 80% yield of **4a** as a waxy solid, mp 39–41 °C. This epoxide was extremely labile and underwent slow conversion to **5a**, even in base-washed glassware. Exposure of **4a** to hydrochloric acid gave a 93% yield of [8]paracyclophane (**5a**), which was identical in all respects with the literature compound.[1d,6]

The observation that **5a** was present as an impurity in the oxidative decarboxylation of **2a** suggested that **3a** might be directly oxidized to give **5a**. Heating of **3a** with 1.5 equiv of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone at 95 °C for 12 days gave a 79% yield of **5a**.[7]

In a similar series of experiments, **2b** was hydrolyzed to the corresponding diacid in 98% yield. Lead tetraacetate decarboxylation gave 12% **3b**, while electrochemical oxidative decarboxylation produced 17% **3b**. Epoxidation of **3b** with *m*-chloroperbenzoic acid gave 98% **4b**, which on treatment with catalytic amounts of trifluoroacetic acid gave [7]paracyclophane (**5b**) in 88% yield. This material was spectroscopically identical with that previously reported.[1b]

We had previously reported the synthesis of both **6a** and **6b**[3] via the addition of perfluoro-2-butyne to the appro-

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(2) M. G. Newton, T. J. Walter, and N. L. Allinger, *J. Am. Chem. Soc.*, **95**, 5652 (1973); N. L. Allinger, J. T. Sprague, and T. Liljefors, *ibid.*, **96**, 5100 (1974); S. L. Kammula, L. D. Iroff, M. Jones, Jr., J. W. van Straten, W. H. de Wolf, and F. Bickelhaupt, *ibid.*, **99**, 5815 (1977); H. Schmidt, A. Schweig, W. Thiel, and M. Jones, Jr., *Chem. Ber.*, 111, 1958 (1978).

(3) P. G. Gassman, S. R. Korn, and R. P. Thummel, *J. Am. Chem. Soc.*, **96**, 6948 (1974); P. G. Gassman, S. R. Korn, T. F. Bailey, T. H. Johnson, J. Finer, and J. Clardy, *Tetrahedron Lett.*, 3401 (1979).

(4) Satisfactory elemental analysis and/or exact-mass molecular weights were obtained on all new compounds.

(5) In addition to **3a**, **5a** was formed as a 2% byproduct.

(6) Surprisingly, treatment of **4a** with *n*-butyllithium also produced **5a** (48% yield). The mechanism of this process is unknown.

(7) Tetracyanoethylene was also effective in converting **3a** into **5a** (49% yield).

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