The addition of sodium superoxide ($NaO_2$) to molten LiF–NaF–KF yields a yellow solution. The spectrum of this solution consists of an absorption peak at 372 nm and a shoulder at 260 nm, which is on the side of an absorption edge in the ultraviolet region. The 372-nm peak agrees with the spectrum ascribed to superoxide ion in liquid ammonia by Thompson and Kleinberg.[1] Diffuse reflectance spectra of $NaO_2$ reported by Griffiths, Lott, and Symons[2] also place the superoxide absorbance in the region of 370 nm. The 260-nm shoulder was not reported in either investigation.

The superoxide ion can be generated by the addition of either of the fluorinating agents $MnF_3$ or $CoF_3$ to the molten solvent which contains oxide ion.[3] When $MnF_3$ is added to the melt, a spectrum attributable to Mn(III) is observed in the region of 525 nm. The melt spectrum of Mn(III) is similar but not identical with that reported in other media;[4] differences in the spectra will be described in a future paper. Along with the expected spectrum of Mn(III), the absorption spectrum of $O_2^-$ is noted. The addition of $CoF_3$ to the molten solvent results in no spectral evidence for Co(III); instead the Co(II) spectrum similar to that seen by Young and White[5] is observed along with the 372-nm peak and the 260-nm shoulder. A previous study indicated that Co(III) decomposed under these conditions.[6]

In molten LiF–NaF–KF which contains oxide ion, spectral evidence indicates that $O_2^-$ is formed by the reaction of the melt with elemental oxygen and, further, is generated electrochemically at a platinum anode. Electrochemical evidence for the stability of peroxide in molten LiF–NaF–KF at 600° was reported by Pizzini and Morlotti.[7] Zambonin and Jordan have reported the existence of superoxide in molten nitrate based on electrochemical studies.[8]

The molten solution of $O_2^-$ in LiF–NaF–KF is quite stable if the melt is contained in a silica cell or platinum windowless cell.[9] In the former cell, the solution could be maintained under a pressure of *ca.* 5 $\mu$ for a period of 2 hr with no decrease in the absorbance of the 372 nm peak. The oxidation ability of the solution was demonstrated by the addition of Co metal with a resultant disappearance of the $O_2^-$ spectrum and the appearance of the Co(II) spectrum. The $O_2^-$ ion was found to be unstable in graphite. The absorbance at 372 nm gradually disappeared over a period of several hours probably owing to the formation of volatile CO or $CO_2$.

Based on spectral evidence, the superoxide ion can be generated by the addition of solid $UO_2$ to a solution of Mn(III) in molten LiF–NaF–KF. In this study an excess amount of $MnF_3$ was added to molten LiF–NaF–KF solvent in a graphite cell. Superoxide ion was generated, from oxide impurities in the solvent, and removed by reaction with the container as stated previously. It is interesting to note that the resultant melt at this point was markedly improved in clarity and exhibited only the spectrum of Mn(III). Upon the addition of $UO_2$, the reaction of Mn(III) with $O^{2-}$ supplied by $UO_2$ resulted in the reappearance of the absorption peak at 372 nm. This generation of superoxide ion from oxide ion in the presence of $MnF_3$ could lead to a spectrophotometric method for the determination of oxide directly in molten salts. Further studies of this seemingly unusual solute species are underway to confirm its identity.

The spectral studies described herein were performed in a 1-cm$^2$ quartz cell, a $^3/_8$-in. i.d. graphite captive liquid cell,[9] or a 0.25-in. i.d. platinum captive liquid cell.[9] The captive liquid cells were contained in a vacuum furnace especially designed for spectrophotometric studies of molten salts.[9] A helium atmosphere was maintained within the furnace, and the absorption spectra were obtained with a Cary Model 14-M spectrophotometer. Additions of solute to the molten solvent were made through a metal ball valve. The solutes, as solids, were loaded into a compartment that is an integral part of the valve body; this operation was performed in an inert atmosphere box. The compartment was then sealed to avoid contact with the atmosphere.

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(1) J. K. Thompson and J. Kleinberg, *J. Am. Chem. Soc.*, **73**, 1243 (1951).

(2) T. R. Griffiths, K. A. K. Lott, and M. C. R. Symons, *Anal. Chem.*, **31**, 1338 (1959).

(3) Oxide ion is a common impurity in LiF–NaF–KF. In a silica container, oxide ion will also be formed, in an equilibrium concentration, by the reaction of fluoride ion with $SiO_2$: $4F^- + SiO_2 \rightleftarrows SiF_4 + 2O^{2-}$.

(4) R. Dingle, *Inorg. Chem.*, **4**, 1287 (1965).

(5) J. P. Young and J. C. White, *Anal. Chem.*, **32**, 799 (1960).

(6) Electrochemical studies made by authors.

(7) S. Pizzini and R. Morlotti, *Electrochim. Acta*, **10**, 1033 (1965).

(8) P. G. Zambonin and J. Jordan, *J. Am. Chem. Soc.*, **89**, 6365 (1967).

(9) J. P. Young, *Inorg. Chem.*, **6**, 1486 (1967).

## Perfluoro-1-methylpropenylsilver

*Sir:*

We wish to report the preparation of *trans*-perfluoro-1-methylpropenylsilver, the first example of a perfluorovinylsilver compound, by the nucleophilic addition of silver fluoride to perfluoro-2-butyne.

$$AgF + CF_3C{\equiv}CCF_3 \longrightarrow trans\text{-}CF_3CF{=}C(CF_3)Ag$$

Unlike 1-methylpropenylsilver, which decomposes at room temperature,[1] perfluoro-1-methylpropenylsilver is a thermally stable compound and was isolated by sublimation *in vacuo* at temperatures up to 175°. Its structure was supported by cleavage reactions of the carbon–silver bond and by its absorption spectra and method of preparation.[2] Only the *trans* isomer appeared to be formed. It was found to be soluble in donor-type aprotic solvents, such as ethers and nitriles, and was attacked by oxygen and by water in solution.

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(1) G. M. Whitesides and C. P. Casey, *J. Am. Chem. Soc.*, **88**, 4541 (1966).

(2) The state of aggregation of *trans*-$CF_3CF{=}C(CF_3)Ag$ has not been determined in solution. A peak corresponding to $(CF_3CF{=}CAgCF_3)_4$ was found in the mass spectrum of the crystalline material.

It was alkylated by methyl iodide with the elimination of silver iodide. Other syntheses in which silver halide is eliminated by reaction with metallic and nonmetallic halides have been established by preliminary experiments.

When 0.100 mol of AgF, 0.110 mol of $CF_3C{\equiv}CCF_3$, and 100 ml of $CH_3CN$ were sealed in a glass ampoule and shaken for 36 hr at ~25°, the AgF and $CF_3C{\equiv}CCF_3$ dissolved to form, after standing, a clear supernatant solution and a small amount of black sediment.[3] The clear solution was decanted and distilled at 0.05 mm to yield after removal of the $CH_3CN$ 23.4 g of a white crystalline sublimate, collected largely at a bath temperature of 150°, and 0.4 g of silver-containing residue. During the decantation and distillation 0.026 mol (gas volume) of $CF_3C{\equiv}CCF_3$ contaminated with $CF_3CH{=}CFCF_3$ and $CH_3CN$ was collected in a cold trap. The nmr spectra of $CF_3CF{=}C(CF_3)Ag$ and of its reaction products supported a *trans* structure for the silver compound and the occurrence of the carbon-silver bond cleavage reactions stereospecifically with retention of configuration. Its properties were: in a sealed tube, sintered, 176–177°, melted with some decomposition to yield a silver mirror, ~180°; ir (KBr) 1675 (m), 1634 (w), 1311 (s), 1244 (s), 1192 (s), 1122 (s), 1088 (s, shoulder), 1021 (w), 893 (m), 850 (m), 735 (w) cm$^{-1}$; nmr $^{19}F$ ($CH_3CN$) 50.7 (d, $J$ = 15.1 Hz, $C\mathit{F}_3CAg$), 70.0 (d, $J$ = 12.5 Hz, $CFC\mathit{F}_3$), and 95.1 (m, $J$ = 14.2 Hz, $C\mathit{F}CF_3$) ppm upfield from external $CFCl_3$.[4] Reaction of $CF_3C{\equiv}CCF_3$ with CsF in $CH_3CN$ under the above conditions yielded an intractable solid polymer with the initial formation of $CF_3CF{=}C(CF_3)Cs$; in the presence of water *trans*-$CF_3CH{=}CFCF_3$ was formed. Other reactions also have shown the facile addition of fluoride ion to $CF_3C{\equiv}CCF_3$.

Perfluoro-1-methylpropenylsilver reacted with oxygen and with water in solution to form $CF_3CF{=}CHCF_3$.[5] Reaction in $CH_3CN$ with HOH or HCl was shown to yield *trans*-$CF_3CF{=}CHCF_3$; bromine yielded $CF_3CF{=}CBrCF_3$ (85% *trans*).[6] Reaction products were isolated by glpc and/or distillation and determined quantitatively by glpc using 20% dioctyl phthalate on Chromosorb P columns at 25–80°. The addition of 1 ml of $H_2O$ to 0.023 mol of $CF_3CF{=}C(CF_3)Ag$ in 25 ml of $CH_3CN$ at 25° yielded 0.022 mol of *trans*-$CF_3CF{=}CHCF_3$ and black silver oxide; 12 ml of a 2.56 *M* solution of HCl in $CH_3CN$ and 0.028 mol of $CF_3CF{=}C(CF_3)Ag$ in 25 ml of $CH_3CN$ gave 0.025 mol of *trans*-$CF_3CF{=}CHCF_3$ and 0.027 mol of AgCl; 3.5 g of $Br_2$ in 10 ml of $CH_3CN$ and 0.023 mol of $CF_3CF{=}C(CF_3)Ag$ in 15 ml of $CH_3CN$ gave 0.021 mol of $CF_3CF{=}CBrCF_3$ and 0.023 mol of AgBr. Immediate precipitation of the silver oxide and of the silver halides was observed.

The properties of *trans*-$CF_3CF{=}CHCF_3$ were: ir (gas) 1739 (w) (C=C), 1404 (m), 1311 (s), 1271 (s), 1221 (s), 1195 (s), 1066 (m), 1031 (w), and 868 (m) cm$^{-1}$ (lit.[7] ~95% *trans* isomer ir 3.38 (2958), 5.68 (1760), 7.10 (1408), 7.31 (1365), 8.18 (1222), 8.35 (1197), 11.55 (868), and 13.55 μ (738 cm$^{-1}$ calcd); nmr $^{19}F$ ($CH_3CN$) 60.5 (quadruplet, $C\mathit{F}_3CH$), 74.7 (d, $CFC\mathit{F}_3$), and 120.5 (m, $C\mathit{F}CF_3$) ppm upfield from external $CFCl_3$ (lit.[8] nmr $^{19}F$ (neat) 60.4 ($C\mathit{F}_3CH$), 74.6 ($C\mathit{F}_3CF$), and 117.2 ($C\mathit{F}CF_3$) ppm upfield from internal $CFCl_3$); nmr $^1H$ ($CH_3CN$) δ 6.33. The nucleophilic addition of HF to $CF_3C{\equiv}CCF_3$ with $CsHF_2$ in $CH_3OH$[9] also gave the above product as shown by glpc and an identical ir spectrum.

The fluorination of $CF_3CBr{=}CBrCF_3$ with CsF in $CH_3CN$ at reflux temperature with continuous removal of the $CF_3CF{=}CBrCF_3$ yielded a mixture of the *cis* and *trans* isomers, bp 46.5–48.0°, $n^{20}D$ 1.3259, which were separated by glpc. The properties of *trans*-$CF_3CF{=}CBrCF_3$ were: ir (gas) 1672 (w), 1366 (m), 1258 (s), 1230 (s), 1193 (s), 1142 (w), 1031 (m), 979 (m), 868 (w) cm$^{-1}$; nmr $^{19}F$ (neat) 63.8 (d, $C\mathit{F}_3CBr$), 69.3 (broad, $CFC\mathit{F}_3$), and 104.2 (m, $C\mathit{F}CF_3$) ppm upfield from external $CFCl_3$; mass spectrum molecular ions at 260 and 262 ($C_4F_7{}^{79}Br$ and $C_4F_7{}^{81}Br$). The properties of *cis*-$CF_3CF{=}CBrCF_3$ were: ir (gas) 1674 (w), 1320 (s), 1288 (m), 1236 (m), 1205 (s), 1147 (w), 1031 (w), 972 (m), 853 (w), 741 (w), and 702 (m) cm$^{-1}$; nmr $^{19}F$ (neat) 61.0 (pentuplet, $C\mathit{F}_3CBr$), 67.0 (m, $CFC\mathit{F}_3$), and 97.2 ppm (m, $C\mathit{F}CF_3$).

Perfluoro-1-methylpropenylsilver was found to react completely with $CH_3I$ to form *trans*-$CF_3CF{=}C(CH_3)CF_3$ and AgI. An ampoule was charged with 25 ml of $CH_3I$ and 0.021 mol of $CF_3CF{=}C(CF_3)Ag$, undissolved, and shaken at ~25° for 72 hr. Reaction appeared to be complete within 24 hr or less. Analysis of the reaction product showed 0.018 mol of *trans*-$CF_3CF{=}C(CH_3)CF_3$ (uncorrected glpc determination with $CCl_2FCClF_2$ internal standard), bp 37.8–37.9°, $n^{20}D$ 1.286, and 0.001 mol of $CF_3CH{=}CFCF_3$.

The *cis* and *trans* isomers of $CF_3CF{=}C(CH_3)CF_3$ were separated from the reaction product of equimolar quantities of $CH_3Li$ and $CF_3CF{=}CFCF_3$ in ether at −78°. The properties of *trans*-$CF_3CF{=}C(CH_3)CF_3$ were: ir (gas) 2985 (w), 1715 (w), 1464 (w), 1393 (m), 1285 (s), 1222 (s), 1181 (s), 1134 (w), 1038 m, and 843 (w) cm$^{-1}$; nmr $^{19}F$ (neat) 66.2 (d, $C\mathit{F}_3CCH_3$), 69.1 (broad, $CFC\mathit{F}_3$), and 119.9 (m, $C\mathit{F}CF_3$) ppm upfield from $CFCl_3$; mass spectrum (high resolution) molecular ion at 196.0125 ($C_5F_7H_3$, 196.0122). The properties of *cis*-$CF_3CF{=}C(CH_3)CF_3$ were: ir 1709 (w), 1342 (s), 1321 (s), 1211 (s), 1190 (s), 1099 (s), 1031 (m), 877 (w), 750 (w), and 702 (m) cm$^{-1}$; nmr $^{19}F$ (neat) 63.3 (pentuplet, $C\mathit{F}_3CH_3$), 68.7 (m, $CFC\mathit{F}_3$), and 119.0 (m, $C\mathit{F}CF_3$) ppm upfield from external $CFCl_3$.

The *cis* and *trans* configurations for the above compounds were assigned primarily on the basis of their nmr spectra following earlier work with fluoroolefins, particularly that by Tiers.[10] As stated above, our as-

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(3) This method has been previously applied to the synthesis of perfluoroalkylsilver compounds: W. T. Miller, Jr., and R. J. Burnard, *J. Am. Chem. Soc.*, **90**, 7367 (1968). All reactions were carried out under nitrogen.

(4) The nmr spectra were determined by James MacNiel.

(5) Presumably, $CF_3CF{=}C(CF_3)\cdot$ free radicals were formed by the oxygen reaction.

(6) Increasing the isolation time further diminished the isomer purity, suggesting that 100% *trans*-$CF_3CF{=}CBrCF_3$ was formed initially by reaction with $Br_2$.

(7) J. T. Maynard, *J. Org. Chem.*, **28**, 112 (1963).

(8) R. D. Chambers and A. J. Palmer, *Tetrahedron Letters*, 2799 (1968).

(9) Experiment by A. B. Clayton.

(10) For a systematic account see: J. W. Emsley, J. Feeney, and L. H. Sutcliffe, "High Resolution Nuclear Magnetic Resonance Spectroscopy," Vol. II, Pergamon Press, New York, N. Y., 1966, pp 909–916. The generalizations that $|J(\textit{cis}\text{-}CF_3,CF_3)| > |J(\textit{trans}\text{-}CF_3,CF_3)|$ and $|J(\textit{cis}\text{-}F,CF_3)| > |J(\textit{trans}\text{-}F,CF_3)|$ were most useful. For example, our spectra gave, for $CF_3CF{=}CBrCF_3$, $|J(\textit{trans}\text{-}F,CF_3)|$ = 10 Hz and $|J(\textit{cis}\text{-}F,CF_3)|$ = 25 Hz. In all cases $|J(\textit{trans}\text{-}CF_3,CF_3)| < 2$ Hz and $|J(\textit{cis}\text{-}CF_3,CF_3)| > 10$ Hz. Assignments were also consistent with chemical shifts and with the greater volatility and faster glpc elution ob-

signments require that the carbon–silver bond cleavage reactions proceeded stereospecifically with retention of configuration and are in agreement with Whitesides and Casey's finding that *cis*-1-propenyl(tri-*n*-butylphosphine)silver was converted by iodine cleanly into *cis*-1-iodopropene.[1] It was also shown that *trans*-$CF_3CF{=}CHCF_3$ was formed by treating the AgF–$CF_3C{\equiv}CCF_3$ reaction mixture directly with HCl in $CH_3CN$. Thus the configuration of the silver compound appeared to be unchanged by sublimation and the AgF addition reaction stereospecific.[11] In aprotic solvents the *trans* addition of AgF would be expected to take place most easily by a two-step mechanism with initial addition of fluoride ion.

Preliminary experiments with $CF_3CF{=}C(CF_3)Ag$ and a variety of metallic and nonmetallic halides suggest wide application of the perfluorovinylsilver compounds for syntheses in which AgX is eliminated and a perfluorovinyl group introduced in place of halogen.[13] The addition of $CF_3CF{=}C(CF_3)Ag$ to excess cupric bromide in $CH_3CN$ yielded $CF_3CF{=}C(CF_3)C(CF_3){=}CFCF_3$[14] and *trans*-$CF_3CF{=}CBrCF_3$, and analogous coupling reactions can be anticipated.[15] The high degree of thermal stability found for $CF_3CF{=}C(CF_3)Ag$ and implied for other perfluorosilver compounds with silver attached to sp$^2$ carbon[16] will permit the use of a wide range of synthesis conditions including relatively high temperatures. Stereospecific reactions will permit the preparation of pure isomers. Our results also suggest that the preparation of useful silver compounds with other electronegative organic radicals can be accomplished.

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served for the *trans* isomers of $CF_3CF{=}CBrCF_3$ and $CF_3CF{=}C(CH_3)CF_3$.

(11) The chemical evidence for the configuration of the silver compound is consistent with the nmr data but does not constitute independent proof. It rests upon the assumption that both *cis*- and *trans*-$CF_3CF{=}C(CF_3)Ag$ do not yield only *trans* products. Other vinyl organometallic compounds such as those of lithium and mercury characteristically react with retention of configuration.[12]

(12) G. E. Coates and K. Wade, "Organometallic Compounds," Vol. I, 3rd ed, Methuen and Co., Ltd., London, 1967.

(13) For example, *trans*-$CF_3CF{=}C(CF_3)HgCl$ and $[$*trans*-$CF_3CF{=}C(CF_3)]_2Hg$ are readily prepared from $HgCl_2$: unpublished work with R. W. Ashcraft.

(14) First prepared by $CF_3C{\equiv}CCF_3 + F_2 \rightarrow CF_3CF{=}C(CF_3)C(CF_3){=}CFCF_3$, bp 74.1°, $n^{20}{}_D$ 1.2849, $d^{20}{}_4$ 1.6377: unpublished work with J. O. Stoffer.

(15) A. Cairncross and W. A. Sheppard have reported that perfluorophenylcopper yields perfluorobiphenyl with cupric bromide (*J. Am. Chem. Soc.*, **90**, 2186 (1968)).

(16) The thermal stability of perfluorophenylsilver has been found to be comparable to that of *trans*-$CF_3CF{=}C(CF_3)Ag$: unpublished work with K. K. Sun.

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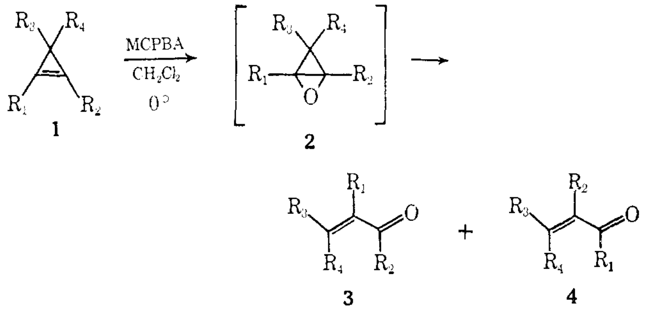
## The Peracid Oxidation of Cyclopropenes. Evidence for 2-Oxabicyclo[1.1.0]butane Intermediates

*Sir:*

The 1-azabicyclo[1.1.0]butane system constitutes the only heterosubstituted bicyclo[1.1.0]butane to have been characterized to date.[1–4] However, the 2-oxa- and 2,4-dioxabicyclo[1.1.0]butane systems have been proposed as possible intermediates in the photoisomerization of $\alpha,\beta$-unsaturated keto derivatives[5,6] and in the peracid oxidation of diphenylacetylene,[7,8] respectively.

We wish to report the preliminary results of our investigation of the peracid oxidation of three alkyl-substituted cyclopropenes, **1a–c**. In all cases the production of the $\alpha,\beta$-unsaturated aldehyde and/or ketone derivatives **3** and **4** in high yield is best rationalized by initial epoxidation of **1** to give an unstable intermediate, 2-oxabicyclo[1.1.0]butane (**2**), followed by rearrangement.[9] In a typical experiment, the cyclopropenes **1a–c**[10,11] were treated with <1 equiv of commercial

**Chart I.** Products of the Peracid Oxidation of Cyclopropenes



| Cyclopropene | % yield of **3**[a] | % yield of **4**[a] |
|---|---|---|
| **a**, $R_1$ = Me; $R_2$ = $R_3$ = $R_4$ = H | 41[b] | 59[b] |
| **b**, $R_1$ = $R_3$ = $R_4$ = Me; $R_2$ = H | 64[c] | 36[d] |
| **c**, $R_1$ = $R_2$ = $R_3$ = $R_4$ = Me | 90[e] | |

[a] Represent approximate vpc yields. [b] The identity of vpc retention times with commercially available authentic samples served to establish the structures of methacrolein (**3a**) and methyl vinyl ketone (**4a**). [c] $\alpha,\beta$-Dimethylcrotonaldehyde (**3b**) was collected by preparative vpc and identified from the following spectral data: ir ($CHCl_3$, cm$^{-1}$) 1670 (s), 1660 (s), 1655 (s), 1640 (s); nmr ($CDCl_3$, $\delta$) 10.1 (1 H, s), 2.20 (3 H, q, $J$ = 1–1.5 Hz), 1.97 (3 H, s), 1.74 (3 H, q, $J$ = 1–1.5 Hz); mass spectrum $m/e$ 98. The epoxyformate ester derived from Baeyer-Villiger oxidation of **3b** followed by epoxidation appears as a minor product if MCPBA is in excess. [d] Mesityl oxide was identified by comparison of its vpc retention time with that of commercially available authentic material. [e] $\alpha$-Methylmesityl oxide (**3c**) was collected by preparative vpc and identified from the following spectral data: ir ($CCl_4$, cm$^{-1}$) 1685 (s), 1625 (m); nmr ($CDCl_3$, $\delta$) 2.23 (3 H, s), 1.87 (6 H, s), 1.78 (3 H, s); mass spectrum $m/e$ 112. The formation of **3c** was actually nearly quantitative since the only minor product was identified as the epoxy ketone derived from **3c** by further oxidation.

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(1) A. G. Hortmann and D. A. Robertson, *J. Amer. Chem. Soc.*, **89**, 5974 (1967).

(2) A. G. Hortmann and J. E. Martinelli, *Tetrahedron Lett.*, 6205 (1968).

(3) W. Funke, *Angew. Chem. Intern. Ed. Engl.*, **8**, 70 (1969).

(4) Heterocyclic bicyclobutanes of doubtful validity have been described in the earlier literature. For a review see W. L. Mosby in "The Chemistry of Heterocyclic Compounds," Vol. 15, Part One, A. Weissberger, Ed., Interscience Publishers, New York, N. Y., 1961, pp 7–9.

(5) H. E. Zimmerman and W. R. Elser, *J. Amer. Chem. Soc.*, **91**, 887 (1969).

(6) O. L. Chapman and W. R. Adams, *ibid.*, **90**, 2333 (1968).

(7) R. N. McDonald and P. A. Schwab, *ibid.*, **86**, 4866 (1964).

(8) J. K. Stille and D. D. Whitehurst, *ibid.*, **86**, 4871 (1964).

(9) After completion of this work we became aware of a related study of the reaction of 1,2,3-triphenylcyclopropene with peracids in which *cis*- and *trans*-$\alpha$-phenylchalcone were observed as products: L. E. Friedrich and R. A. Cormier, Abstracts, 158th National Meeting of the American Chemical Society, New York, N. Y., Sept 7–12, 1969, ORGN-151.

(10) For a good review of cyclopropene chemistry see G. L. Closs, *Advan. Alicycl. Chem.*, **1**, 53 (1966).

(11) The cyclopropenes **1a–c**, respectively, were prepared by reported methods in comparable yields: (a) F. Fisher and D. E. Applequist, *J. Org. Chem.*, **30**, 2089 (1965); (b) G. L. Closs, L. E. Closs, and W. A.