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DIRECT SYNTHESIS OF FLUORINATED PEROXIDES. V. THE PREPARATION OF PENTAFLUORO-SULFURPEROXY ESTERS BY REACTIONS OF PENTAFLUROSULFUR HYDROPEROXIDE WITH ACYL FLUORIDES

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SUMMARY

The reaction of SF₅OOH with acyl fluorides in the presence of NaF has been investigated and found to yield new peroxy esters of the type RC(O)OOSF₅. An alternate method of syntheses using SF₅OOCl and acyl chlorides is discussed and the cesium fluoride catalyzed conversion of two of the esters to RCF(OF)OOSF₅ is described.

INTRODUCTION

Highly fluorinated peroxides represent a potentially large class of interesting compounds. These materials often exhibit high thermal stability compared to non-fluorinated analogs and provide novel examples of molecular structure. Since 1967 we have been active in the development of synthetic methods for preparation of such materials and have characterized an extensive series of peroxides containing the CF₃OO - group.² The work with trifluoromethyl peroxides has convinced us that the only suitable synthetic methods are those involving the direct addition of a peroxy group to suitable substrates. The recent synthesis of SF₅OOH,^{3,4} SF₅OOCl⁴ and SF₅OOF⁴ allows us to greatly extend the known examples of pentafluorosulfurperoxy compounds, which previously existed only as isolated examples in the literature.⁵

We have recently synthesized a variety of new pentafluorosulfurperoxyalkyl derivatives by the addition of SF₅OOCl to olefins⁶ and in this paper we report the preparation of several new pentafluorosulfurperoxy esters. These compounds are prepared in high yield by the displacement of fluoride from acyl fluorides with SF₅OO. Reactions of SF₅OOH with SF₅OOC(O)F, CH₃C(O)F, CF₃C(O)F, C₂F₅C(O)F, and [FC(O)CF₂]₂CF₂ are described. In addition, an alternate method for the preparation of CH₃C(O)OOSF₅ is discussed as well as the conversion of SF₅OOC(O)F and (SF₅OO)₂CO to the corresponding fluoroxy compounds.

EXPERIMENTAL

General

All compounds were handled in Pyrex or stainless steel vacuum systems equipped with glass-Teflon or stainless steel valves. Pressures were measured with a Wallace and Tiernan differential pressure gauge. Ir spectra were obtained on a Perkin-Elmer model 180 spectrometer using a 10-cm pathlength glass cell fitted with silver chloride windows. Nmr spectra were recorded on a Varian XL-100-15 nmr spectrometer using ~ 80 mole % CCl_3F solutions of the samples. AB_4 spin systems were analyzed with the aid of the LAOCOON 3 computer program. Vapor pressures were determined by a static method employing the isotensiscopy principle.⁸ Equations describing vapor pressure as a function of temperature were obtained by a least-squares fit of the data.

Reagents

The acid fluorides $\text{CH}_3\text{C}(\text{O})\text{F}$, $\text{CF}_3\text{C}(\text{O})\text{F}$, and $\text{C}_2\text{F}_5\text{C}(\text{O})\text{F}$ were obtained from P.C.R. Inc.

Perfluoroglutaryl fluoride was prepared by fluorination of $\text{CF}_2[\text{CF}_2\text{C}(\text{O})\text{C}(\text{O})\text{CF}_2]$ with SbF_3/Cl_2 at 150° . The $\text{SF}_5\text{OOC}(\text{O})\text{F}^{1,3,4}$, $\text{SF}_5\text{OOH}^{3,4}$ and $\text{SF}_5\text{OOC}^{4}$ were prepared by literature methods.

Reaction of Pentafluorosulphur Peroxides with Acid Fluorides. Reactions between SF_5OOH and $\text{RC}(\text{O})\text{F}$ were carried out in a 100 ml Pyrex bulb, fitted with a glass Teflon valve, containing 10 g of dry sodium fluoride. Stoichiometric amounts of SF_5OOH and the acid fluoride, ~ 2 mmol, were condensed into the bulb and the mixture allowed to warm slowly from -78 to 22° . This minimized the decomposition of SF_5OOH over NaF , known to occur at room temperature. The products were separated by trap-to-trap distillation. While no explosions occurred during this work, fluorinated peroxides can detonate under mechanical or thermal shock. Precautions should be taken when handling these compounds.

The reaction of $\text{CH}_3\text{C}(\text{O})\text{Cl}$ with $\text{SF}_5\text{OOC}^{4}$ was carried out in a 100 ml glass reactor by condensing $\text{SF}_5\text{OOC}^{4}$ onto an excess of $\text{CH}_3\text{C}(\text{O})\text{Cl}$ at -196° and then holding the mixture at -78° for 24 hr. The product, $\text{CH}_3\text{C}(\text{O})\text{OOSF}_5$, was collected by distilling the reaction mixture through a trap held at -63° .

The conversion of $\text{SF}_5\text{OOC}(\text{O})\text{F}$ and $(\text{SF}_5\text{OO})_2\text{CO}$ to $\text{SF}_5\text{OOCF}_2\text{OF}$ and $(\text{SF}_5\text{OO})_2\text{CF}_2\text{O}$ was carried out in a 75 ml ss reactor. The carbonyl derivative was condensed

onto 15 g of dry, powdered CsF at -196° and excess F_2 added. The reaction was then held at -78° for 24 hr and the product collected by pumping through traps held at -78° as the reactor warmed to 22° .

A summary of the reactions is given in Table 1 and details for the compounds follow.

TABLE 1

Summary of SF_5OO- Reactions

Reactants ^a	Conditions	SF_5OO- Product
$CH_3C(O)F$, 2.0; SF_5OOH , 1.7 ^b	-196 to 22° , 4 hr	$CH_3C(O)OOSF_5$, 70%
$CF_3C(O)F$, 2.7; SF_5OOH , 1.5 ^b	-78 to -5° , 16 hr	$CF_3C(O)OOSF_5$, 90%
$C_2F_5C(O)F$, 2.0; SF_5OOH , 1.9 ^b	-78 to -15° , 17 hr	$C_2F_5C(O)OOSF_5$, 84%
$CF_2[CF_2C(O)F]_2$, 1.8; SF_5OOH , 1.8 ^b	-78 to -15° , 24 hr	$F(O)CCF_2CF_2CF_2C(O)OOSF_5$, 82%
$CF_2[CF_2C(O)F]_2$, 0.9; SF_5OOH , 1.9 ^b	-78 to -10° , 24 hr 22° , 2d	$CF_2[CF_2C(O)OOSF_5]_2$, 86%
$CH_3C(O)Cl$, 5.2; SF_5OOC1 , 2.8	-78° , 24 hr	$CH_3C(O)OOSF_5$, 90%
$SF_5OOC(O)F$, 1.7; F_2 , 2.3 ^c	-78° , 24 hr	SF_5OOCF_2OF , 86%
$(SF_5OO)_2CO$, 0.5; F_2 , 0.8 ^c	-78° , 24 hr	$(SF_5OO)_2CFOF$, 60%

a. Amounts in mmol.

b. Reaction run with 10 g of NaF.

c. Reaction run with 15 g of CsF.

$(SF_5OO)_2CO$. 50% yield. Bp 108.6° ; mp -45.5° to -46.5° ; mol wt 345.4, calcd 346.1; ir 1900 (m, C=O), 1880 (m, C=O), 1188(m), 1130(s), 932(vs, SF), 890(s, SF), 868(vs, SF), 740(m, sh), 723(m), 637(w), 611(s, sh), 602(vs, δSF_5), 592(s, sh), 491(w); n.m.r. AB_4 pattern $\phi_A^* = -57.3$, $\phi_B^* = -55.9$, $J_{AB} = 153.3$ Hz, $J/\delta = 1.16$; $\Delta H_{vap} = 9.10$ kcal/mol; $\Delta S_{vap} = 23.8$ eu; $\log P(\text{mm}) = 8.0898-1988.4/T$.

$SF_5OOC(O)CH_3$. 85% yield. Bp 104.2 ; mp -55.6° ; mol wt 201.0, calcd 202.1; ir 1842(s, C=O), 1370(m), 1185(w, sh), 1160(s), 972(w), 924(vs, SF), 879 (VS, SF), 823(s, SF), 738(w), 661(w), 604(s, δSF_5), 590(m, sh), 500(vw), 483(vw, sh); nmr AB_4 pattern $\phi_A^* = -62.4$, $\phi_B^* = -50.2$, $J_{AB} = 156.6$ Hz, $J/\delta = 0.13$, $\delta = -2.36$ (s); $\Delta H_{vap} = 8.68$ kcal/mol; $\Delta S_{vap} = 23.0$ eu; $\log_{10} P(\text{mm}) = 7.9054-1895.0/T$.

$SF_5OOC(O)CF_3$. 90% yield. Bp 45.8° ; mp (glassed at low temperature); mol wt 256.0, calcd 256.1; ir 1861(s, C=O), 1300(m), 1239(s), 1201(s), 1064(s), 928(vs, SF), 886(vs, SF), 855(s, SF), 747(m), 729(w), 604(s, δSF_5), 593(m, sh), 520(w),

504(w); nmr AB_4 pattern $\phi_A^* = -57.9$, $\phi_B^* = -56.7$, $J_{AB} = 154.8$ Hz, $J/\delta = 1.37$, $\phi^*(CF_3) = 171.8$ (s); $\Delta H_{vap} = 6.70$ kcal/mol; $\Delta S_{vap} = 21.0$ eu; $\log_{10} P(\text{mm}) = 7.4747 - 1465.3/T$.

$SF_5OOC(O)C_2F_5$. 84% yield. Bp 60.0; mp-61.0° to 62.5°; mol wt 304.4, ca 306.1; ir 1851(s, C=O), 1338(m), 1254(m, sh), 1236(s), 1197(m, sh), 1108(m), 1000(m), 988(m, sh), 927(vs, SF), 879(vs, SF), 837(m, sh, SF), 802(w), 750(s) $\begin{matrix} A & B & C & D \end{matrix}$ 726(w, sh), 605(s, δSF_5), 589(w, sh), 551(vw); nmr $FSF_4OOC(O)CF_2CF_3$ AB_4 pattern unresolved, major peak at $\phi^* = -56.4$, $\phi_C^* = +119.8$, $\phi_D^* = +82.9$, $J_{CD} = 2.0$ Hz, $\Delta H_{vap} = 8.23$ kcal/mol; $\Delta S_{vap} = 24.7$ eu; $\log_{10} P(\text{mm}) = 8.2786 - 1798.4/T$.

$SF_5OOC(O)CF_2CF_2CF_2C(O)F$. 82% yield. Bp 116.6°; mp(glassed at low temperature); mol wt 382.8, calcd 384.1; ir 1888(s, C=O), 1853(s, C=O), 1318(w, sh) 1276(m, sh), 1208(s), 1147(s), 1097(ms), 1066(m, sh), 1024(m), 932(vs, SF), 893(m, sh, SF), 875(vs, SF), 822(m), 738(w), 718(w), 650(w), 606(s, δSF_5), $\begin{matrix} A & B & C & D & E & F \end{matrix}$ 591(m, sh); nmr $FSF_4OOC(O)CF_2CF_2CF_2C(O)F$ AB_4 pattern unresolved, major peak at $\phi^* = -56.2$, $\phi_C^* = +116.8$ (m), $\phi_D^* = +123.7$ (d), $\phi_E^* = +118.6$ (d,t), $\phi_F^* = -23.6$ (t,t) $J_{CF} = 1.6$ Hz, $J_{DF} = 6.4$ Hz, $J_{EF} = 7.5$ Hz; $\Delta H_{vap} = 9.38$ kcal/mole, $\Delta S_{vap} = 24.1$ eu $\log_{10} P(\text{mm}) = 8.1410 - 2040.4/T$.

$[SF_5OOC(O)CF_2]_2CF_2$. 86% yield, ~2 mm vapor pressure at 22°; mp(glassed at low temperature); mol wt 516.3, calcd 524.2; ir 1853(s, C=O), 1210(s), 1188(m) 1130(s), 1092(w), 965(w, sh), 942(vs, SF), 886(vs, SF), 869(vs, SF) 840(m), $\begin{matrix} A & B & C & D \end{matrix}$ 812(w), 746(w), 603(vs δSF_5), 592(m, sh); nmr $[FSF_4OOC(O)CF_2]_2CF_2$, AB_4 pattern unresolved, major peak at $\phi^* = -56.8$, $\phi_C^* = +116.1$ (s), $\phi_D^* = +122.6$ (s).

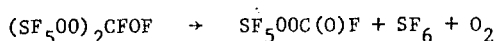
SF_5OOCF_2OF . 90% yield. Mol wt 244.5, calcd 244.1; ir 1295(m), 1269(s), 1245(s), 1208(s), 1175(s), 950(m, sh), 935(vs, SF), 882(vs, SF), 808(w), 751(698(w), 658(vw), 635(w, sh), 609 (s, δSF_5), 590(sh), 560(w); nmr $F^A SF_4^B OOCF_2$ AB_4 pattern unresolved, major peak at $\phi^* = -56.4$, $\phi_C^* = 80.2$, $\phi_D^* = -156.8$, $J_{AC} = 3.8$, $J_{CD} = 33.8$ Hz.

$(SF_5OO)_2CFOF$: ~60% yield. Mol. wt. 385.2, calcd 384.1; ir 1260(s), 1168 1022 (w), 934 (vs, SF), 890 (s, SF), 871 (vs, SF), 746 (m), 606 (s, δSF_5), 596 (m, sh), 574 (w, sh); nmr $(F^A SF_4^B OO)_2CF^C OF^D$ AB_4 pattern unresolved, major peak at $\phi^* = -57.1$, $\phi_C^* = 90.8$, $\phi_D^* = -169.6$, $J_{CD} = 20.0$, $J_{BC} \approx 4$ Hz.

RESULTS

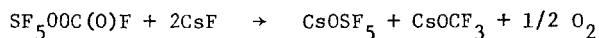
Pentafluorosulfur hydroperoxide undergoes reaction with a variety of acyl fluorides to form pentafluorosulfurperoxy esters in high yield.

These new O-F compounds are unusual as only a few compounds of this type, containing both peroxy and O-F linkages in the same molecule, are known⁵. Neither SF₅OOCF₂OF or (SF₅OO)₂CFOF were studied in much detail and the latter has only limited stability at 22°, decomposing according to the following equation.



These products suggest that SF₅OOF⁴ may be an intermediate in the reaction. The unusually low value for ϕ*OF of the bisperoxy derivative is also noteworthy.

The ease of formation of the OF derivatives suggests that several of the other esters, excluding CH₃C(O)OOSF₅, can be similarly converted to the fluorocompounds. In the course of these and other reactions, it was found that both (SF₅OO)₂CO and SF₅OOC(O)F are unstable in the presence of CsF at temperatures appreciably greater than -78°. For SF₅OOC(O)F, the following reaction occurs.

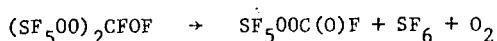


DISCUSSION

The new pentafluorosulfurperoxy esters increase the number of known compounds of this type from one to seven. Only SF₅OOS(O)F⁹ was previously known, illustrating the lack of generality for free radical coupling reactions in the synthesis of fluorinated peroxides. It can be readily assumed, based on the examples in this work, that the reaction of SF₅OOH with acyl fluorides has wide applicability in the synthesis of this class of peroxides. The use of SF₅OOCl is probably also of general applicability, although because of its greater reactivity and because of its instability, it would offer little advantage. However, this reaction suggests an obvious pathway for the preparation of pentafluorosulfuroxy esters of the type RC(O)OSF₅.

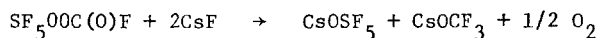
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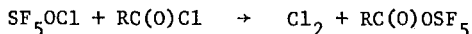


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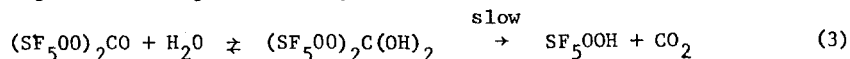
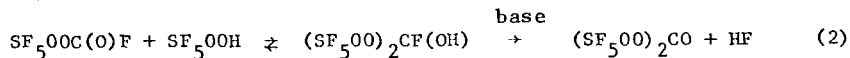
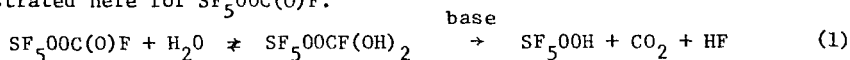
Pentafluorosulfur hypochlorite SF₅OCl¹², is quite analogous to SF₅OOCl in its reaction chemistry. Because SF₅OH does not exist, the preparation of

SF₅O esters can not be accomplished in the same way as SF₅OO esters. However, the use of SF₅OCl in oxidative displacement reaction of the type



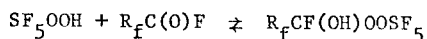
is suggested by this work. At present, FC(O)OSF₅⁹ is the only known compound of this type.

It is interesting to speculate on the mechanism of the reaction of SF₅OOH with acyl fluorides. As with earlier studies of CF₃OOH, the reaction is catalyzed by NaF¹³. While there seems to be little data available on the mechanism of substitution reactions of acyl fluorides, especially perfluoroacyl fluorides, we believe sufficient data are available to suggest a plausible pathway¹⁴. The pronounced tendency of perfluoroketones and aldehydes to undergo addition reactions with active hydrogen compounds¹⁵, probably extends to perfluoroacyl fluorides. However, the subsequent elimination of HF from the latter is expected to be favorable and base catalyzed. Indeed, the hydrolysis of SF₅OO(O)F and CF₃OO(O)F provide evidence for this. These reactions are relatively slow with excess water and an appreciable intermediate concentration of (SF₅OO)₂CO and (CF₃OO)₂CO form in each case, suggesting the following reaction sequence, illustrated here for SF₅OO(O)F.



In the hydrolysis of SF₅OO(O)F, glass functions as the base forcing the equilibrium to the right. Reaction (2) is fast in the presence of a strong base such as NaF and somewhat slower than (1) in the presence of glass. Reaction (3) is slower than both (1) and (2). In the hydrolysis of both CF₃OO(O)F and SF₅OO(O)F, complete conversion to R_fOOH is difficult. After removal of CO₂ and R_fOOH after a given reaction time, on standing further, additional CO₂ and R_fOOH are obtained. Examination of the vapor in equilibrium with the excess water always reveals (R_fOO)₂CO.

In our opinion, the reactions of $R_f\text{OOH}$ with acyl fluorides proceeds in a similar manner, as shown for SF_5OOH .



The base catalysis by NaF is thus viewed as favoring the addition-elimination reaction by promoting the loss of HF, rather than an initial interaction of NaF with SF_5OOH to enhance the nucleophilicity of SF_5OO .

The ^{19}F nmr results for the new esters, along with data for other SF_5OO compounds, provide an interesting comparison of the effects of various groups J/δ for the AB_4 pattern of the SF_5 groups as shown in Table II.

TABLE 2

^{19}F nmr of the AB_4 Spin System in SF_5OO - Derivatives.

Compound	δ_A^*	δ_B^*	J_{AB}	J/δ
$\text{CH}_3\text{C(O)OOSF}_5^{\text{a}}$	-62.4	-50.2	156.6	0.13
$\text{CF}_3\text{C(O)OOSF}_5^{\text{a}}$	-57.9	-56.7	154.8	1.37
$(\text{SF}_5\text{OO})_2\text{CO}^{\text{a}}$	-57.3	-55.9	153.3	1.16
$\text{FC(O)OOSF}_5^{\text{9}}$	-57.7	-56.3	156.0	1.11
$\text{ClCH}_2\text{CH}_2\text{OOSF}_5^{\text{6}}$	-63.6	-45.6	155.0	0.086
$\text{SF}_5\text{OOSF}_5^{\text{16}}$	-56.7	-57.3	~ 150	2.5
$\text{HOOSF}_5^{\text{4}}$	-62.5	-51.8	155.7	0.15
$\text{ClOOSF}_5^{\text{4}}$	-58.8	-56.3	155.0	0.60
$\text{FSO}_2\text{OOSF}_5^{\text{16,b}}$	~ 58	~ 60	155	1.83
$\text{CF}_3\text{OOSF}_5^{\text{16,c}}$	~ 57.3	~ 56.0	155	2.86

a. This work.

b. A value of $\delta^* -43$ was assumed for $\text{SF}_5\text{OOSO}_2\text{F}$ as the authors in ref. 16 only reported chemical shifts relative to the basal fluorines of the SF_5 group.

c. No standard was given for this compound in ref. 16 so δ^* values were calculated assuming $\delta^* \text{CF}_3 = 69.0$. This assumption should be quite good as $\delta^* \text{CF}_3\text{OO}$ occurs over only a very narrow range.²

These data clearly indicate a trend in ϕ_A^* and ϕ_B^* as function of the electronegativity of the group attached to SF₅O. The ϕ_A^* values move to lower field and ϕ_B^* values to higher field as the electronegativity decreases. These opposing trends would indicate that any simple explanation for this is probably better left unstated at the present time. Similar trends are observed for SF₅O-derivatives, but in these compounds the variation in ϕ_B^* covers a much greater range and ϕ_B^* is frequently more negative than ϕ_A^* .¹⁶

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