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TECHNICAL Report No. 9

AEROSOL DIRECT FLUORINATION "INDIRECT SYNTHESES OF PERFLUOROCYCLOKETONES"

by

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Aerosol Direct Fluorination - Indirect Syntheses of Perfluorocycloketones

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Submitted to

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Ring opening during the aerosol direct fluorination of cyclic ketones can be circumvented by the aerosol direct fluorination of the appropriate methoxycycloalkanes or the ethylene glycol ketals of the cyclic ketones followed by sulfuric acid hydrolyses of the perfluorinated analogs. Aerosol direct fluorinations of methoxycyclopentane and methoxycyclohexane produce their respective perfluoroanalogs in effluent concentrations of 57% and 90% by weight and in 22% and 32% isolated yields respectively. Sulfuric Acid hydrolyses at 340° to 360° of the <u>F</u>-methoxycycloalkanes produced <u>F</u>-cyclopentanone (89% yield, 61% conversion) and F-cyclohexanone (82% yield, 28% conversion) respectively. The aerosol fluorinations of the ethylene glycol ketals of cyclopentanone and cyclohexanone produce F-dioxaspiro[4.4] nonane and F-dioxaspiro[4.5]decane in effluent concentrations of 74% and 76% by weight and in 14% and 12% isolated yields respectively. Sulfuric acid hydrolyses at 500% C produced F-cyclopentanone (45% yield, 23% conversion) and F-cyclohexanone (100% yield, 36% conversion) respectively. Physical loss of starting material caused by condensation within the aerosol fluorinator is the major reason for reduced yields. Hydrolysis yields are based on starting materials consumed and not recovered

Aerosol Direct Fluorination - Indirect Syntheses of Perfluorocycloketones

by

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The aerosol direct fluorination method provides a continuous process for the production of perfluorocarbons from hydrocarbons with efficient fluorine utilization and minimal fragmentation.¹ The application of this process to alkanes, ethers, cycloalkanes, ketals and ketones has been demonstrated. $^{1-3}$ Whereas the aerosol direct fluorination of alicyclic ketones yields the corresponding perfluoroketone as the majority product,² aerosol fluorination of cyclic ketones has produced none of the perfluorinated analogs. In the case of cyclopentanone, the major product was F-pentanoyl fluoride, resulting from the opening of the cyclopentyl ring²; aerosol direct fluorination of cyclohexanone produced only non-volatile products; no evidence for the formation of F-cyclohexanone was found.⁴ However, employing a two-step process perfluorocyclopentanone and perfluorocyclohexanone have been synthesized. Sulfuric acid hydrolysis of the appropriate perfluorinated ketal or ether, formed via aerosol fluorination of the corresponding ketal or ether, produced the corresponding perfluorocycloketone in high yields.

Previous preparations of <u>F</u>-cyclopentanone and <u>F</u>-cyclohexanone are relatively few in number; the only synthesis involving direct fluorination is that reported by Holub and Bigelow wherein <u>F</u>-cyclopentanone was formed in trace amounts from the direct fluorination of cyclopentanone.⁵ <u>F</u>-cyclopentanone and <u>F</u>-cyclohexanone have been prepared via the treatment of the corresponding 2,2-dichloroperfluoro cyclic ketones or 1,2-dichloroperfluorocycloalkene epoxides with potassium fluoride in tetramethylene sulfone⁶, and

by the contact of Lewis acids or bases with the appropriate perfluoro olefin epoxides.⁷ In addition to the above methods, Tatlow, et al.,⁸ prepared <u>F</u>-cyclopentanone by the cleavage of methyl nonafluorocyclopentyl ether with sulfuric acid, and <u>F</u>-cyclohexanone by the cleavage of methyl or fluoromethyl undecafluorocyclohexyl ether with sulfuric acid.

Discussion

The syntheses of <u>F</u>-cyclopentanone and <u>F</u>-cyclohexanone were effected via two separate two-step reaction sequences. Scheme 1 outlines the syntheses of the perfluorocycloketones in a two-step process from the corresponding methyl ethers. <u>F</u>-Methoxycyclopentane (IIa) and <u>F</u>-methoxycyclohexane (IIb) obtained from the aerosol direct fluorination of the corresponding hydrocarbon ethers (Ia,Ib) in 22% and 32% yields respectively, were converted to <u>F</u>-cyclopentanone (IIIa) and <u>F</u>-cyclohexanone (IIIb) by hydrolysis with 100% sulfuric acid at 340°-360°C in 89% and 82% yields respectively. <u>F</u>-methoxycyclopentane and <u>F</u>-methoxycyclohexane comprised 57% and 90% respectively of the total products collected by weight from the aerosol reactor. Physical losses due to unfluorinated starting materials "freezing out" in the first reaction zone reduce overall yields to the values cited.⁹ <u>F</u>-methoxycyclopentane and <u>F</u>-methoxycyclohexane have been previously prepared by the

Scheme 1



photochemical reaction of trifluoromethyl hypofluorite and the corresponding perfluorocycloalkene.¹⁰,¹¹

The perfluoroethers IIa and IIb are stable compounds: after 16 hours at 200° C in the presence of 100% sulfuric acid, <u>F</u>-methoxycyclopentane showed no signs of reaction. At 340-360°C in the presence of 100 % sulfuric acid both perfluoroethers form their respective perfluoroketones (IIIa,IIIb) in high yields. In each case only the perfluoroketone and unreacted perfluoroether were found in the reaction mixture. Any other products were present only in trace amounts.

Both perfluoroketones are extremely hygroscopic and react readily with water to form the much less volatile monohydrates^{6a}. This suggests that in the aerosol direct fluorinations of cyclopentanone² and cyclohexanone⁴ any perfluoroketone formed might have become hydrated due to reaction with residual water inside the reactor, explaining the fact that no perfluoroketones could be isolated from those reactions.

Scheme 2 outlines the syntheses of <u>F</u>-cyclopentanone and <u>F</u>-cyclohexanone in a two-step process from the appropriate ketals. Aerosol direct

Scheme 2



fluorinations of 1,4-dioxaspiro[4.4]nonane (IVa) and 1,4-dioxaspiro[4.5]decane (IVb) produce the previously unknown <u>F</u>-1,4-dioxaspiro[4.4]nonane (Va) and <u>F</u>-1,4-dioxaspiro[4.5]decane (Vb)(74% and 76% respectively of the total product collected by weight) in percent yields based on throughput of 14% and 12%, respectively. As previously mentioned, low yields are mostly due to physical losses in the reactor.⁹

Both perfluoroketals are extremely stable compounds: \underline{F} -1,4-dioxaspiro-[4.5]decane showed no signs of reaction after 26 hours at 350°C in the presence of 100 % sulfuric acid. \underline{F} -1,4-Dioxaspiro[4,4]nonane (Va) and \underline{F} -1,4-dioxaspiro[4,5]decane (Vb) were, however, converted to \underline{F} -cyclopentanone and \underline{F} -cyclohexanone via hydrolysis with 100% sulfuric acid at 500°C in 45% and in 100% yield respectively. In the case of \underline{F} -cyclopentanone, the lower yield is believed to be due to more extensive hydration of the perfluoroketone at the higher temperature. \underline{F} -cyclopentanone is more affected because more ring-strain would be relieved upon hydrate formation than in the case of \underline{F} -cyclohexanone, and hence one would predict \underline{F} -cyclopentanone to be more hygroscopic than \overline{F} -cyclohexanone.

Experimental

The basic aerosol fluorinator design and a basic description of the process are presented elsewhere¹; a modified aerosol generator was adapted to a flash evaporator fed by a syringe pump (Sage Model 341a) driving a 5.000 ml Precision Sampling Corp. "Pressure-lok" syringe.³ Workup of products following removal of hydrogen fluoride consisted of vacuum line fractionation, infrared assay of fractions, gas chromatographic separation of components using either a 7 meter x 3/8" 13 % Fluorosilicone QF-1 (Analabs) stationary phase on 60-80 mesh, acid washed Chromosorb P conditioned at 225°C (12 hours), or a 4 meter x 3/8" 10 % SE-52 phenyl-methyl silicone rubber on acid washed 60-80 mesh Chromosorb P conditioned at 250°C (12 hours). Following gas chromatographic separation (Bendix Model 2300, sub-ambient multi-controller) all products of significance were collected, transferred to the vacuum line, assayed and characterized by vapor phase infrared spectrophotometry (Perkin Elmer 1330), electron impact (70 eV) and chemical ionization (CH₄ plasma) mass spectrometry (Hewlett Packard GC/MS,

5710A GC, 5980A MS, 5934A computer), and ¹⁹F nuclear magnetic resonance (JEOL FX90Q, omniprobe) in CDCl3 with 1 % CFCl3 internal standard. Elemental analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, N.Y. The above characterizations and detailed aerosol reaction parameters (6 pages) are available as <u>Supplementary Material</u>, ordering information is given on any current masthead page.

<u>Aerosol Fluorination of Methoxycyclopentane</u>. Methoxycyclopentane was prepared by the method of Vogel from cyclopentanol.¹¹ A pump speed corresponding to 3.7 mmol per hour was established and 2.40 ml (2.05g, 20.5 mmol) methoxycyclopentane was delivered over a 5.5 hour period. Details of the aerosol parameters are available as <u>Supplementary Materials</u>. For the photochemically finished reaction 2.42g of crude product was isolated; separation on the QF-1 column (temperature program: 20°C, 10 min; 5°C/min to 100°C; 100°C, 1 min; 50°C/min to 180°C) yielded 1.39g (57 %) pure <u>F</u>-methoxycyclopentane, corresponding to a yield of 22 % based on total methoxycyclopentane injected. The ¹⁹F nmr spectrum of <u>F</u>-methoxycyclopentane agreed with that appearing in the literature, ¹¹

<u>Hydrolysis of F-methoxycyclopentane</u>. 0.321g of <u>F</u>-methoxycyclopentane was condensed onto an approximately equal volume of fuming sulfuric acid (30 % SO₃) which had been degassed and vacuum-stripped to remove the SO₃; the reaction tube was then sealed under vacuum and heated in a tube furnace at 360° for 24 hours. The tube was then opened on the vacuum line and the reaction mixture fractionated through -131°C and -196°C cold traps, most of the material collected in the -131°C trap. Products from the -131°C fraction were separated on the SE-52 column (temperature program: -10°C, 5 min; 0.5°C/min to 20°C) and identified as <u>F</u>-cyclopentanone¹¹ (.140g) and unreacted F-methoxycyclopentane (.103g). Percent yield of F-cyclopentanone

based on <u>F</u>-methoxycyclopentane reacted was thus 89%, the percent conversion for the reaction was 61%.

<u>Aerosol Fluorination of methoxycyclohexane</u>. Methoxycyclohexane was prepared by the method of Vogel from cyclohexanol.¹² A pump speed corresponding to 3.8 mmol per hour was established and 3.30 ml (2.89g, 25.3 mmol) of methoxycyclohexane delivered over a 6.75 hour period. From the 3.32g of crude product collected 2.99g (90%) of pure <u>F</u>-methoxycyclohexane was isolated (QF-1 program: 35°C, 10 m; 4°C/m to 100°C, 100°C/m; 50°C/m to 180°C) which corresponded to a yield of 32% based on total methoxycyclohexane injected. The ¹⁹F nmr spectrum of <u>F</u>-methoxycyclohexane was in agreement with that appearing in the literature.¹¹

Hydrolysis of F-methoxycyclohexane. 0.308g of <u>F</u>-methoxycyclohexane was treated with 100% sulfuric acid as described above and heated for 14 hours at 340°C. After workup of the reaction mixture as for <u>F</u>-methoxycyclopentane, separation on the SE 52 column (0°C, 10 m; 1°C/m to 100°C) yielded <u>F</u>-cyclohexanone¹¹ (0.066g) and unreacted <u>F</u>-methoxycyclohexane (.202g). The percent yield of <u>F</u>-cyclohexanone based on the amount of <u>F</u>-methoxycyclohexane reacted was 82%, the percent conversion to product for the reaction was 28%.

<u>Aerosol Fluorination of 1,4-Dioxaspiro[4.4]nonane</u>. 1,4-Dioxaspiro-[4,4]nonane was prepared by the method of Daignault and Eliel from cyclopentanone.¹³ A pump speed corresponding to 4.2 mmol per hour was established, and 3.0 ml (3.21g, 25.1 mmol) of 1,4-dioxaspiro[4.4]nonane was delivered over a 6 hour period. Details of the aerosol fluorination parameters are available as <u>Supplementary Materials</u>. The crude product (1.65g) was separated on the QF-1 column (30°C, 5 m; 5°C/m to 100°C; 100°C,

1 m; 50°C/m to 180°C) and yielded 1.22g (74%) pure <u>F-1,4-dioxaspiro[4.4]-</u> nonane, corresponding to a 14% yield based on total 1,4-dioxaspiro[4.4]nonane injected. The ¹⁹ NMR consists of three singlets of equal intensity at $\phi = -85.32$ ppm; -130.36 ppm and -131.69 ppm. Elemental Analysis: Calculated for C₇F₁₂O₂: C 24.44%, F 66.26%; Found C 24.22%, F 66.25%.

<u>Hydrolysis of F-1,4-Dioxaspiro[4.4]nonane</u>. 0.156g <u>F-1,4-Dioxaspiro-</u> [4.4]nonane was treated with 100% sulfuric acid (as prepared previously) and was heated for 24 hours at 450°C. Separation of the products on the SE-52 column (-10°C, 5 min; 0.5°C/min to 50°C) yielded 0.024g <u>F</u>-cyclopentanone (isolated as the monohydrate) and 0.076g unreacted <u>F-1,4-dioxaspiro-</u> [4.4]nonane. The percent yield of <u>F-cyclopentanone</u> based on the amount of <u>F-1,4-dioxaspiro[4.4]nonane</u> reacted was 45%, the percent conversion to product for the reaction was 23%.

<u>Aerosol Fluorination of 1,4-Dioxaspiro[4.5]decane</u>. 1,4-Dioxaspiro-[4.5]decane was prepared by the method of Daignault and Eliel from cyclohexanone.¹³ A pump speed corresponding to 4.3 mmol per hour was established and 3.0 ml (3.1g, 21.5 mmol) of 1,4-dioxaspiro[4.5]decane was delivered over a 5 hour period. The crude products (1.28g) were separated on the QF-1 column (50°C, 5 m; 2°C/m to 100°C; 100°C, 1 m; 50°C/m to 180°C) and yielded 0.97g (76 %) pure <u>F</u>-1,4-dioxaspiro[4.5]decane, corresponding to a percent yield of 12 % based on total 1,4-dioxaspiro[4.5]decane injected. <u>F</u>-1,4-Dioxaspiro[4.5]decane ¹⁹F NMR consisted of a singlet at $\phi = -83.22$ ppm and a broad multiplet at $\phi = -132.02$ ppm of 4:10 relative intensity. Elemental Analyses: Calculated for C₈F₁₄O₂ C 24.38%, F 67.49%; found C 23.85%, F 66.78%.

Hydrolysis of F-1,4-dioxaspiro[4.5]decane. 0.198g F-1,4-Dioxaspiro-[4.5]decane was treated with 100% sulfuric acid (as prepared previously)

and was heated for 18 hours at 500°C. Separation of the products on the SE-52 column (0°C, 10 m; 1°C/m to 100°C) yielded 0.050g <u>F</u>-cyclohexanone and 0.127g unreacted <u>F</u>-1,4-dioxaspiro[4.5]decane, both identified from their infrared spectra. The percent yield of <u>F</u>-cyclohexanone based on <u>F</u>-1,4-dioxaspiro[4.5]decane reacted was 100 %, the percent conversion to products for the reaction was 36%.

Acknowledgement. This work was supported in part by the Office of Naval Research whose support is gratefully acknowledged.

<u>Supplementary Material Available</u>: Characterization data for products (IR, MS, ¹⁹F NMR, Aerosol Parameters; 6 pages). Ordering information is given on any current masthead page.

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APPENDIX I

Supplementary Material

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Characterization of Intermediate Products

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TABLE 1

TYPICAL AEROSOL FLUORINATION REACTION PARAMETERS

Starting Compound	Fluor (mL/m Mod 1	fine Fl	wo 3	Helfu main	I 2'	hom bom) Mod 2	Mod 3	Reac	T not: Mod 1	(°C) Mod 2	Kod 3	Hydrocarbon throughput mmole/hr	Produced distribution % collected	Product Yield X Theoretical
Methoxycyclopentane Methoxycyclohexane 1,4-Dioxasplro[4,4]nonane 1,4-Dioxaspiro[4,5]decane	20 0 C	2 2 2 2 2	40 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	500 700 750	400 % 750 % 650 % 650 %	0 150 150 150	150 150 150	150 150 150	150 160 175 225	-20 -20 -20	-10 -10 -10	RT RT	3.7 3.8 4.2 4.3	57.4 90.0 74.0 76.1	21.5 32.3 14.2 11.5

TABLE 2

CHARACTERIZATION OF PRODUCTS

F-Methoxycyclopentane



IR: 1320 m, 1280 vs, 1235 vs, 1215 s, 1190 s, 1150 m, 995 s, 985 s, 890 w, 855 w, 730 w, 660 w cm⁻¹

19 F nmr:a

Chemical Shift, ϕ (ppm)	Int.	coupling constants, Hz	assignment
-55.42	3	$J_{16} = 10.0$	1
		$J_{12} = 6.0$	
		$J_{13} = 3.9$	
-124.68			2a
	4	$J_{2a,3b} = 259.4$	
-135.68		,	3Ъ
-129.34			4a
	4	$J_{4a.5b} = 254.8$	
-132.86			5b
-137.96	1	Complex multiplet	6

Mass Spectraa,b

<u>EI(70eV)</u>: 297 (1) C₆F₁₁O, M-F; 231 (8) C₅F₉, M-OCF₃; 181 (13) C₄F₇; 131 (51) C₃F₅; 100 (16) C₂F₄; <u>69 (100) CF₃</u>.

 $\begin{array}{c} \underline{CI(CH_4)}: & 297 \ (1) \ C_6F_{11}O, \ M-F; \ 183 \ (35) \ C_4F_7H_2; \ 175 \ (48) \ C_5F_6H; \ 153 \\ (23) \ C_5F_4O; \ \underline{132 \ (100) \ C_3F_5H}; \ 101 \ (29) \ C_2F_4H; \ 100 \ (13) \ C_2F_4; \\ 69 \ (34) \ CF_3 \end{array}$

aSee ref. 11

bSee ref. 10



F-Methoxycyclohexane

IR: 1300 s, 1270 vs, 1250 vs, 1230 s, 1195 vs, 1150 s, 1055 w, 995 s, 980 vs, 880 w, 850 m, 755 m, 730 m, 690 w, 660 w, 630 m, 610 m cm⁻¹

 $\frac{\frac{19}{F \text{ nmr}} \cdot a}{F_{12}(CF_3)} \phi = -53.83 \text{ ppm, mult.}$

F₁₁(-F) $\phi = -142.77$ ppm, mult. F₁-F₁₀: three overlapping AB quartets $\phi = -122.79$ to -134.63 ppm matching literature spectrum^a

Mass Spectra

 $\frac{\text{EI}(70\text{eV}):}{(7)} \begin{array}{c} 281 & (4) \\ C_6 F_{11}, \\ M \\ -0 \\ CF_3; \\ 259 & (1) \\ C_6 F_9 \\ 0; \\ 231 & (7) \\ C_5 F_9; \\ 181 \\ (7) \\ C_4 F_7; \\ 131 & (52) \\ C_3 F_5; \\ 100 & (6) \\ C_2 F_4; \\ \underline{69} & (100) \\ CF_3. \end{array}$

CI(CH4): 161 (23) C7F4H; 131 (100) C3F5; 100 (17) C2F4; 69 (65) CF3.

^aSee ref.11

TABLE 2b

F-Cyclopentanone

<u>IR</u>:^{a,b} 1830 (vw), 1810 w, 1345 m, 1300 m, 1200 vs, 1145 w, 1115 w, 1010 m, 965 s, 725 m cm⁻¹

19 F nmr:a

 $\phi = -126.52$ ppm, m [4] $\phi = -136.76$ ppm, m [4]

Mass Spectra:^a

EI(70eV): 228 (1) C_5F_80 , M; 200 (2) C_4F_8 ; 181 (2) C_4F_7 ; 131 (78) C_3F_5 ; 100 (100) C_2F_4 ; 69 (11) CF_3 .

<u>CI(CH₄)</u>: 209 (1), C₅F₇O, M-F; 182 (32) C₄F₇H; <u>132 (100) C₃F₅H</u>; 101 (70) C₂F₄H.

^aSee ref. 8 ^bSee ref. 6a

F-Cyclohexanone

a,b

IR 1795 m, 1300 s, 1255 s, 1185 vs, 1220 m, 1080 m, 1025 w, 990 s, 960 vs, 805 w, 725 w, 615 w cm⁻¹

¹⁹F nmr:^a $\phi = -125.15$ ppm, s [4] $\phi = -133.23$ ppm, m broad [6]

Mass Spectra^a

 $\underline{EI}(70eV): 278 (2) C_6F_{10}O, M; 231 (1) C_5F_9; 181 (12) C_4F_7; \underline{131} (100) \\ C_3F_5; 100 (23) C_2F_4; 93 (5) C_3F_3; 81 (3) C_2F_3; 69 (10) CF_3.$

^aSee ref. 8

bsee ref. 6a



<u>IR</u>:^{a,b} 1405 w, 1310 s, 1275 s, 1245 s, 1210 vs, 1160 vs, 1080 m, 1060 m, 1035 m, 980 vs, 810 m, 725 m, 655 w, 595 m, 520 m cm⁻¹.

19F nmr:a

$\phi = -85.32,$	S	[4]	CF_{2a}
$\phi = -130.36$	ppm, s	[4]	CF _{2b}
$\phi = -131.69$	ppm, s	[4]	CF _{2c}

Mass Spectra:

 $\begin{array}{c} \underline{\text{EI}(70\text{eV})}: & 325 \ (4) \ C_7 F_{11} O_2, \ \text{M-F}; \ 225 \ (5) \ C_5 F_7 O_2; \ 209 \ (6) \ C_5 F_7 O; \ 194 \\ (48) \ C_4 F_6 O_2; \ 131 \ (100) \ C_3 F_5; \ 100 \ (60) \ C_2 F_4; \ 69 \ (19) \ CF_3. \end{array}$

CI(CH4): 325 (100) C7F1102, M-F; 117 (98) C5F3; 97 (19) C2F30.

Elemental	Analysis	%C	%F	ΧH
Calc.	for C7F1202	24.44	66.26	0.00
	Found	24.22	66.25	0.00



F-1,4-Dioxaspiro[4.5]decane

 $\frac{IR}{m} = 1400 \text{ w}, 1310 \text{ m}, 1280 \text{ s}, 1250 \text{ vs}, 1220 \text{ s}, 1190 \text{ vs}, 1160 \text{ vs}, 1050 \text{ w}, 980 \text{ vs}, 970 \text{ vs}, 820 \text{ w}, 730 \text{ w}, 625 \text{ w}, 600 \text{ w} \text{ cm}^{-1}.$

¹⁹F nur: $\phi = -83.22$ ppm, s [4]

 $\phi = -132.02$ ppm, m broad [10]

Mass Spectra^a

 $\frac{EI(70eV)}{C_4F_7}: \begin{array}{c} 375 \ (1) \ C_8F_{13}O_2, \ M-F; \ 259 \ (3) \ C_6F_9O; \ 244 \ (4) \ C_5F_8O_2; \ 181 \ (8) \\ C_4F_7; \ \underline{131} \ (100) \ C_3F_5; \ 100 \ (20) \ C_2F_4; \ 97 \ (13) \ C_2F_3O; \ 69 \ (31) \\ CF_3. \end{array}$

 $\frac{\text{CI(CH}_4):}{\text{C}_4\text{F}_8\text{O}_2;} \begin{array}{c} 414 \ (11) \ \text{C}_8\text{F}_{15}\text{O}_2\text{H}, \ \text{M+HF}; \ 348 \ (4) \ \text{C}_7\text{F}_{13}\text{OH}; \ 232 \ (5) \\ \hline \text{C}_4\text{F}_8\text{O}_2; \ 182 \ (12) \ \text{C}_3\text{F}_{6}\text{O}_2; \ 172 \ (24) \ \text{C}_8\text{F}_4; \ 151 \ (18) \ \text{C}_3\text{F}_{6}\text{H}; \ 143 \\ \hline (13) \ \text{C}_4\text{F}_5; \ \underline{131} \ (100) \ \text{C}_3\text{F}_5; \ 117 \ (66) \ \text{C}_5\text{F}_3; \ 100 \ (19) \ \text{C}_2\text{F}_4; \ 97 \\ \hline (71) \ \text{C}_2\text{F}_3\text{O}; \ 95 \ (42) \ \text{C}_5\text{FO}; \ 69 \ (1) \ \text{CF}_3. \end{array}$

Elemental	Analysis	%C	%F	%H
Calc.	for C ₈ F ₁₄ O ₂ :	24.38	67.49	0.00
	Found	23.85	66.78	0.00

APPENDIX II

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1

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