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ELEMENTAL FLUORINE BASED SYNTHESIS OF PENTAFLUORO
PHENYL AND OTHER AROMATIC PERFLUOROPOLYETHER POLYMERS

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13. ABSTRACT (Maximum 200 words)

Since we successfully obtained a fused perfluoro (benzofuran) from perfluoro (dicyclohexyl ether), reductive defluorination of the perfluorinated ethers containing three perfluoro (cyclohexyl) groups would be interesting. The three isomers of o-, m-, and p- perfluoro (dicyclohexanoxyl cyclohexane) were prepared by liquid-phase direct fluorination of o-, m-, and p- diphenoxyl benzene. After several runs of liquid-phase direct fluorination, enough amount of o-perfluoro (dicyclohexanoxyl cyclohexane) was collected to carry out the following reductive defluorination. The reductive defluorination was carried out from -70 to 70 degree for 2 days, but the ortho-ether, however, kept unreacted. One of the reasons for that is perhaps steric hindrance. Reductive defluorination of the meta- and para- ethers are under investigation.

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Second Annual Technical Report

to

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH

Washington, DC 20332

ELEMENTAL FLUORINE BASED SYNTHESSES OF PENTAFLUORO PHENYL
AND OTHER AROMATIC PERFLUOROPOLYETHER POLYMERS

Grant Number F49620-92-J-0104

February 1, 1993 - January 31, 1994

Presented by

Professor Richard J. Lagow

Department of Chemistry

The University of Texas at Austin

Austin, Texas 78712

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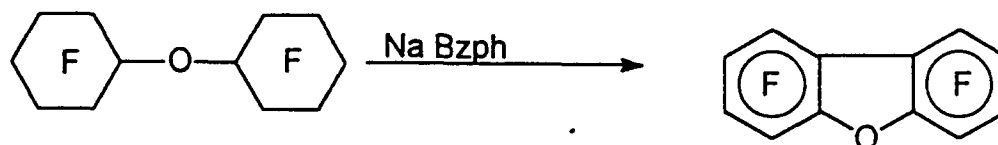
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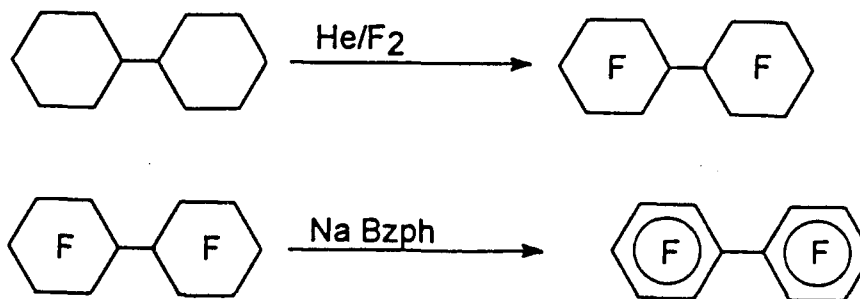
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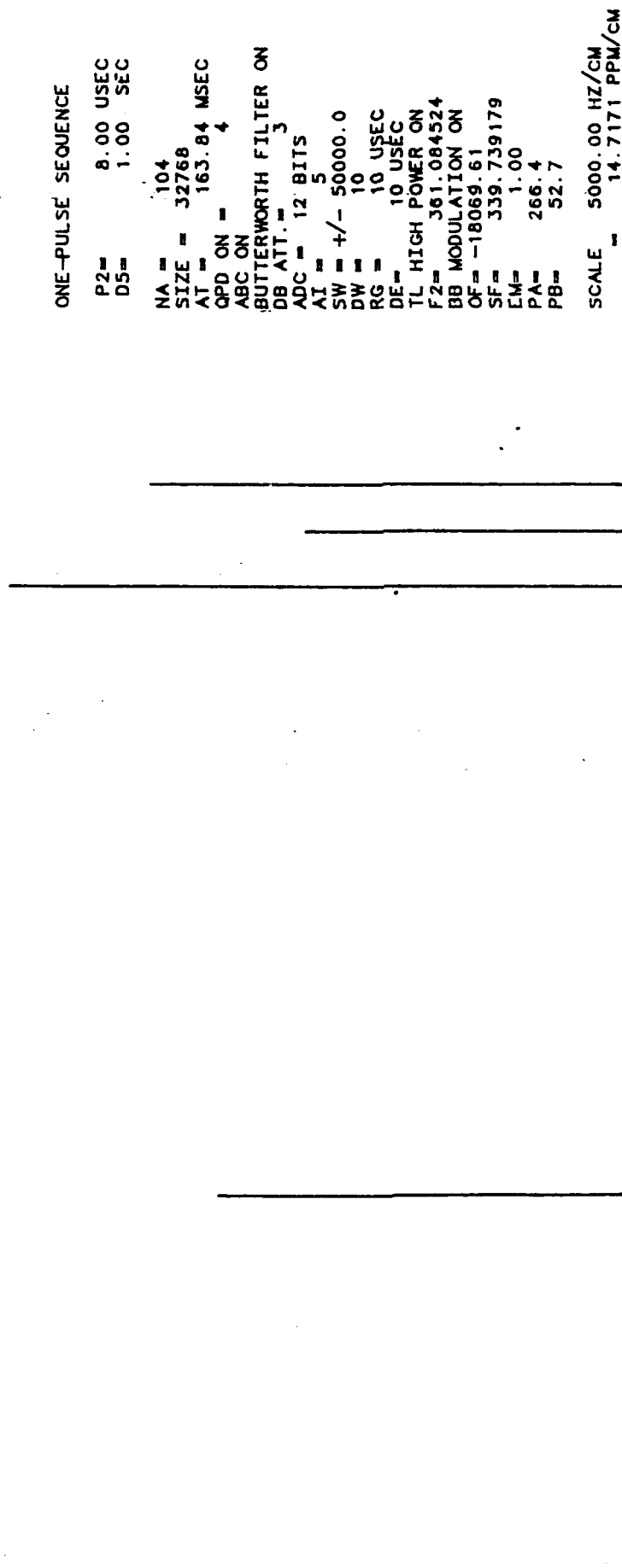
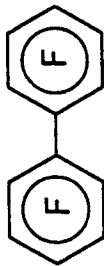
In last annual technical report, reductive defluorination of perfluoro(dicyclohexylether) was carried out with sodium benzophenone and the two perfluoro(cyclohexyl) groups were interestingly fused to form perfluoro(bezofuran). What about other perfluoro(dicyclohexyl) compounds ? Will they form fused products or other interesting compounds after the reductive defluorination ?



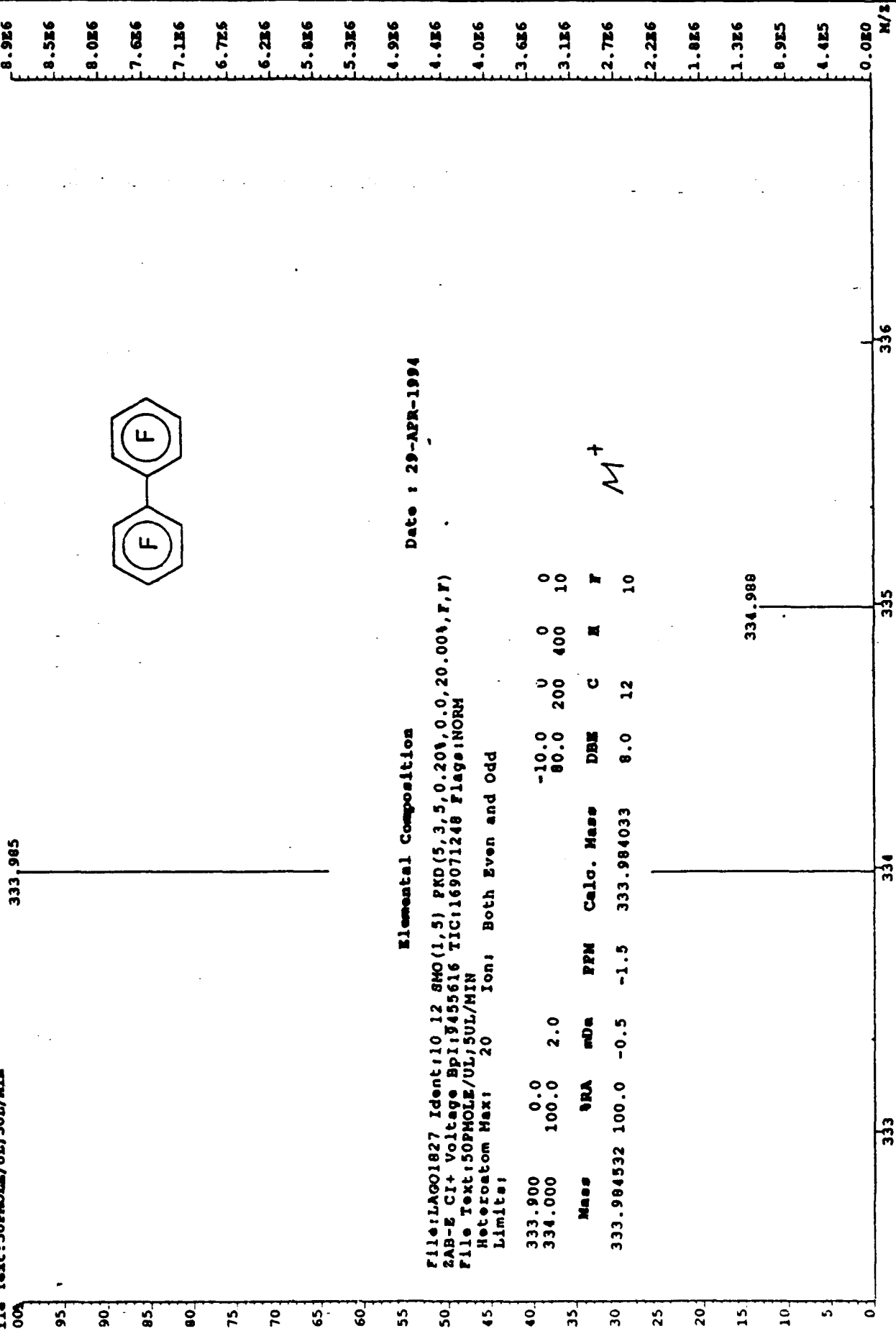
Perfluoro(dicyclohexyl) was prepared in 91% yield by liquid-phase direct fluorination of dicyclohexyl. Reductive defluorination of the perfluorinated compound produced a nonfused product, perfluoro(diphenyl), other than a fused product.



311 . 002 MJB 06MAY94
JNG, CFCL3, CDCL3, F19



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Elemental Composition

Date: 29-APR-1994

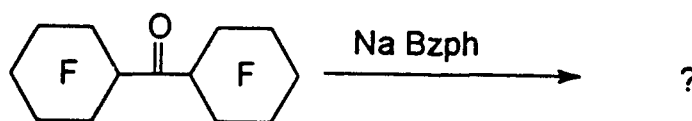
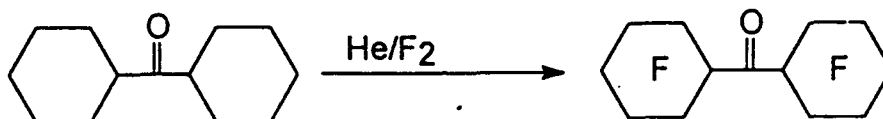
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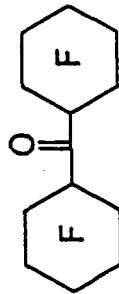
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						12
						10

M⁺

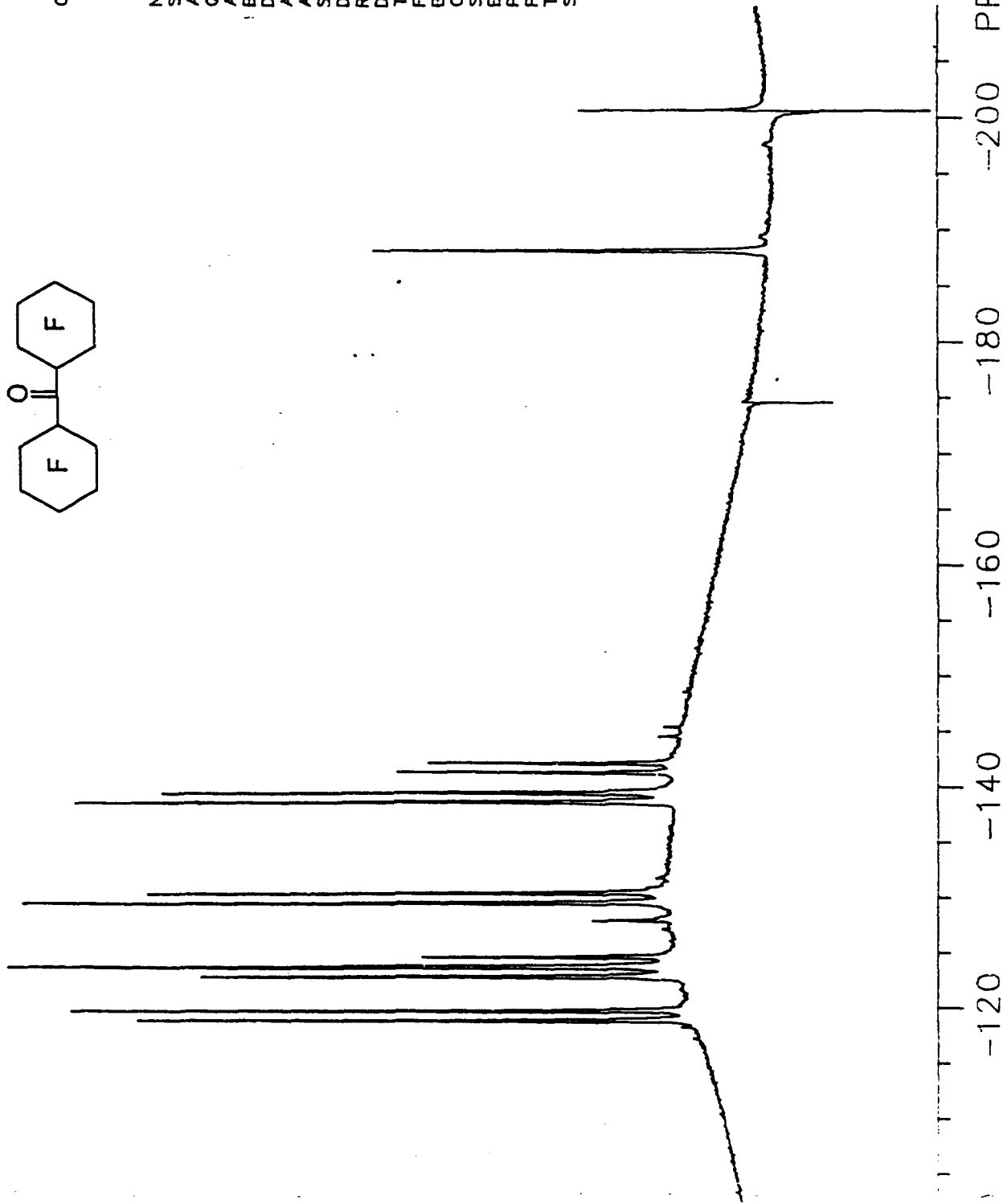
Perfluoro(dicyclohexyl) ketone was prepared in 82% yield by liquid-phase direct fluorination of dicyclohexyl ketone. Reductive defluorination of the perfluorinated ketone is under investigation.



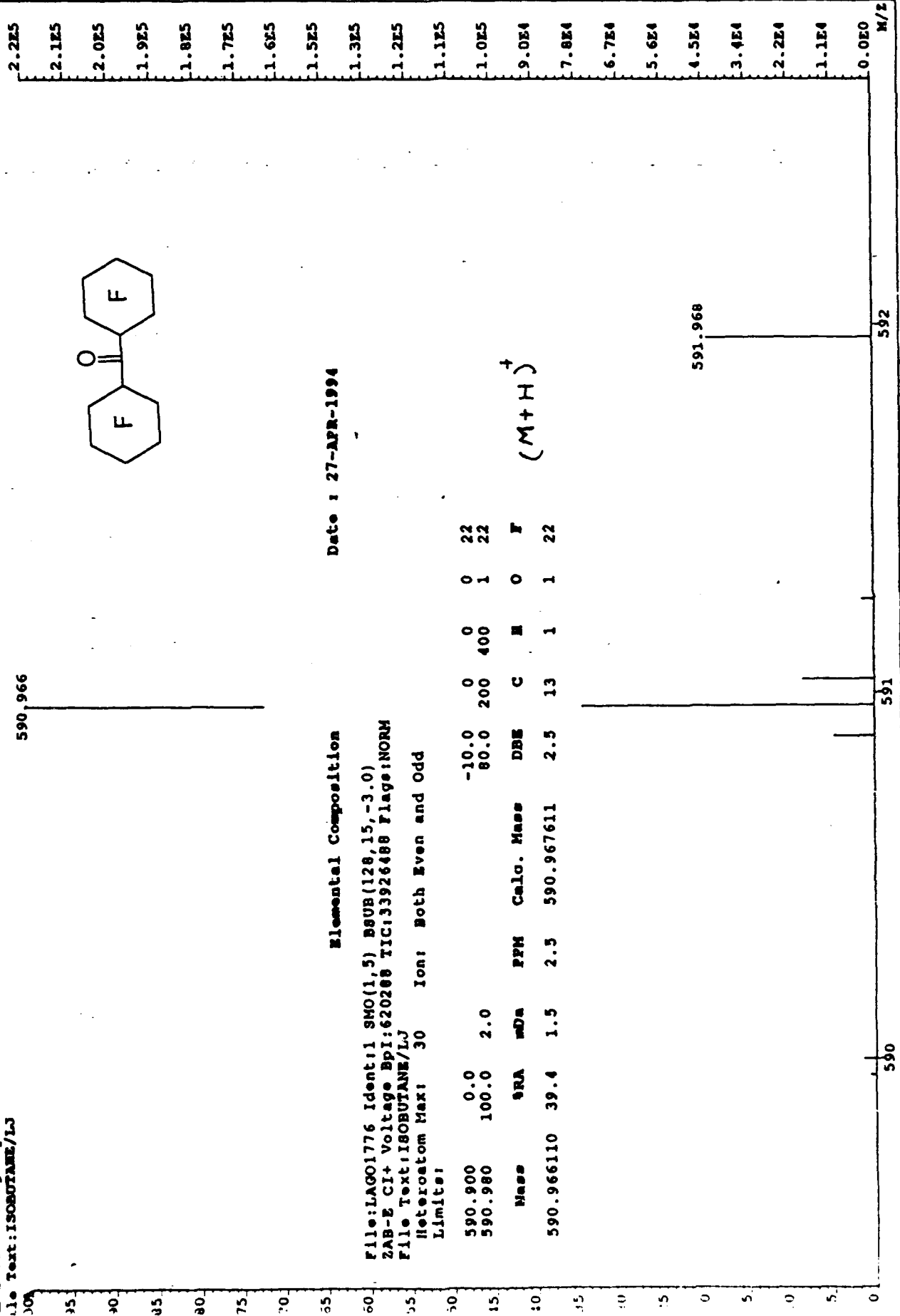
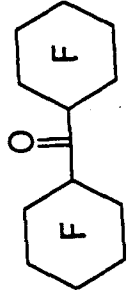
3111 002 MJB 13MAY94
 ING, CFCL3, CDCL3, F19, 20C



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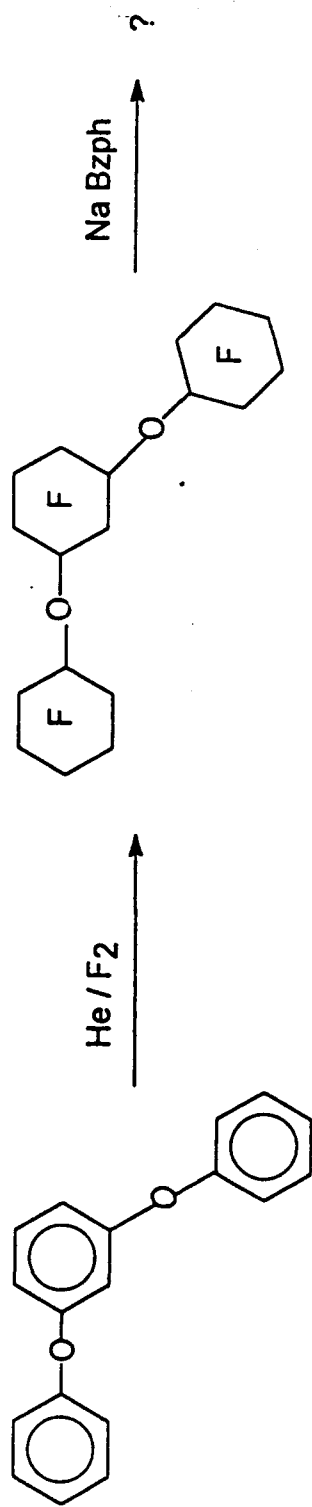
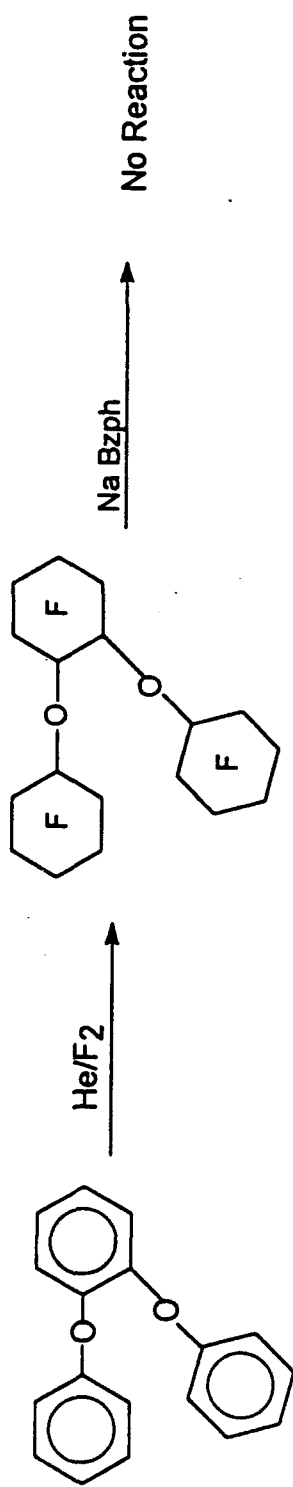
Elemental Composition

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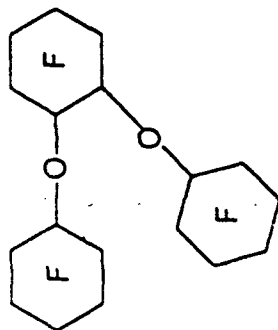
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590.980	100.0	2.0			80.0	200	400	1	22
Mass	%RA	mDa	PPM	Calcd. Mass	DBE	C	H	O	F
590.966110	39.4	1.5	2.5	590.967611	2.5	13	1	1	22

(M+H)⁺

Since we successfully obtained a fused perfluoro(benzofuran) from perfluoro(dicyclohexyl ether), reductive defluorination of the perfluorinated ethers containing three perfluoro(cyclohexyl) groups would be interesting. The three isomers of o-, m-, and p-perfluoro(dicyclohexanoxyl cyclohexane) were prepared by liquid-phase direct fluorination of o-, m-, and p- diphenoxyl benzene. After several run of liquid-phase direct fluorination, enough amount of o-perfluoro(dicyclohexanoxyl cyclohexane) was collected to carry out the following reductive defluorination. The reductive defluorination was carried out from -70 to 70 °C for 2 days, but the ortho-ether, however, kept unreacted. One of the reasons for that is perhaps steric hindrance. Reductive defluorination of the meta- and para- ethers are under investigation.

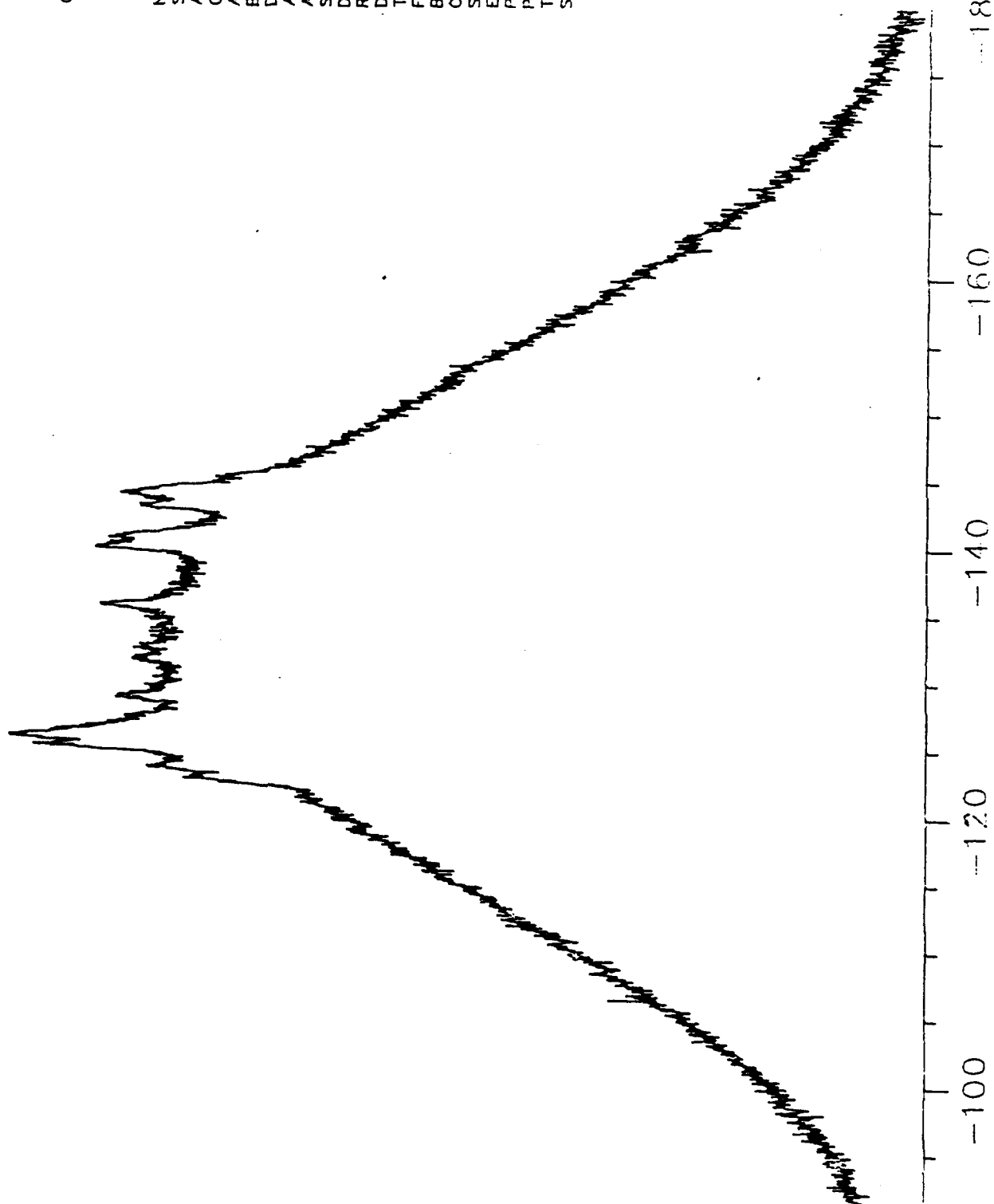


S222 . 004 MJB 11MAR94
 UNG, CDCL3, F19, -30

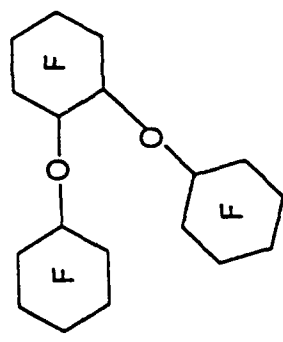


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Elemental Composition

Date: 9-MAR-1994

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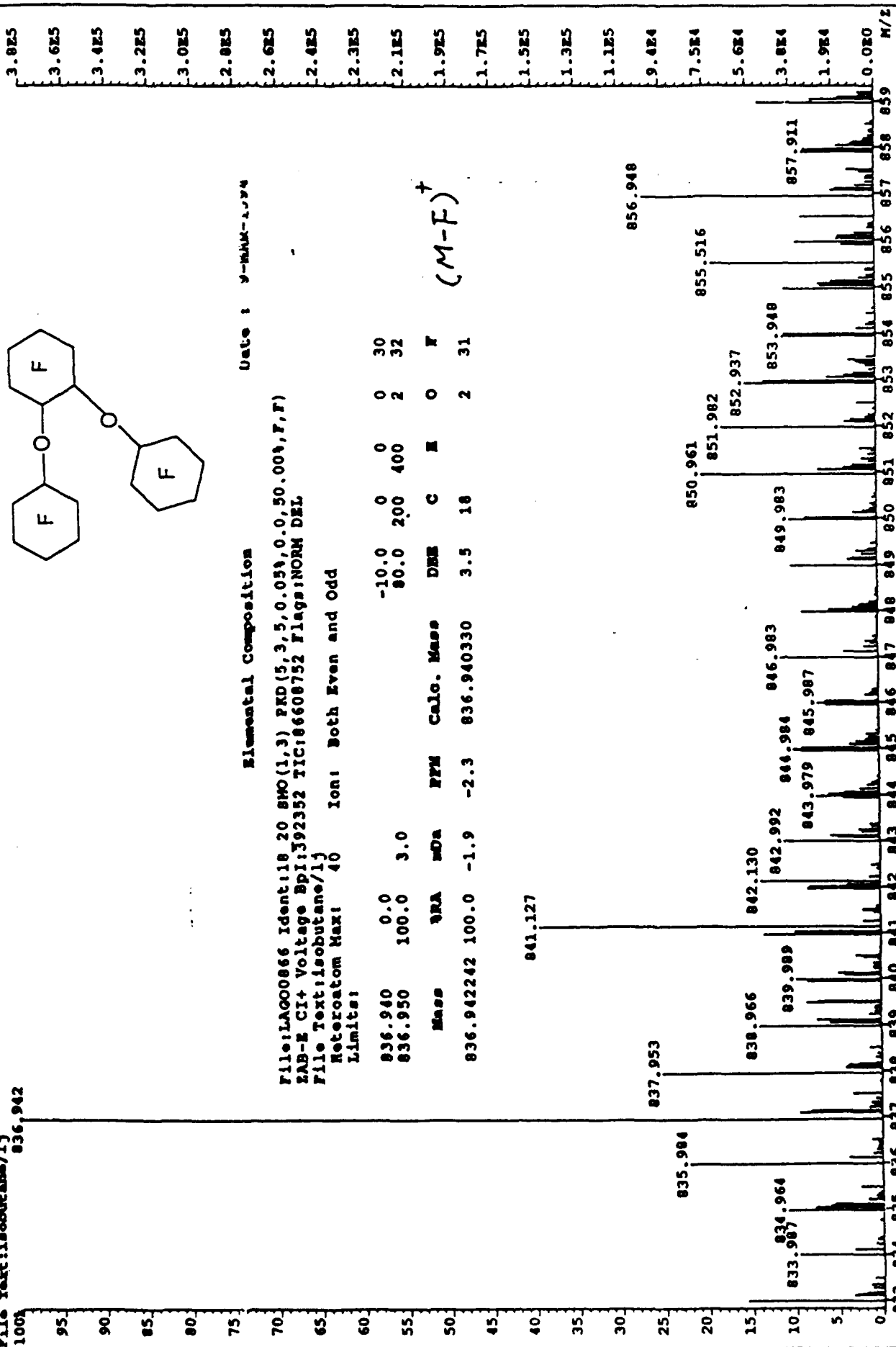
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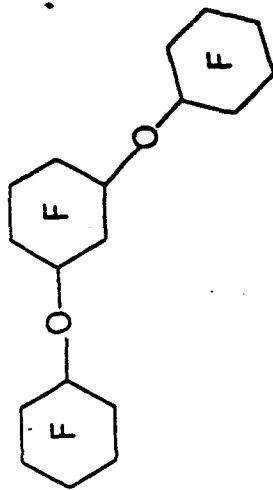
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836.950	100.0	3.0			80.0	200	400	2	32
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(M-F)⁺

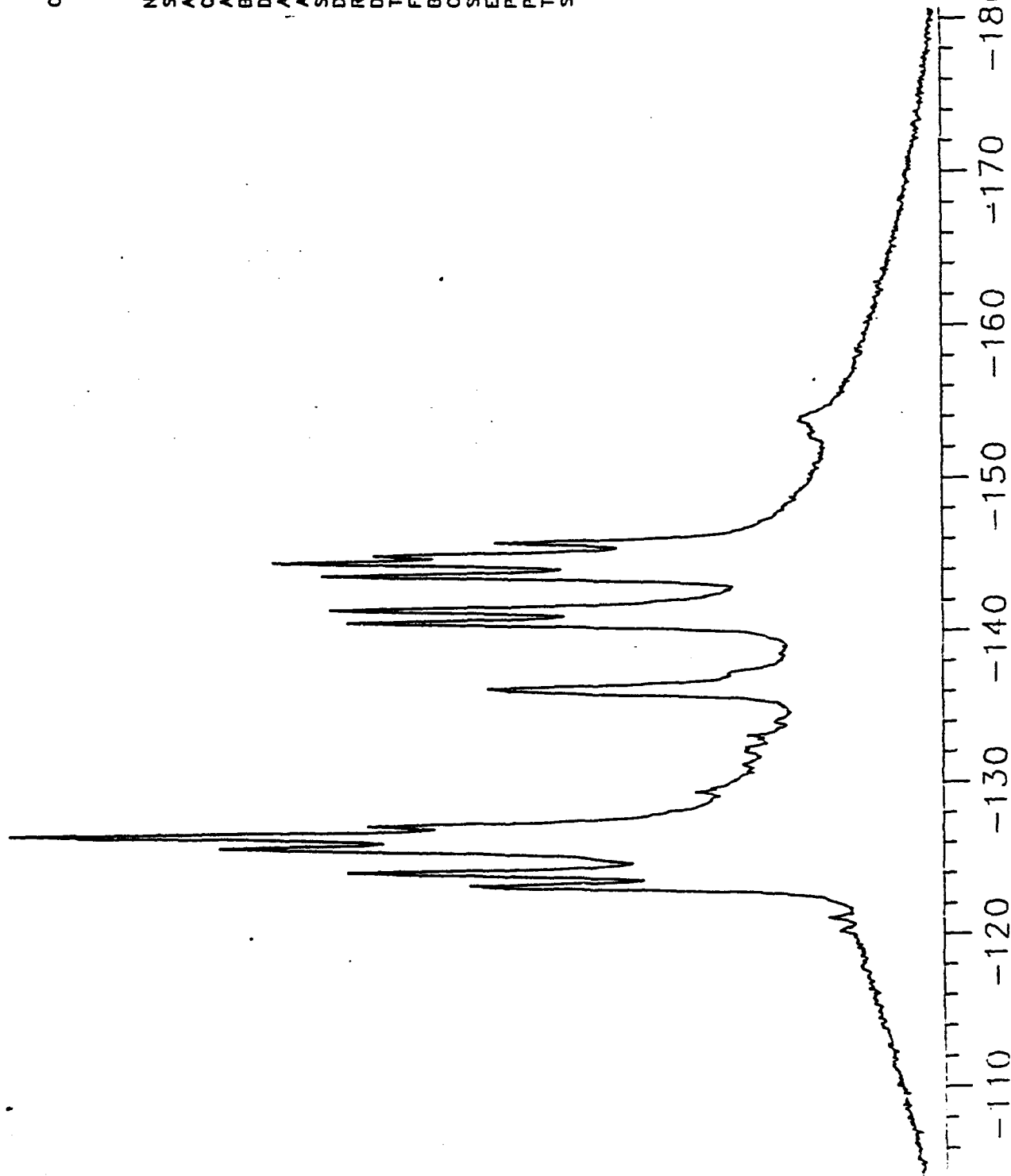


00 004 MJB 24NOV93
 IG, CDCL3, CFCL3, F19, -30



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 = 4.0008 PPM/CM



854.947

PRK

Elemental Composition

Date 21-JAN-1994

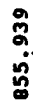
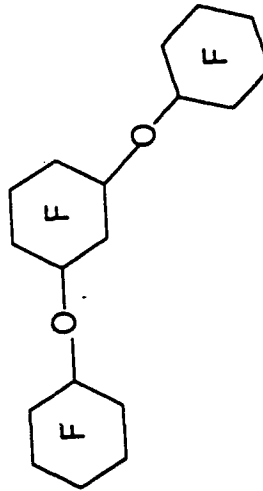
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Limits:

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056.000	100.0 2.0

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80.0	200	400	2	32
DBE	C	M	O	F
3.0	18		2	32

$$M^+$$


856.954

057.362

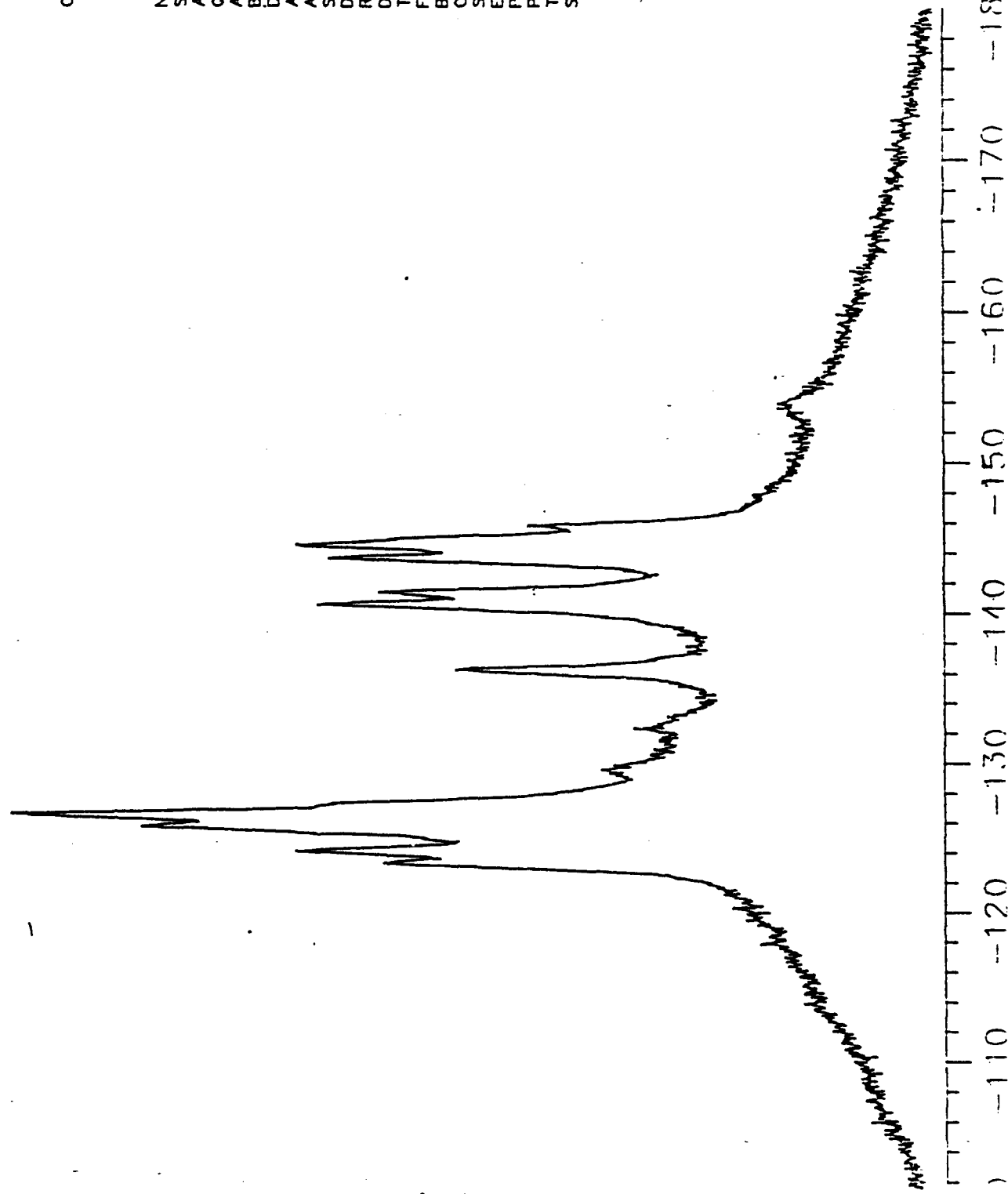
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857,

2/H

3200 . 002 MJB 06JAN94
JNG, CDCL3, CFCL3, F19



ONE-PULSE SEQUENCE

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D5= 1.00 SEC
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836.935

Elemental Composition

Date : 21-JAN-1994

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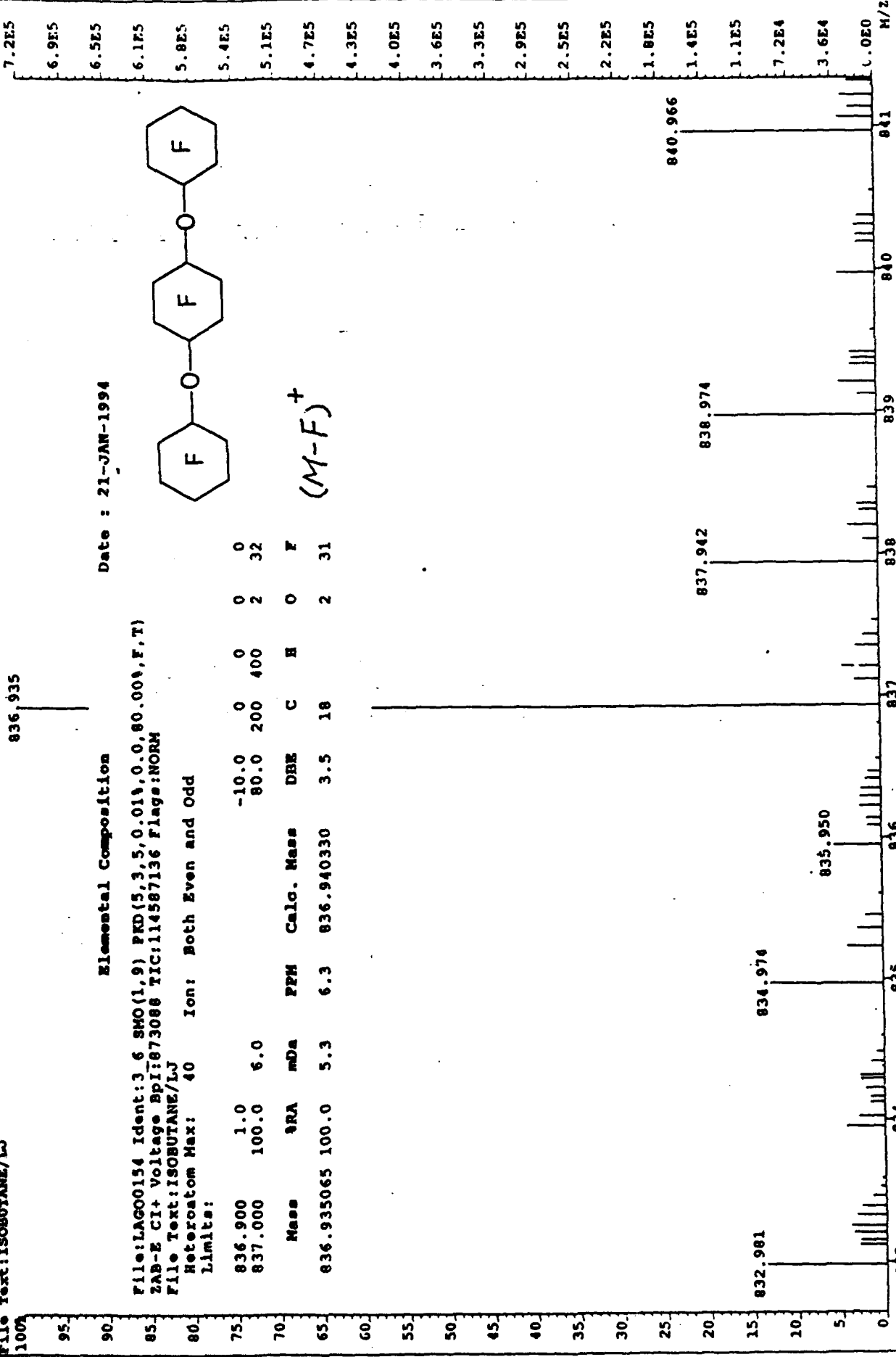
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Heteroatom Max: 40 Ion: Both Even and Odd

836.900	1.0	-10.0	0	0	0	0
837.000	100.0	6.0	80.0	200	400	2 32
Mass	%RA	mDa	PPM	Calcd. Mass	DBE	C H O F
836.935065	100.0	5.3	6.3	836.940330	3.5	18 2 31

(M-F)⁺



Synthesis of perfluoro(hexamethylcyclohexane-1,3,5-trione) was reported in last report. Now we want to show its unusual solid state conformation. The molecule is unusually flattened, as can be seen from the sum of the six ring torsion angles $31 + 18 + 14 + 32 + 18 + 12 = 125^\circ$. By contrast, the sum of the ring torsional angles in cyclohexane and hexamethylcyclohexane-1,3,5-trione are 336 and 192° , respectively. In comparison with boat conformation of hexamethylcyclohexane-1,3,5-trione, conformation of the perfluorinated analogue is a twisted boat in order to avoid the three axial trifluoromethyl groups, coming much too close and creating a serious strain.

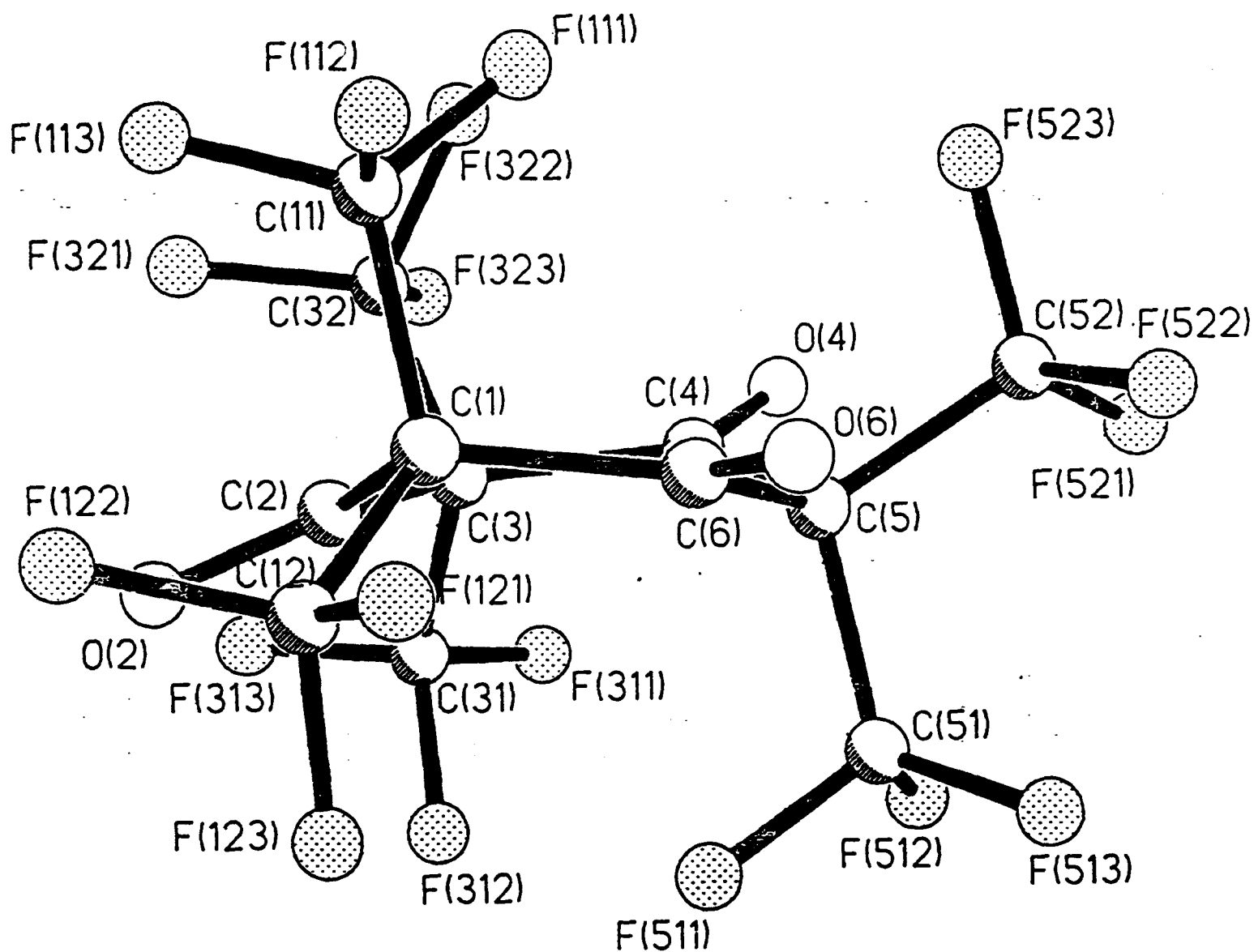
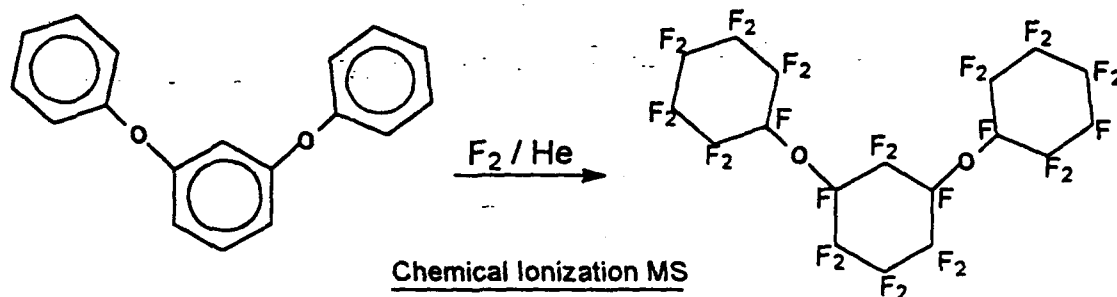


Fig.1. Molecular structure of $C_{12}F_{18}O_3$ (1).;

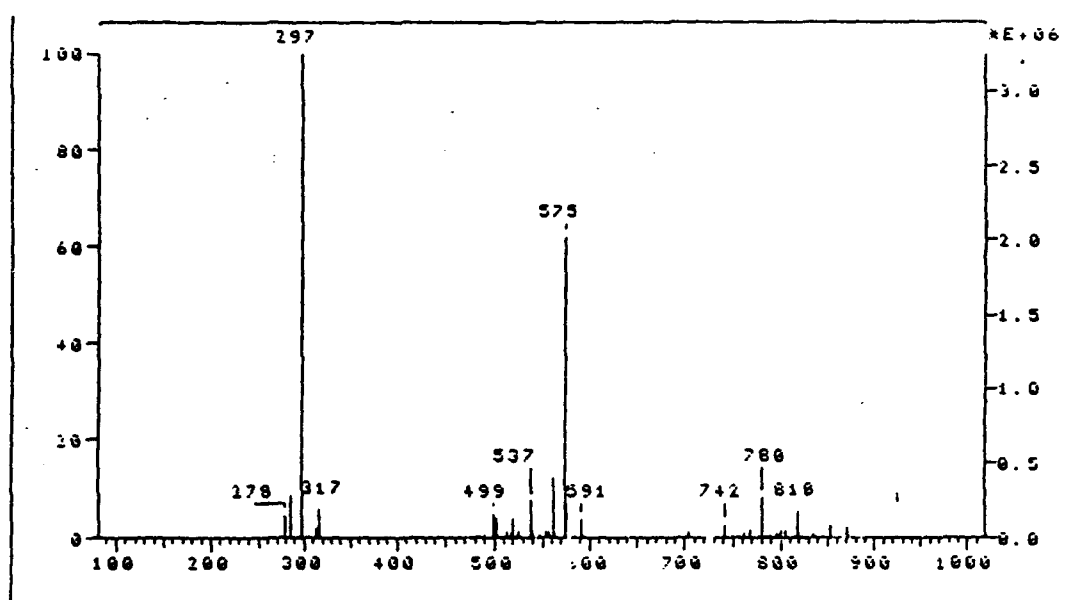
Selected bond lengths (Å): C(2)-O(2) 1.188(5), C(4)-O(4) 1.179(5), C(6)-O(6) 1.178(5); bond angles (°): C(2)-C(1)-C(6) 115.7(4), C(2)-C(3)-C(4) 115.8(4), C(6)-C(5)-C(4) 116.0(4); torsion angles (°): C(6)-C(1)-C(2)-C(3) -30.8(6), C(1)-C(2)-C(3)-C(4) 17.9(6), C(2)-C(3)-C(4)-C(5) 14.4(6), C(3)-C(4)-C(5)-C(6) -32.3(6), C(4)-C(5)-C(6)-C(1) 18.3(6), C(2)-C(1)-C(6)-C(5) 11.8(6).

Other homologs of diphenyl ether such as 1,3-, and 1,4- diphenoxy benzene have also been perfluorinated for subsequent reductive defluorination.

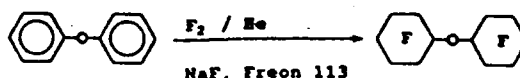


Chemical Ionization MS

Parent	-	872
P-F	-	853
P- (C ₆ F ₁₁)	-	591
P- (C ₆ F ₁₁ O)	-	575
P- (C ₆ F ₁₁ O-C ₆ F ₁₀)	-	297
P- 2x (C ₆ F ₁₁ O)	-	278



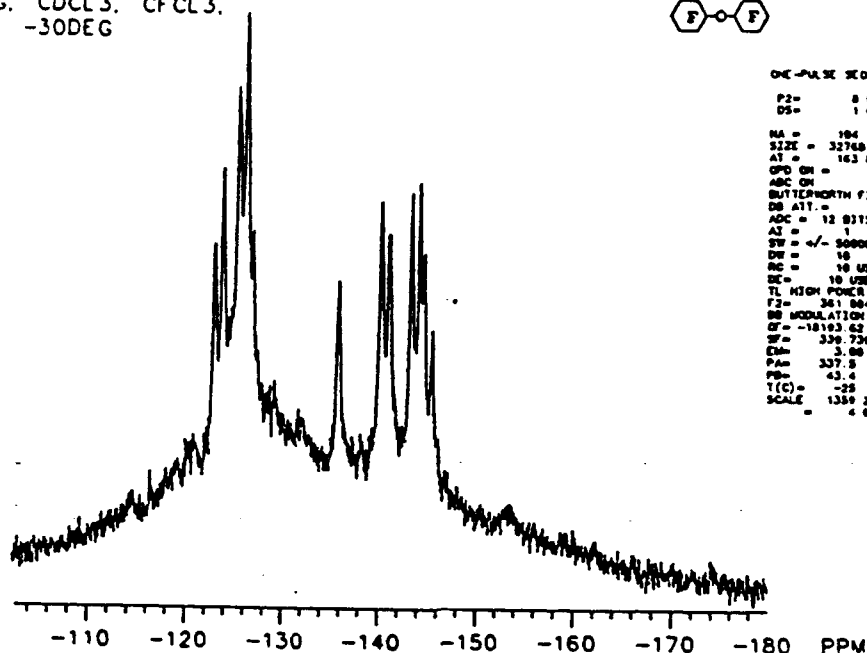
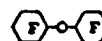
We have done considerable work on defluorination of cyclohexyl substituted fluorine compounds. This work is now becoming more and more successful and has been aided very considerably by the publication of a full manuscript by Dr. Guido Pez on methods using sodium benzophenone (J. Org. Chem. 1992, 57, 2856-2860). Yet removing extra fluorines remains one of the problems that we are in the process of solving. There are a series of sodium substituted defluorination reagents. One of the mildest is sodium phenylthiolate by Professor David McNicol of Glasgow. The synthesis of perfluoro biscyclohexyl ether was accomplished in 83% yield.



The ^{19}F NMR Spectrum of Biscyclohexyl Ether

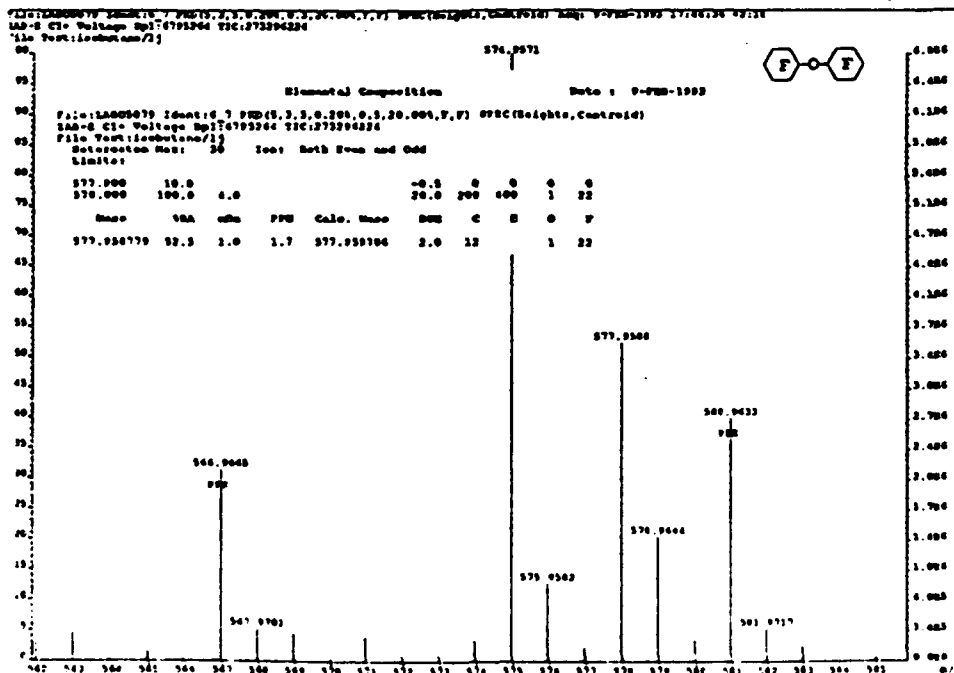
5051 004 MJB
JNG. CDCL3. CFCL3.
19. -30DEG

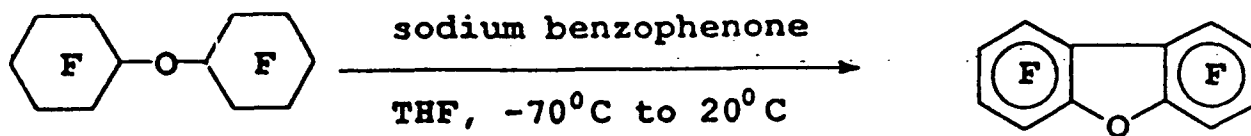
20OCT93



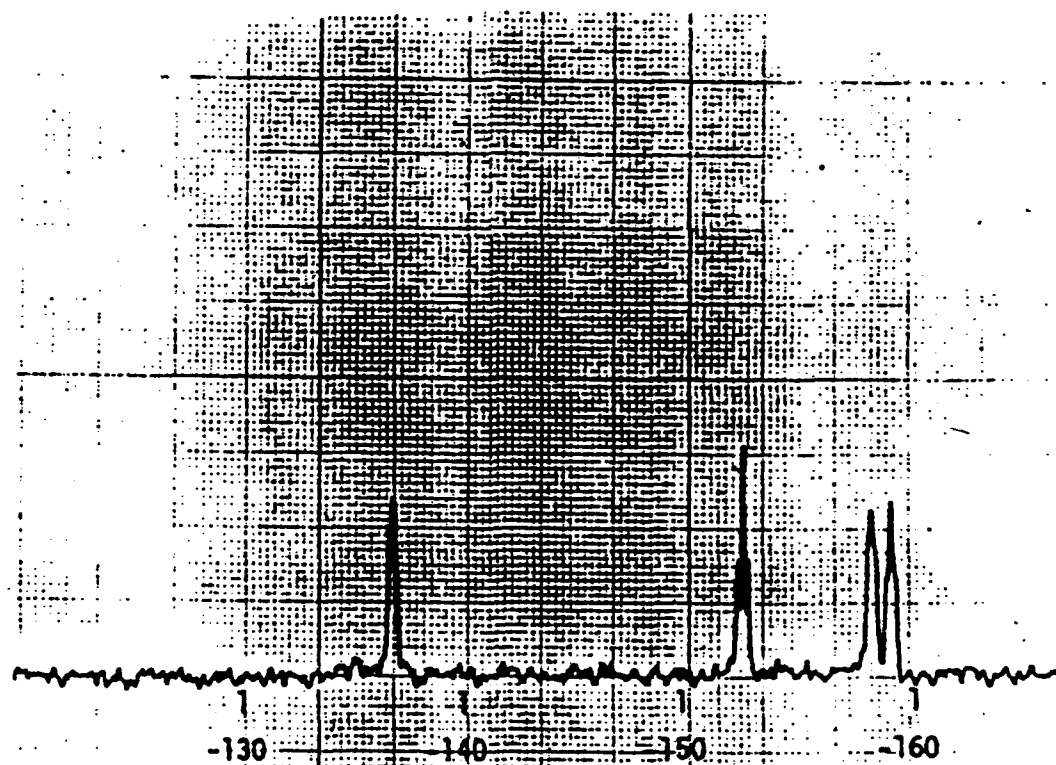
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DB ATT = 3
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AZ = 1
SW = +/- 50000.0
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RG = 10 USEC
SC = 10 USEC
TL HIGH POWER ON
F2= 361.004524
BB MODULATION ON
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SF = 336.720170
EPR = 3.00
PAM = 337.5
PDM = 43.4
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SCALE = 1350.25 Hz/CH
= 4.0000 PPM/C

The high resolution mass spectrum of the final product contains a parent peak at 577.9588.





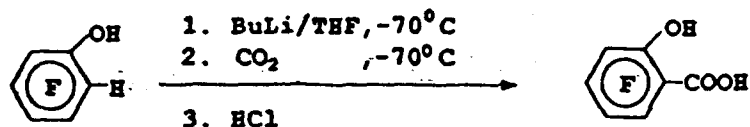
¹⁹F NMR Spectrum of the Defluorinated Product



This is a very interesting case. Two extra fluorine were removed and we obtained the very interesting perfluoro furan compound shown above. We are now think we have been successful in applying it to a four-membered perfluoro cyclohexyl ether (page 7) (degree of polymerization four) and we shall see if we get the furan structure on that one as well. If successful it should produce an oligomer containing three furan units.

Preparation of Perfluorinated Salicylic Acid

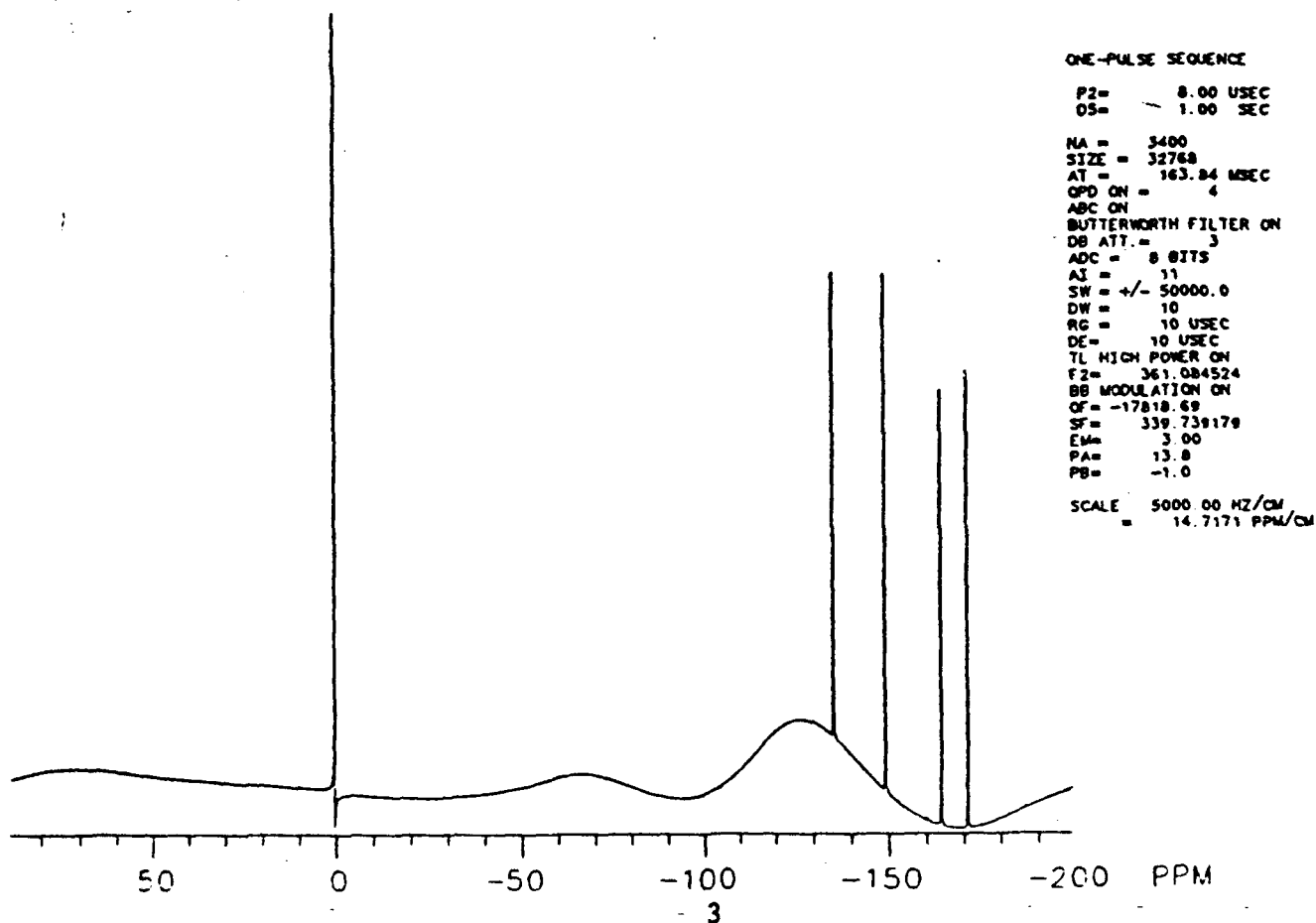
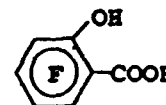
By first synthesizing in 60% yield the perfluoro aromatic precursor, namely 1-hydroxy-2-fluorobenzene, we then synthesized 2,3,4,5-tetrafluoro-6-hydroxybenzoic acid by the following procedure:



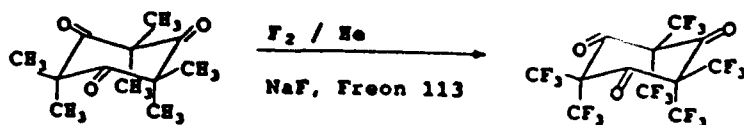
This step resulted in perfluorinated salicylic acid in 90% yield. Esters of salicylic acid are currently the most widely used lubrication antifriction additives to motor oils. We are now experimenting with making substituted hydrocarbon ester tails with one chlorine to make the product more soluble in polyalphaolefins and petroleum oils.

The ^{19}F NMR Spectrum of Perfluoro Salicylic Acid

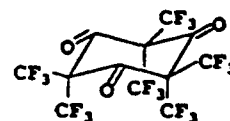
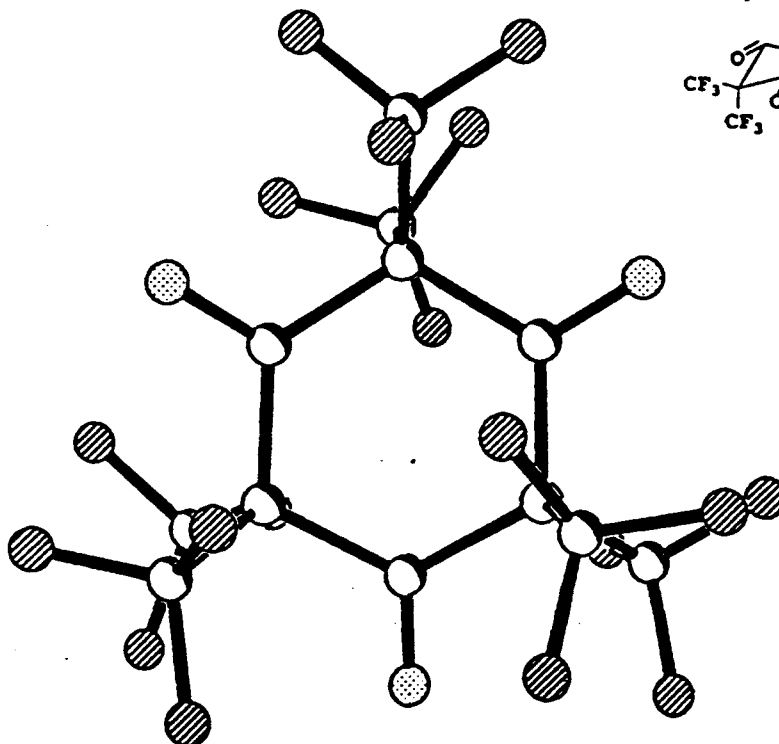
S060 . 002 MJB 22JUL93
JNG, D6-ACETONE, CFCL3



Very unusual perfluoro polyketone structures have been prepared by Dr. Kuangsen Sung. In particular, Dr. Sung has prepared a perfluorinated ketone from a hydrocarbon starting material in 72% yield.

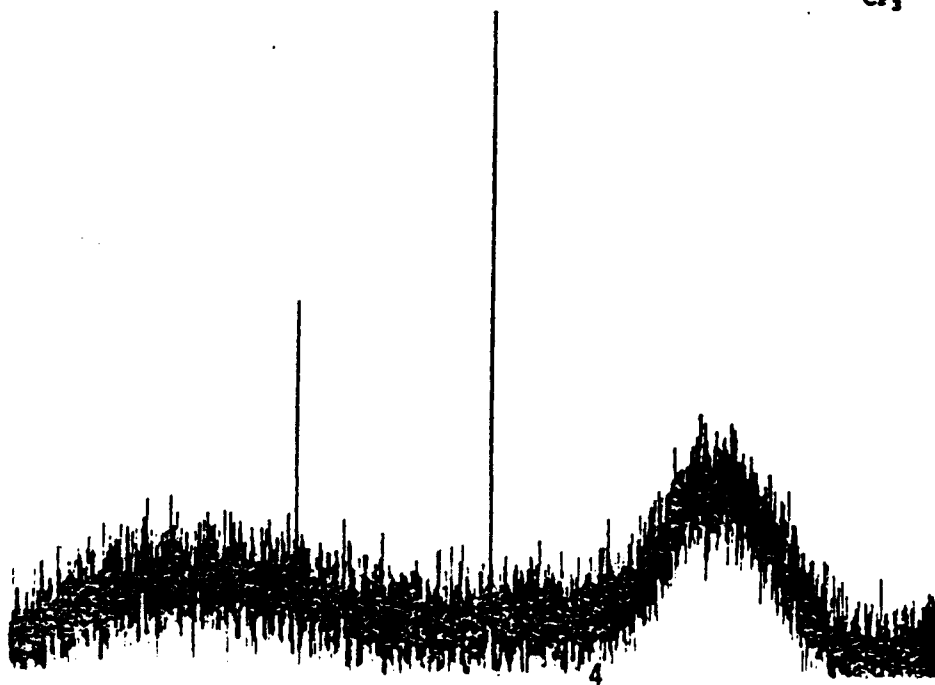
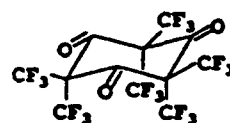


We have also obtained the crystal structure of this unusual new fluorocarbon material.



The ^{19}F NMR Spectrum of the Perfluorinated Ketone

SD70A. 002 BAS 25AUG93
JN3. ACETONE-D6, CFCL3, F19



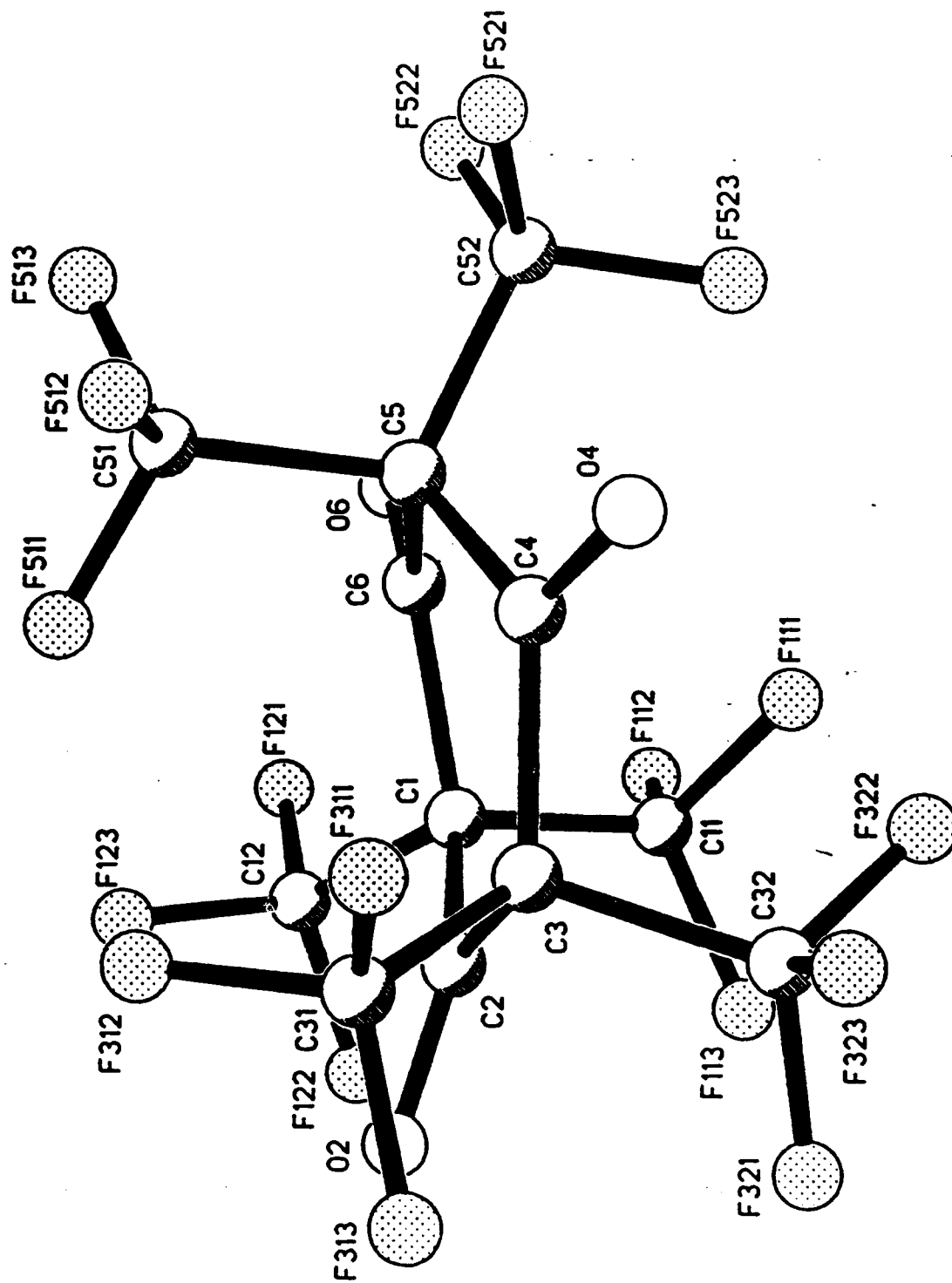
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ABC ON
BUTTERWORTH FILTER ON
DB ATT. = 3
ADC = 12.8175
AI = 1
SW = +/- 50000.0
DSW = 18
RG = 10 USEC
DE = 10 USEC
TL HIGH POWER ON
F2 = 341.084524
BB MODULATION ON
CF = -17654.26
SF = 339.739179
EM = 3.00
PA = 159.1
PB = 54.8

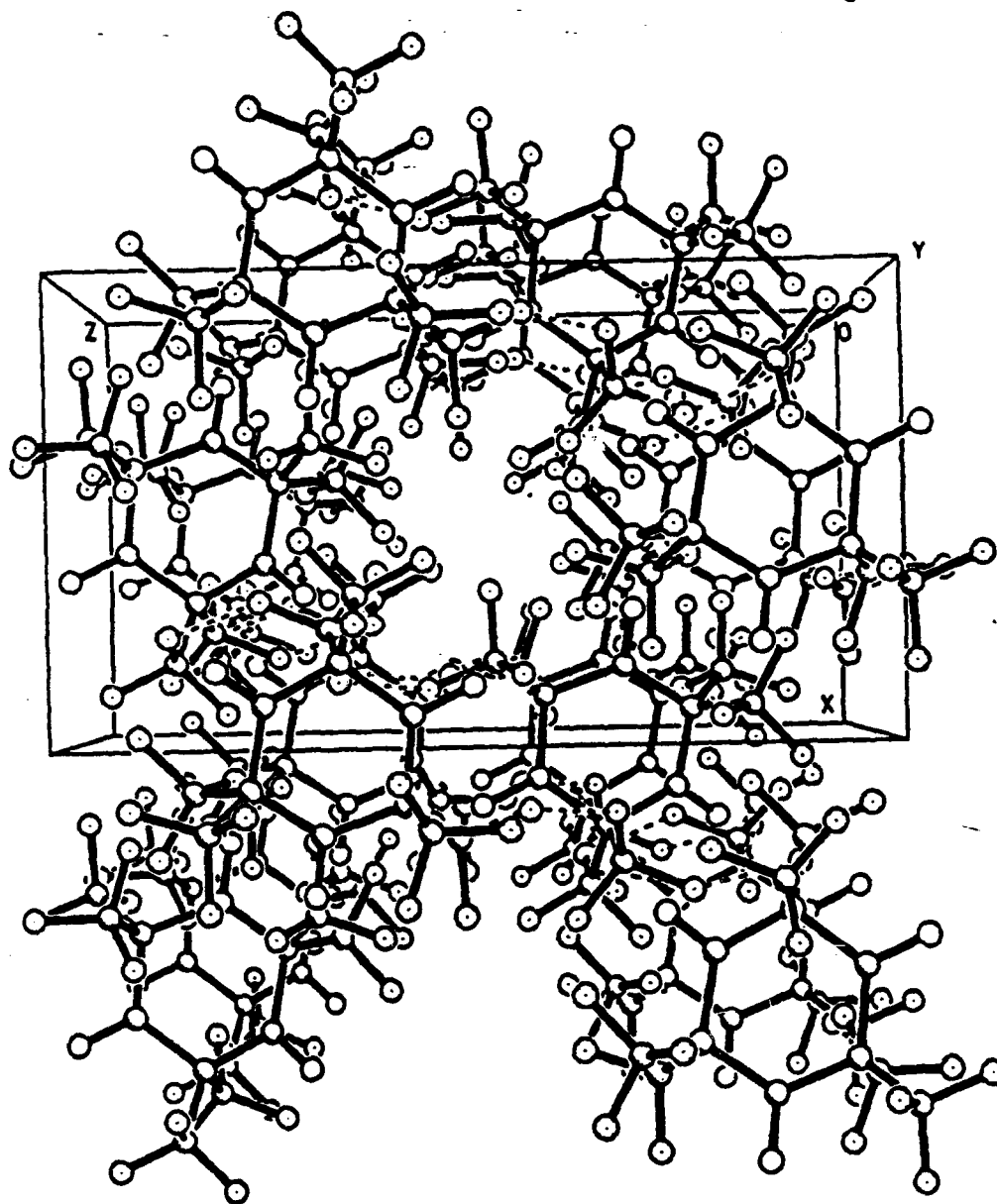
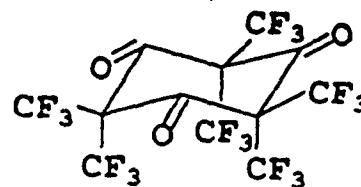
SCALE = 5000.00 HZ/CM
= 14.7171 PPM/CM

C12 F18 03

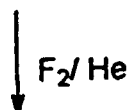
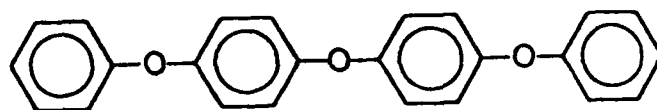


We think the zeolitic solid state structure of this very interesting perfluoro polyketone is most unusual and there may be interesting chemistry associated with the pockets of such a material.

Zeolitic Structure of



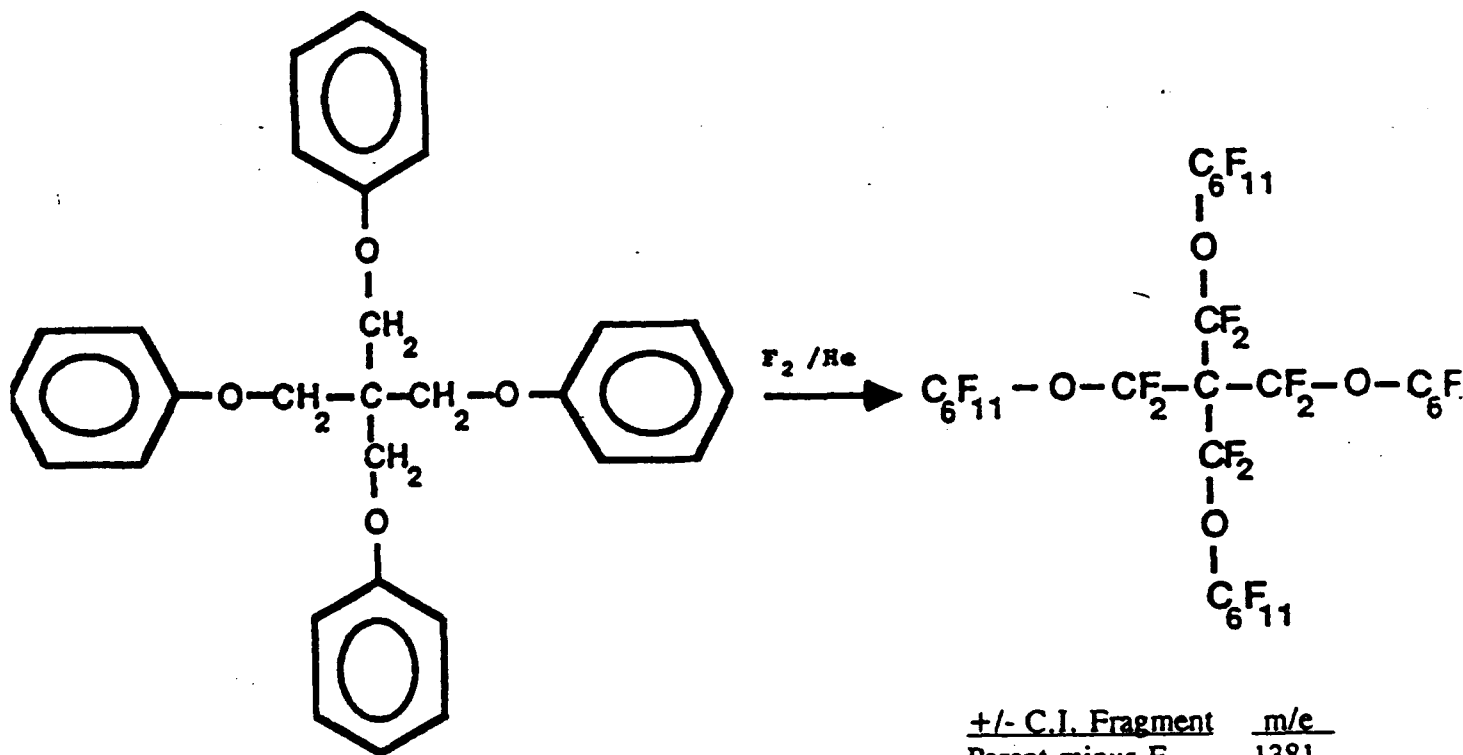
A number of other perfluoro organometallic compounds have been prepared for their conversion to perfluoro aromatic analogs.



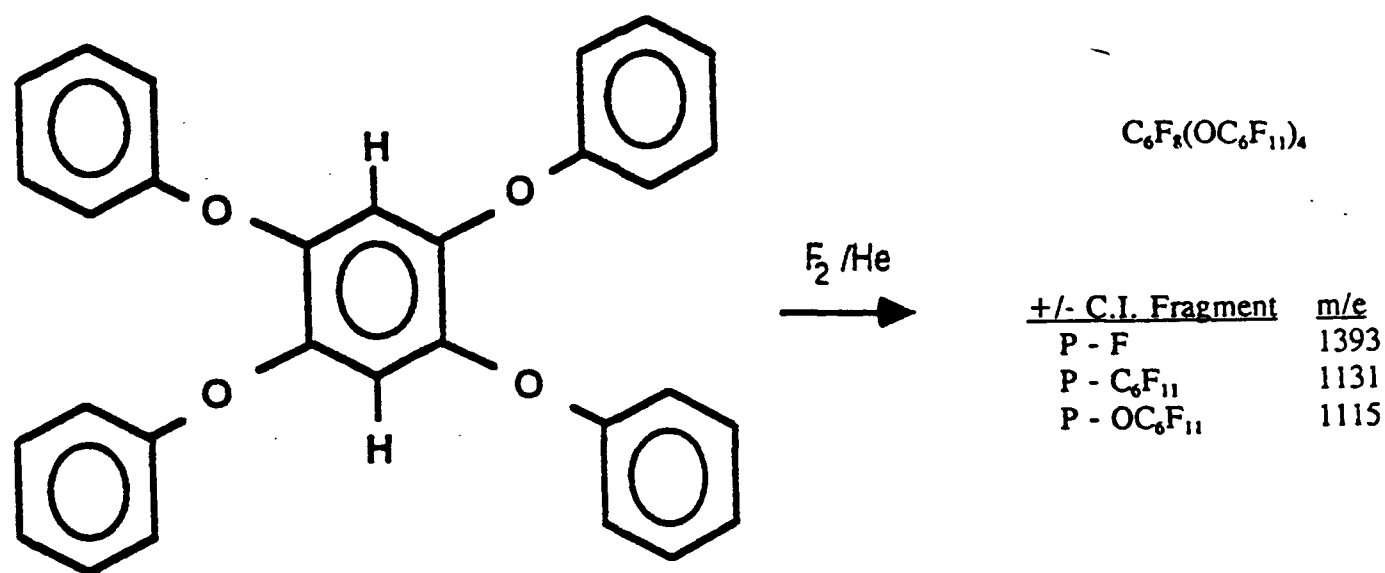
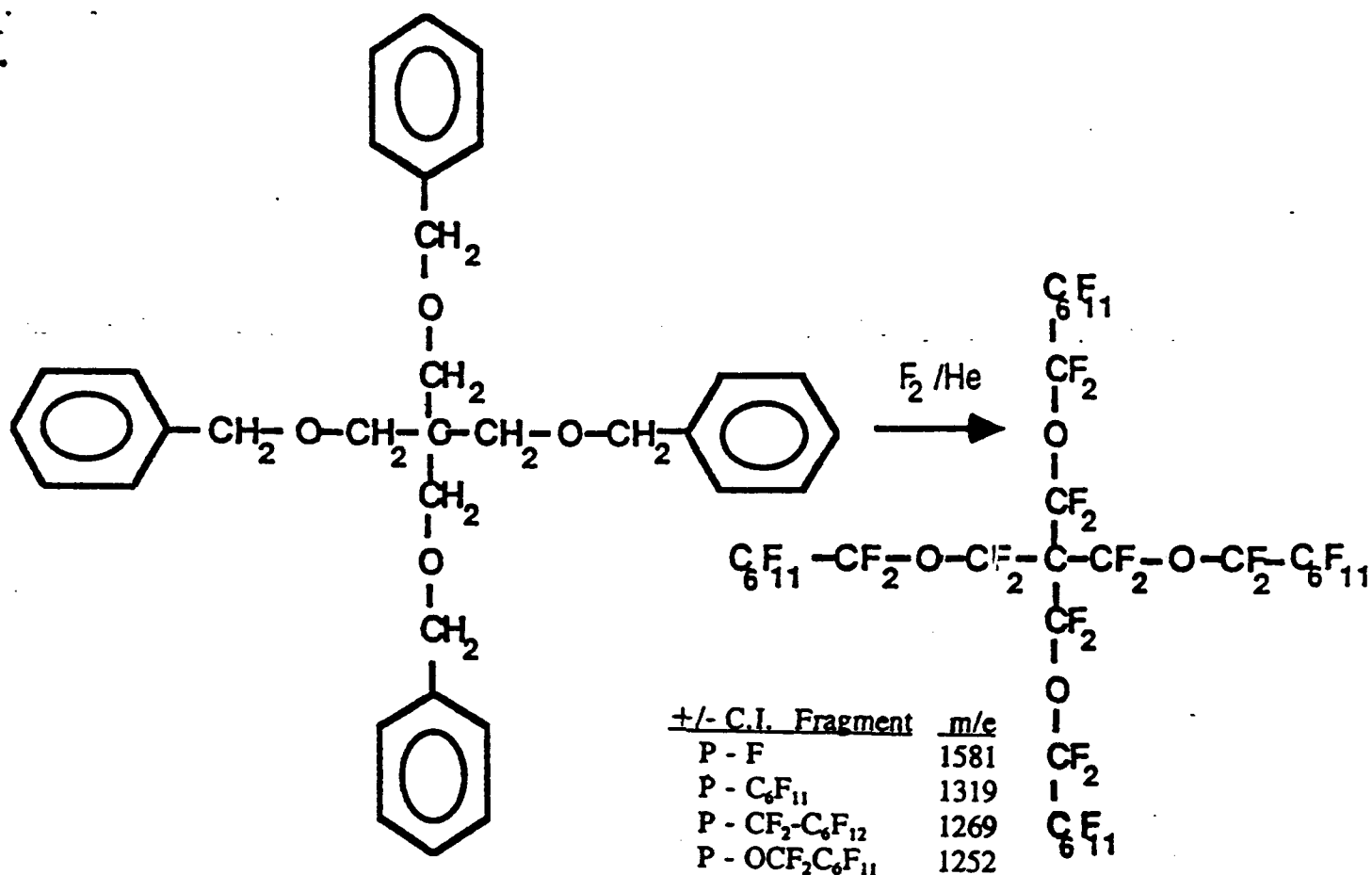
F

This structure is very interesting for under certain circumstances we hope to get the furan structure and with other reagents we hope to get the perfluorinated phenyl material. The perfluorinated cyclohexyl analog is already in hand.

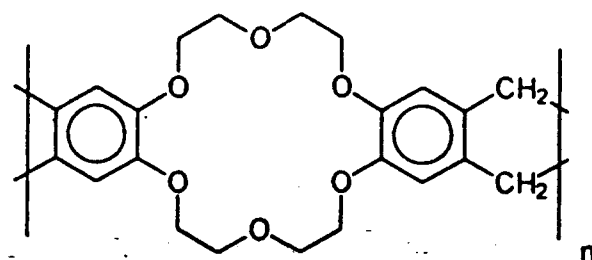
<u>+/- C.I. Fragment</u>	<u>m/e</u>
P - F	1115
P - C_6F_{11}	853
P - OC_6F_{11}	837
P - $C_6F_{10}OC_6F_{11}$	575
P - $OC_6F_{10}OC_6F_{11}$	559



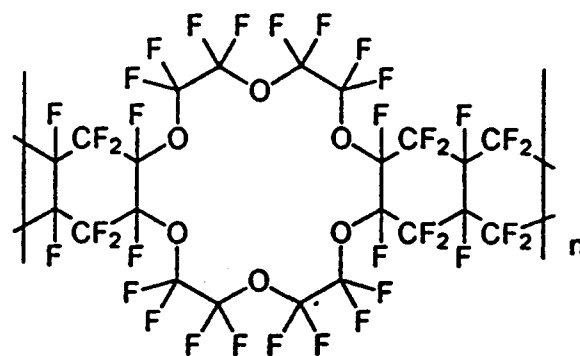
<u>+/- C.I. Fragment</u>	<u>m/e</u>
Parent minus F	1381
P - C_6F_{11}	1119
P - OC_6F_{11}	1103
P - $CF_2-O-C_6F_{11}$	1053



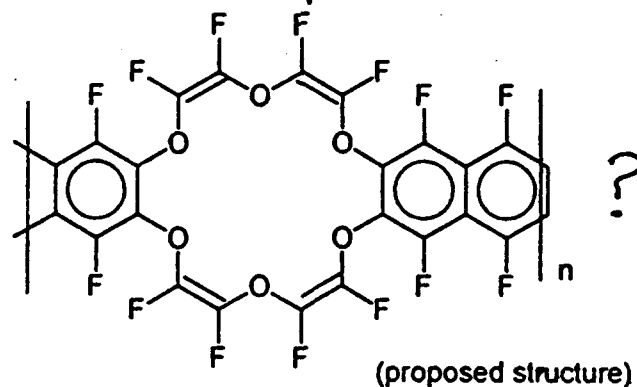
Mr. Han-Chao Wei of our research program has fluorinated this interesting crown ether polymer. We are very interested in exploring the defluorination process to see if we obtain the defluorinated material as indicated in the proposed structure.



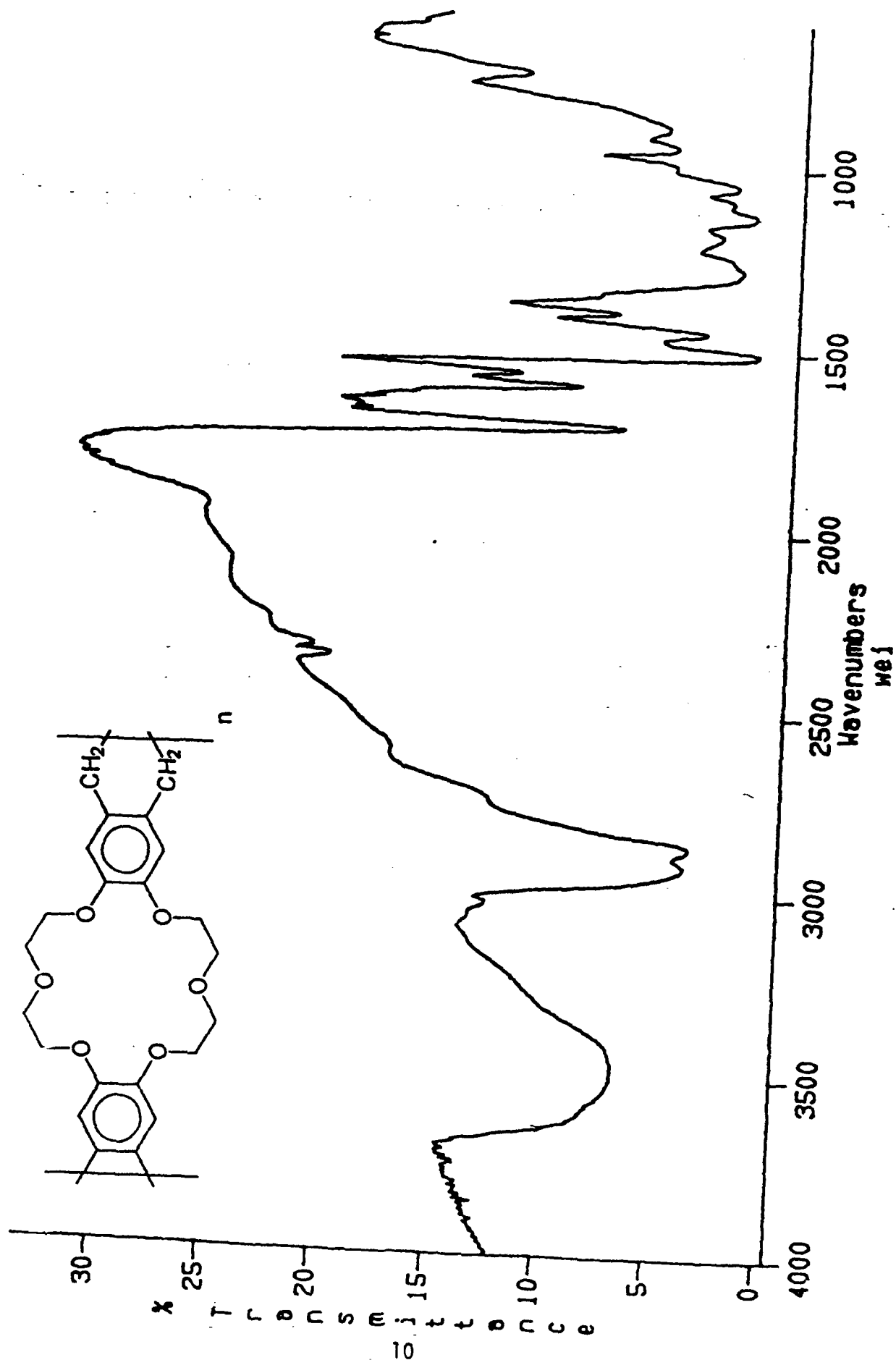
↓
F₂/He
NaF/CCl₄



? ↓ defluorination process



He(cm ³ /min.)	F ₂ (cm ³ /min.)	Time(hr.)
50	0	0.5
50	2	24
25	2	24
10	3	24
3	3	24
0	3	24
25	0	0.5



Elemental analyses of the Perfluoro-crown ether-polymer

	<u>Found</u>	<u>Calculated</u>
Carbon —	24.38% 24.34%	24.92%
Fluorine —	64.68% 64.59%	65.11%
Oxygen —	----	9.97%

***Elemental analyses were done by Schwarzkopf Microanalytical Laboratory
in New York**