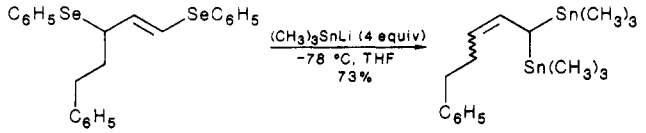
ratio obtained in the first reaction was equilibrated to a 75/25 mixture on treatment with (trimethylstannyl)lithium at 25 °C, indicating that the *E* isomer was the more stable product.

In the above example, and in all other cases we have examined, the *regiochemical* preference was for the isomer with the tin at the less substituted end of the allyl or allenyl–propargyl fragment largely independent of the starting position of the phenylseleno group. The *stereochemistry* was kinetically controlled, provided that the reaction was quenched at low temperature.

We have not determined whether the regiochemistry observed at low temperature is the result of kinetic or thermodynamic factors, since in no case was the less stable regioisomer obtained. We have seen no indications of an induction period for the reaction (it is usually over within minutes at −78 °C). Single-electron transfer as well as S_N2 mechanisms has been proposed for the reaction of stannyllithium reagents with allyl halides and tosylates.[11]

Allyl selenides containing acetoxy and benzoyloxy groups did not react cleanly, but compounds containing cyano, *tert*-butyldimethylsiloxy, amino,[1a] and nonactivated phenylseleno groups gave the desired products. The alkylated 1,3-bis(phenylseleno)propene[1d] **9** gave bisstannylated product **10** when excess lithium reagent was used. Apparently the first substitution proceeded as usual to give a trimethylstannylated allyl selenide, which then reacted a second time.



9 **10**, *E*/*Z* = 3/2

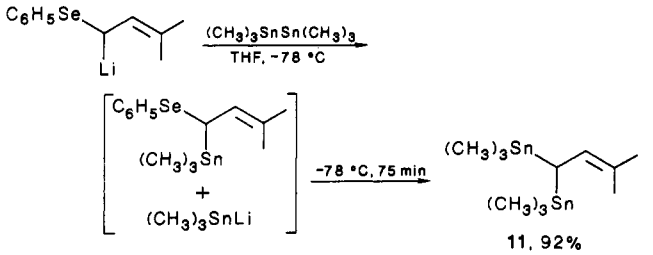
Allenyl and propargyl selenides work well in the reaction, giving the less substituted of the two possible isomers.

Two 1,1-bis(phenylseleno)alkanes were treated with (trimethylstannyl)lithium. Each gave cleanly the product of monosubstitution. Even under forcing conditions no 1,1-bis(trimethylstannyl) product was detected in any of these cases. In the 4-*tert*-butylcyclohexane system only the axial trimethylstannyl compound was formed. Stereochemistry was determined from the characteristic ^{77}Se chemical shifts of axial and equatorial seleno groups.

Sulfides[12] and sulfones will also react with tin lithium reagents, but much slower than do the selenides. Thus benzyl phenyl sulfide and sulfone reacted slowly with (trimethylstannyl)lithium at room temperature (≈40% reaction after several hours at 25 °C), and (phenylseleno)(phenylthio)methane gave exclusively the product of selenium substitution (<0.3% of $PhSeCH_2SnMe_3$ was formed).

Hexamethyldistannane is reactive toward nucleophiles.[13] We have taken advantage of this reactivity to achieve the preparation of 1,1-bis(trimethylstannyl) compounds using selenides as starting materials. The lithioallyl selenide **1b**, prepared by using lithium diisopropylamide, was treated with hexamethyldistannane to give a 92% yield of **11**.[14]

The course of the reaction probably involves the "counterattacking" sequence shown.[15]



11, 92%

The procedures described here make available a variety of trimethylstannyl compounds from conveniently available precursors.

Acknowledgment. We thank the National Science Foundation and PPG Industries for support of this work.

Registry No. 1a, 74472-76-7; **1b**, 56529-39-6; **2**, 56253-58-8; **3**, 112042-24-7; **4**, 61713-47-1; **5**, 112042-06-5; **7**-(*E*), 112042-12-3; **7**-(*Z*), 112042-19-0; **8**, 17417-78-6; **9**, 112042-21-4; **10**-(*E*), 112042-22-5; **10**-(*Z*), 112042-23-6; **11**, 86309-32-2; $BrCH_2CH_2CH_2C_6H_5$, 637-59-2; $BrCH_2C_6H_5$, 100-39-0; $BrCH_2CH{=}CHC_6H_5$, 4392-24-9; $H_2C(O)$, 50-00-0; $C_6H_5SeCH(CH{=}CH_2)(CH_2CH_2CH_2C_6H_5)$, 112042-05-4; $C_6H_5CH(SeC_6H_5)(CH_2CH{=}CHC_6H_5)$, 112042-07-6; $C_6H_5SeCH(CH{=}CH_2)[(CH_2CH(CH_3)(OSiMe_2$-*t*-Bu)], 112042-08-7; $C_6H_5SeC(CH_2C_6H_5){=}C{=}CH_2$, 112042-09-8; $C_6H_5SeCH(C{\equiv}CCH_3)(CH_2C_6H_5)$, 112042-10-1; $C_6H_5SeCH_2SeC_6H_5$, 20343-90-2; (*E*)-$C_6H_5CH_2CH_2CH_2CH{=}CHCH_2Sn(CH_3)_3$, 112042-11-2; (*Z*)-$C_6H_5CH_2CH_2CH_2CH{=}CHCH_2Sn(CH_3)_3$, 112042-18-9; $C_6H_5CH{=}CHCH_2CH(C_6H_5)[Sn(CH_3)_3]$, 112042-13-4; (*E*)-$(CH_3)_3SnCH_2CH{=}CHCH_2CH(CH_3)(OSiMe_2$-*t*-Bu), 112042-14-5; (*Z*)-$(CH_3)_3SnCH_2CH{=}CHCH_2CH(CH_3)(OSiMe_2$-*t*-Bu), 112042-20-3; $C_6H_5CH_2C{\equiv}CCH_2Sn(CH_3)_3$, 112042-15-6; $C_6H_5CH_2CH{=}C{=}C(CH_3)[(Sn(CH_3)_3]$, 112042-16-7; $C_6H_5SeCH_2Sn(CH_3)_3$, 103596-69-6; $(CH_3)_3SnSn(CH_3)_3$, 661-69-8; benzeneselenol, 645-96-5; 3-bromo-1-cyclohexene, 1521-51-3; methyloxirane, 75-56-9; 4-*tert*-butylcyclohexanone, 98-53-3; 3-phenylselenylcyclohexene, 83442-20-0; 1,1-bis(phenylselenyl)-4-*tert*-butylcyclohexane, 71518-68-8; 3-(trimethylstannyl)cyclohexene, 17314-43-1; *cis*-1-(phenylselenyl)-4-*tert*-butyl-1-(trimethylstannyl)cyclohexane, 112042-17-8.

(11) Wickham, G.; Young, D.; Kitching, W. *J. Org. Chem.* **1982**, *47*, 4884. Dumartin, G.; Quintard, J. P.; Pereyre, M. *J. Organomet. Chem.* **1980**, *185*, C34. Ashby, E. C.; Pham, T. N. *Tetrahedron Lett.* **1987**, *28*(28), 3183.

(12) Takeda, T.; Ando, K.; Mamada, A.; Fujiwara, T. *Chem. Lett.* **1985**, 1149.

(13) For example, only a single NMR resonance is seen for mixtures of Me_6Sn_2 and Me_3SnLi: Kobayashi, K.; Kawanisi, M.; Kozima, S.; Hitomi, T.; Iwamura, H.; Sugawara, T. *J. Organomet. Chem.* **1981**, *217*, 315.

(14) Under similar conditions, benzyl chloride gives ≈50% yield of bis(trimethylstannyl)phenylmethane.

(15) Hwu, J. R., and Wetzel, J. M. (*J. Org. Chem.* **1985**, *50*, 400), have reported the utilization of hexamethyldisilane as a deoxygenating agent by a pathway involving generation of trimethylsilyl anion by reaction of substrate with the disilane, followed by a second reaction of the anion with the silylated substrate.

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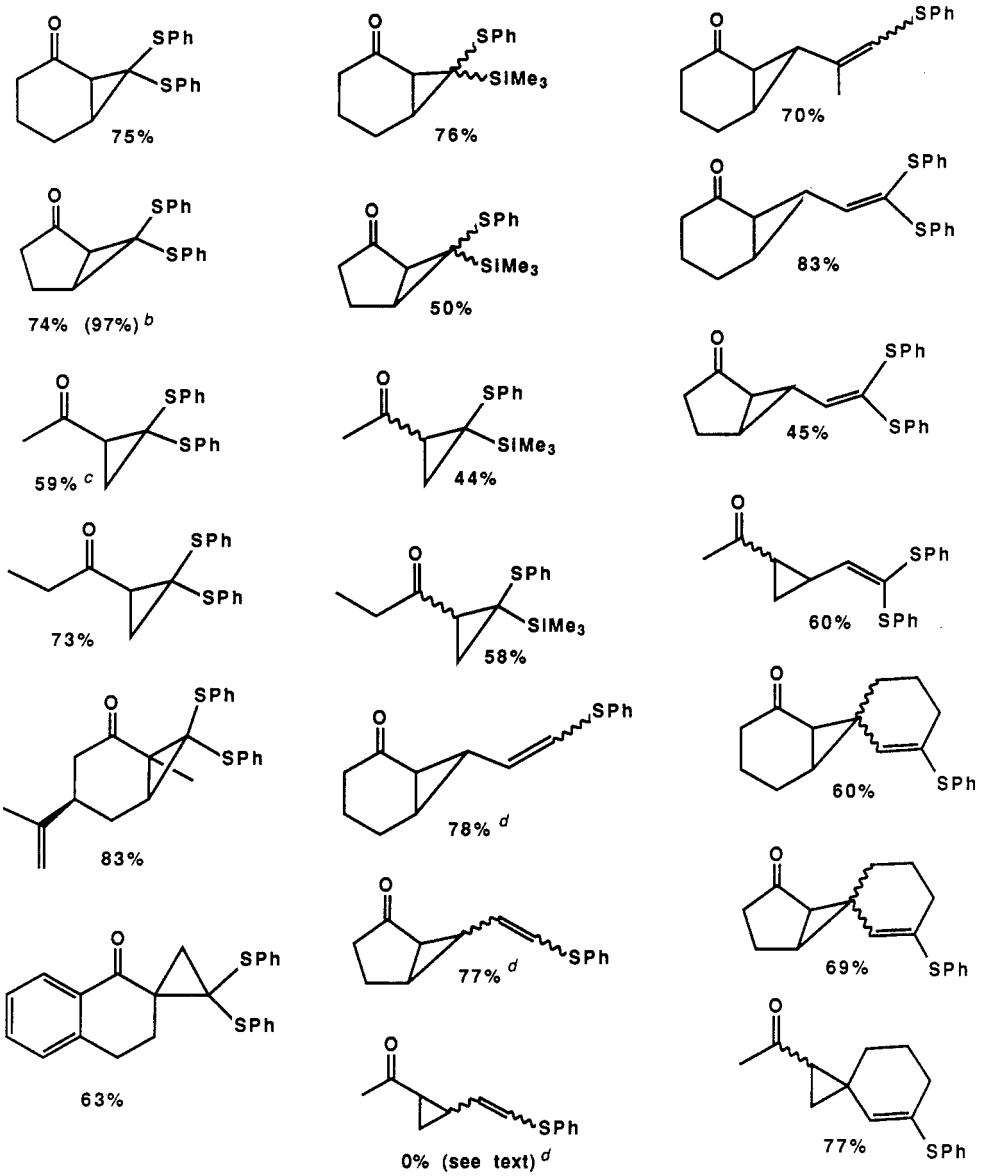
A Novel One-Flask Cyclopropanation and Vinylcyclopropanation Method for α-Enones. Copper(I)-Induced γ-Thiophenoxide Removal from Conjugate Adducts

Summary: A novel concept for performing a one-flask cyclopropanation of conjugated enones consists of conjugate addition of an appropriate phenylthio-stabilized organolithium compound followed by treatment of the resulting enolate anion with cuprous trifluoromethanesulfonate. The cyclopropanes generated by this procedure can be simple ones, substituted for example by silyl- and

0022-3263/88/1953-0457$01.50/0

Table I. Yields and Products of Cyclopropanation of Enones[a]



[a] Conjugate additions performed at −78 °C unless otherwise indicated. [b] Based on consumed tris(phenylthio)methane; some enolization occurred. [c] Reference 15. [d] Additions performed at −100 °C.

sulfur-bearing groups, or they can be vinylcyclopropanes.

Sir: Cyclopropyl ketones are versatile synthetic intermediates.[2] We now reveal a new concept that allows preparation of these substances by a one-flask cyclopropanation of α-enones. Most present procedures for performing the latter transformation are variations of the concept depicted in eq 1.[3] The most common leaving group is a thioether (Y = R′R″S$^+$) or derivative[3a] but halides and other groups[3b] are sometimes used. Although widely employed, this procedure suffers from the difficulty or impossibility of preparing many interestingly functionalized anions or ylides 1 and from the fact that some ylides are too nonnucleophilic to execute the addition step efficiently and are thus subject to carbene formation.

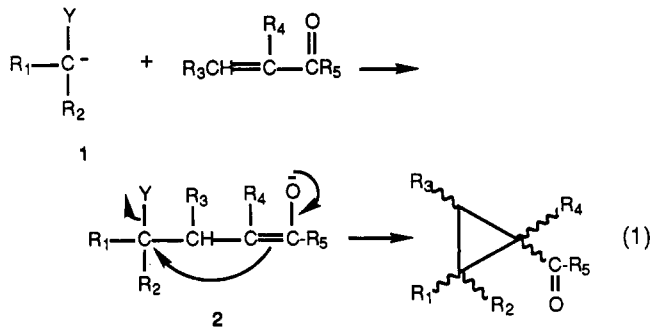
Our procedure avoids these problems by utilizing stable, highly nucleophilic carbanions 1 exhibiting a strong tendency to undergo conjugate rather than 1,2-addition[4] and in which Y is a poor leaving group *until activated by a Lewis acid* which is added after formation of the enolate

(1) Taken in part from the Ph.D. Thesis of Michael Myers, Department of Chemistry, University of Pittsburgh, 1987.

(2) (a) Staley, S. W. In *Selective Organic Transformations*; Thyagarajan, B. S., Ed.; Wiley: New York, 1972. Ochiai, M.; Sumi, K.; Fujita, E. *Chem. Pharm. Bull.* **1983**, *31*, 3931. Attah-Poku, S. K.; Antczak, K.; Alward, S. J.; Fallis, A. G. *Can. J. Chem.* **1984**, *62*, 1717. Kulinkovich, O. G.; Tishchenko, I. G.; Roslik, N. A.; Reznikov, I. V. *Synthesis* **1983**, 383. Giacomini, E.; Loreto, M. A.; Pellacani, L.; Tardella, P. A. *J. Org. Chem.* **1980**, *45*, 519. Blake, K. W.; Gillies, I.; Denney, R. C. *J. Chem. Soc., Perkin Trans. 1* **1981**, 700. Demuth, M.; Raghavan, P. R. *Helv. Chim. Acta* **1979**, *62*, 2338. Donnelly, J. A.; Macken, P. J.; O'Brien, S. *Israel J. Chem.* **1981**, *21*, 185. Dieter, K. R.; Pounds, S. *J. Org. Chem.* **1982**, *47*, 3174. Miller, R. D.; McKean, D. R. *J. Org. Chem.* **1981**, *46*, 2412. (b) Hudlicky, T.; Kutchan, T. M.; Naqvi, S. M. *Org. React. (N.Y.)* **1985**, *33*, 248. (c) Corey, E. J.; Myers, A. G. *J. Am. Chem. Soc.* **1985**, *107*, 5574.

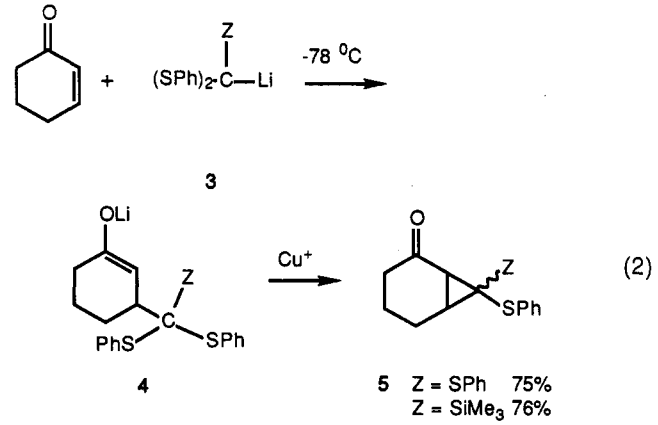
(3) (a) Corey, E. J.; Chaykovsky, M. *J. Am. Chem. Soc.* **1965**, *87*, 1353. Johnson, C. R. In *Comprehensive Organic Chemistry*; Jones, D. N., Ed.; Pergamon Press: New York, 1979; Vol. 3; Part 11.15, p 247. (b) Hudlicky, T.; Radesca, L.; Luna, H.; Anderson, F. E. *J. Org. Chem.* **1986**, *51*, 4746 and references therein. Cory, R. M.; Renneboog, R. M. *J. Org. Chem.* **1984**, *49*, 3898. (c) In a less general method, the leaving group is attached not to the nucleophile but to the γ-position of the enone: Cooke, M. P., Jr.; Jaw, J. Y. *J. Org. Chem.* **1986**, *51*, 758 and references therein.

(4) Myers, M.; Cohen, T., manuscript in preparation. Cohen, T.; Abraham, W. D.; Myers, M. *J. Am. Chem. Soc.*, in press.



(1)

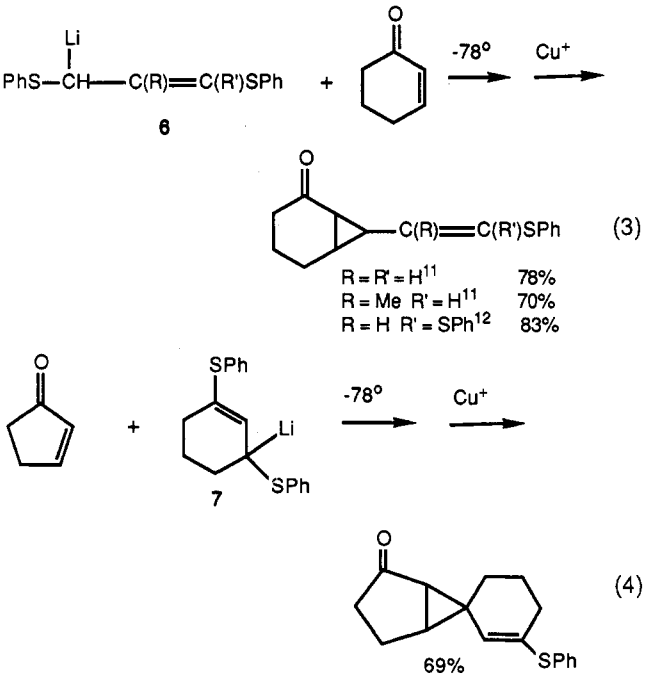
2. The thiophenoxy group, PhS, serves admirably as the masked leaving group which can be readily activated by the addition of the cuprous triflate–benzene complex,[5] a specific Lewis acid toward divalent sulfur.[6-8] As in earlier work,[5,6] the cuprous triflate is capable of removing thiophenoxide ion from a carbon atom which is attached to a carbocation-stabilizing group such as a second phenylthio group or a vinylogous one (-C(R¹)=C(R²)SPh or -C(R)=C(SPh)₂). Two examples of simple (unconjugated) cyclopropyl ketone formation are shown in eq 2.



(2)

A major impetus for this study was the development of methods for producing 1-(phenylthio)-1-(trimethylsilyl)-cyclopropanes which are substrates for production of alkylidene- and allylidenecyclopropanes by a reductive lithiation–Peterson olefination sequence.[9] Indeed, the present procedure bears an interesting relationship to another which we had devised to prepare such compounds in which the olefin acceptor is a weaker electrophile and the carbanionic site on the adduct is such a powerful nucleophile that the thiophenoxide is displaced without activation.[10]

Vinylcyclopropanes are valuable substrates for the preparation of cyclopentenes via the vinylcyclopropane rearrangement, a reaction which has been applied to vinylcyclopropyl ketones.[2b] This class of cyclopropanes has not been generally available by the sulfur ylide method.[3a] A number of usefully functionalized compounds of this type can be prepared by the present procedure. Three examples are shown in eq 3. When the allylic sulfur stabilized anion is in a ring (e.g. 7),[11] a spiro vinylcyclopropane results (e.g. eq 4).



(3)

(4)

The cyclopropyl ketones produced in this manner are shown in Table I.[13,14] As indicated elsewhere,[4] the conjugate addition step at low temperature was successful in all cases. In the case of 4 (Z = SPh) and analogues, closure occurred at −78 °C whereas −23 °C was required with 4 (Z = $SiMe_3$) and analogues, a reflection of the far greater carbocation stabilizing ability of S(II) than of Si(IV). In

(5) Cohen, T.; Ruffner, R. J.; Shull, D. W.; Fogel, E. R.; Falck, J. R. *Org. Synth.* **1980**, *59*, 202.

(6) Cohen, T.; Herman, G.; Falck, J. R.; Mura, A. J., Jr. *J. Org. Chem.* **1975**, *40*, 812. Cohen, T.; Kuhn, D.; Falck, J. R. *J. Am. Chem. Soc.* **1975**, *97*, 4749.

(7) Semmelhack, M. F.; Tomesch, J. C. *J. Org. Chem.* **1977**, *42*, 2657. Knapp, S.; Trope, A. F.; Theodore, M. S.; Hirata, N.; Barchi, J. J. *J. Org. Chem.* **1984**, *49*, 608. Knapp, S.; Trope, A. F.; Ornaff, R. M. *Tetrahedron Lett.* **1980**, *21*, 4301. Cohen, T.; Mura, A. J., Jr.; Shull, D. W.; Fogel, E. R.; Ruffner, R. J.; Falck, J. R. *J. Org. Chem.* **1976**, *41*, 3218. Masamune, S.; Hirama, M.; Mori, S.; Ali, S. A.; Garvey, D. S. *J. Am. Chem. Soc.* **1981**, *103*, 1568. Cohen, T.; Gapinski, R. E.; Hutchins, R. R. *J. Org. Chem.* **1979**, *44*, 3599. Cohen, T.; Weisenfeld, R. B.; Gapinski, R. E. *J. Org. Chem.* **1979**, *44*, 4744. Kozikowski, A. P.; Ames, A. *J. Am. Chem. Soc.* **1980**, *102*, 860. See also: Kozikowski, A. P. *Tetrahedron* **1985**, *41*, 4821.

(8) Beak has recently used cuprous bromide to remove thiophenoxide ion from β-lithio phenylthio ethers. Beak, P.; Wilson, K. D. *J. Org. Chem.* **1987**, *52*, 218.

(9) Cohen, T.; Sherbine, J. P.; Matz, J. R.; Hutchins, R. R.; McHenry, B. M.; Willey, P. R. *J. Am. Chem. Soc.* **1984**, *106*, 3245. Cohen, T.; Jung, S.-H.; Romberger, M. L.; McCullough, D. W. *Tetrahedron Lett.*, in press.

(10) Cohen, T.; Sherbine, J. P.; Mendelson, S. A.; Myers, M. *Tetrahedron Lett.* **1985**, *26*, 2965. See also: Schaumann, E.; Friese, C.; Spanka, C. *Synthesis* **1986**, 1035.

(11) Cohen, T.; Bennett, D. A.; Mura, A. J., Jr. *J. Org. Chem.* **1976**, *41*, 2506.

(12) Dziadulewicz, E.; Gallagher, T. *Tetrahedron Lett.* **1985**, *26*, 4547.

(13) Yields are for isolated material. All new compounds have been characterized by ^1H NMR and IR spectroscopy and by high resolution mass spectrometry. X-ray crystal structure analysis was used to characterize 5 (Z = SPh).

(14) A typical procedure: To a three-necked 100-mL reaction flask fitted with an argon inlet and a septum is added 1.54 g (4.53 mmol) of tris(phenylthio)methane in 20 mL of THF at −78 °C followed by addition of 3.36 mL of *n*-BuLi (4.53 mmol). The yellow solution is stirred at −78 °C for 30 min at which time cyclohexenone (0.43 mL, 4.53 mmol) is added dropwise. The solution is stirred until disappearance of the starting materials is indicated by TLC (ca. 30 min). Cuprous triflate (2.15 g, 4.27 mmol), which has been prepared previously[5] and stored under argon in a sealed ampule, is transferred under argon to a solid addition tube which is then attached to the reaction vessel. The CuOTf is added to the reaction vessel in small portions over 1 h and the resulting brown homogeneous mixture is allowed to stir for an additional 2 h at −78 °C (or the temperature indicated for the type of cyclopropane desired). The reaction mixture is quenched with ca. 4 mL of a 5% NaOH solution and allowed to warm to room temperature. The resulting orange slurry is poured on to an ether/silica gel column and eluted rapidly with ether. The solvents are removed in vacuo to afford a brown oil which crystallizes upon standing. An alternative workup procedure was developed for the extremely acid-sensitive spirocyclopropanes and can be applied to all other syntheses: The reaction mixture is quenched with small equal amounts of saturated NH_4Cl and 5% NaOH. The resulting slurry is poured on to a layered pad of $MgSO_4$ and Florisil in a fritted funnel and is eluted with ether by rapid vacuum filtration. The solvent is removed in vacuo and the oil is immediately subjected to flash chromatography.

(15) Kulinkovich, O. G.; Tishchenko, I. G.; Reznikov, I. V.; Roslik, N. A. *J. Org. Chem. USSR* **1982**, *18*, 1446.

all of the other cases, the mixture was warmed to ambient over a 3-h period after the addition of cuprous triflate at the temperature at which conjugate addition occurred. Conversion of the conjugate adducts to cyclopropanes was efficient except in the case of the adduct of **6** (R = R′ = H) with methyl vinyl ketone which, even in the presence of excess cuprous salt, gave an inseparable mixture containing substantial conjugate addition product; we suspect that the latter results from protonation of the enolate by the acidic 1,2-hydride transfer product that is formed in lieu of ring-closure product. In a few cases lowered yields resulted from the instability of the products to silica gel chromatography.

We are now studying the synthetic utility of a number of the products in Table I. The concept enunciated herein should be extendable to some other electrophilic olefins, nucleophiles, leaving groups, and Lewis acids.

Acknowledgment. We thank the National Institutes of Health for financial support, Dr. Alvin Marcus for recording the mass spectra, and Dr. Jaime Abola for the X-ray crystal structure of **5** (Z = SPh).

Registry No. **5** (Z = SPh), 112022-43-2; **5** (Z = $SiMe_3$), 112022-44-3; **6** (R = Me, R^1 = H), 112022-59-0; **6** (R = H, R′ = SPh), 112022-60-3; **6** (R = R′ = H), 111742-81-5; **7**, 101185-20-0; 7-(1-methyl-2-(phenylthio)ethenyl)bicyclo[4.1.0]heptan-2-one, 112041-64-2; 6,6-bis(phenylthio)bicyclo[3.1.0]hexan-2-one, 112022-45-4; 6-(phenylthio)-6-(trimethylsilyl)bicyclo[3.1.0]hexan-2-one, 112022-46-5; 7-[2,2-bis(phenylthio)ethenyl]bicyclo[4.1.0]heptan-2-one, 112022-47-6; 1-acetyl-2-(phenylthio)-2-(trimethylsilyl)cyclopropane, 112022-48-7; 6-[2,2-bis(phenylthio)ethenyl]bicyclo[3.1.0]hexan-2-one, 112022-49-8; 2,2-bis(phenylthio)-1-propionylcyclopropane, 83300-59-8; 2-(phenylthio)-1-propionyl-2-(trimethylsilyl)cyclopropane, 112022-50-1; 1-[2,2-bis(phenylthio)ethenyl]-2-acetylcyclopropane, 112022-51-2; 1-methyl-7,7-bis(phenylthio)-4-(propen-2-yl)bicyclo[4.1.0]heptan-2-one (1A,4B,6A), 112022-52-3; 7-[2-(phenylthio)ethenyl]bicyclo[4.1.0]heptan-2-one, 112022-53-4; 2′,3′-didehydro-3′-(phenylthio)spiro[bicyclo[4.1.0]heptane-7,1′-cyclohexan-2-one], 112022-54-5; 3′,4′-dihydro-1′-oxo-2,2-di(phenylthio)spiro[cyclopropane-1,2′(1′*H*)-naphthalene], 112022-55-6; 6-[2-(phenylthio)ethenyl]-bicyclo[3.1.0]hexan-2-one, 112022-56-7; 2′,3′-didehydro-3′-(phenylthio)-2-oxospiro[bicyclo[3.1.0]hexane-6,1′-cyclohexane], 112022-57-8; 4,5-didehydro-5-(phenylthio)-1-acetylspiro[2.5]octane, 112022-58-9; cuprous triflate, 42152-44-3; tris(phenylthio)methane, 4832-52-4; [bis(phenylthio)methyl]trimethylsilane, 37891-39-7; cyclohexenone, 930-68-7; 2-cyclopentenone, 930-30-3; 3-buten-2-one, 78-94-4; 1-penten-2-one, 1629-58-9; 2-methyl-5-(propen-2-yl)cyclohex-2-en-1-one, 99-49-0; 3′,4′-dihydro-1′-oxo-2,2-bis(phenylthio)spiro[cyclopropane-1,2′(1′*H*)-naphthalene], 112022-55-6.

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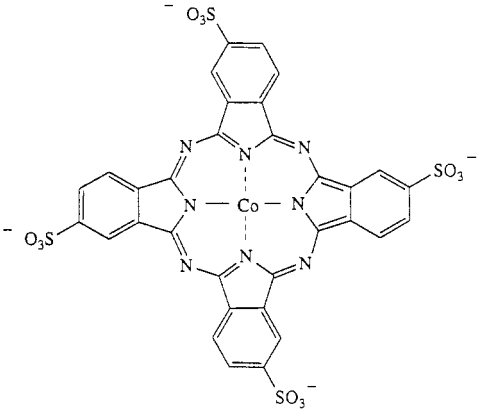
Autoxidation of 2,6-Di-*tert*-butylphenol in Water Catalyzed by Cobalt Phthalocyaninetetrasulfonate Bound to Polymer Colloids

Summary: Cobalt phthalocyaninetetrasulfonate (CoPcTs) bound to 54–74-nm styrene–divinylbenzene copolymer latexes substituted with quaternary ammonium ions catalyzes autoxidation of 2,6-di-*tert*-butylphenol in water ten times faster than does CoPcTs in aqueous solution.

Sir: The oxidation of organic compounds is crucial for treatment of waste water. We have found an efficient autoxidation of 2,6-di-*tert*-butylphenol in water catalyzed by cobalt phthalocyaninetetrasulfonate (**1**, CoPcTs, a mixture of isomers) bound to colloidal ion exchange resins. Previously autoxidations of 2,6-disubstituted phenols in organic solvents have produced mainly the 2,6-disubstituted 1,4-benzoquinone and the 3,5,3′,5′-tetrasubstituted 4,4′-diphenoquinone with Co catalysts[1] and certain copper catalysts.[2] CoPc derivatives have been bound to several other types of polymers for catalysis.[3]



1, CoPcTs

The colloidal polymers are prepared by emulsion copolymerization of (chloromethyl)styrene and divinylbenzene.[4] Treatment with trimethylamine converts the chloromethyl groups to quaternary ammonium chlorides.[4] The divinylbenzene prevents dissolution of the polymer in water as a polyelectrolyte. Addition of an aqueous solution of tetrasodium cobalt phthalocyaninetetrasulfonate[5] to the colloidal ion-exchange resins results in complete binding of the CoPcTs, as long as the amount of sulfonate ions of **1** does not exceed the amount of quaternary ammonium ions of the polymer. Ultrafiltration of the colloids through 0.1-μm cellulose acetate/nitrate membranes revealed no blue CoPcTs in the filtrates. The intensely blue CoPc in water is detectable by human eye to concentrations of $<10^{-7}$ M, which then is an upper limit to the concentration of CoPcTs in the aqueous phase of the heterogeneous catalysts listed in Table I.

Oxidations of 2,6-di-*tert*-butylphenol (**2**) were performed in vigorously mixed aqueous suspensions under 0.9 atm of pressure of dioxygen in flasks connected to a gas buret. The catalyst suspension was kept under air 1 h before use. With all colloidal CoPcTs catalysts except C-9 (Table I)

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