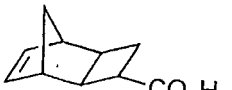
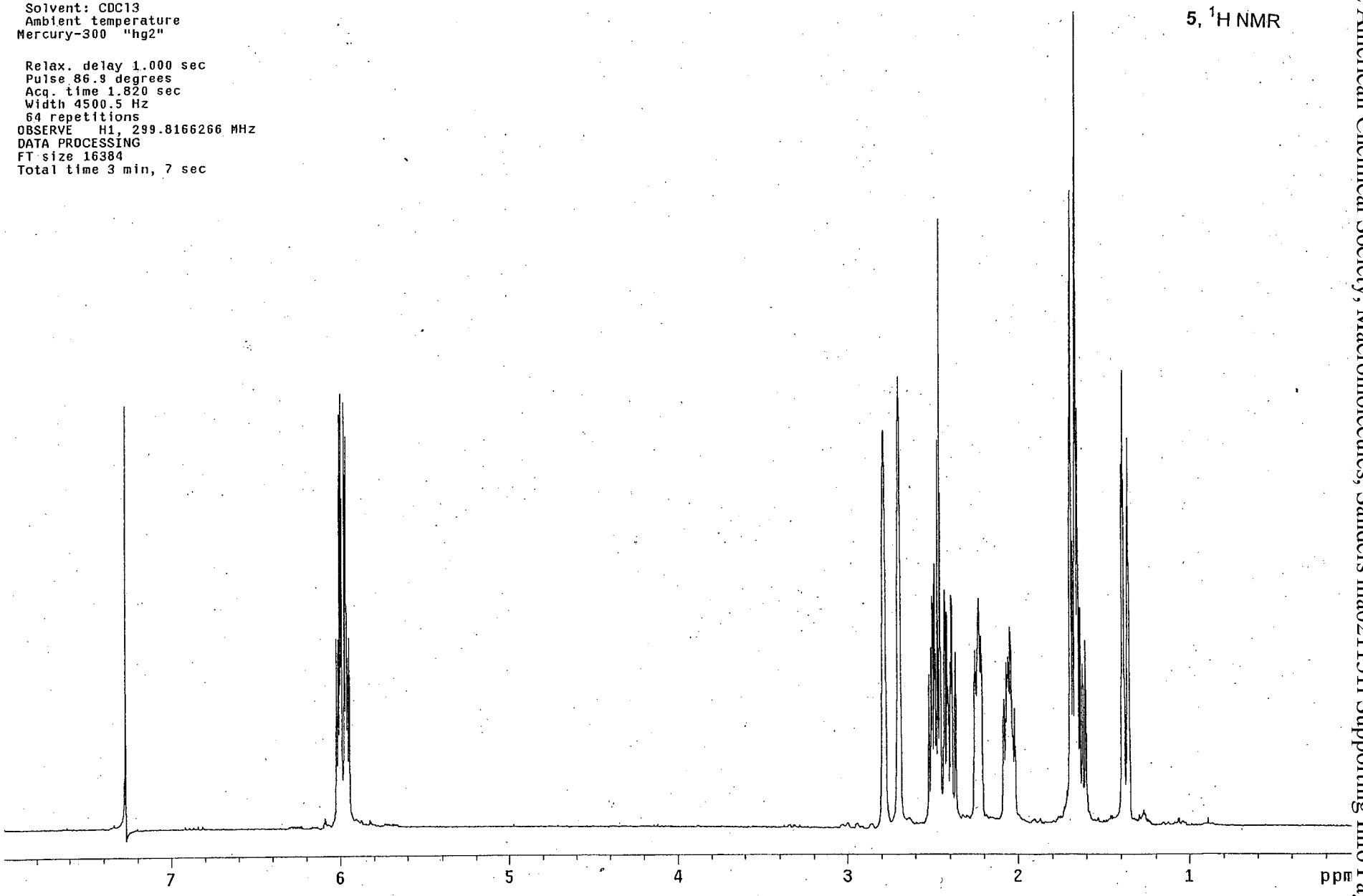
STANDARD 1H OBSERVE

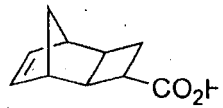
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300 "hg2"

Relax. delay 1.000 sec
Pulse 86.9 degrees
Acq. time 1.820 sec
Width 4500.5 Hz
64 repetitions
OBSERVE H1, 299.8166266 MHz
DATA PROCESSING
FT size 16384
Total time 3 min, 7 sec



5, ^{1}H NMR



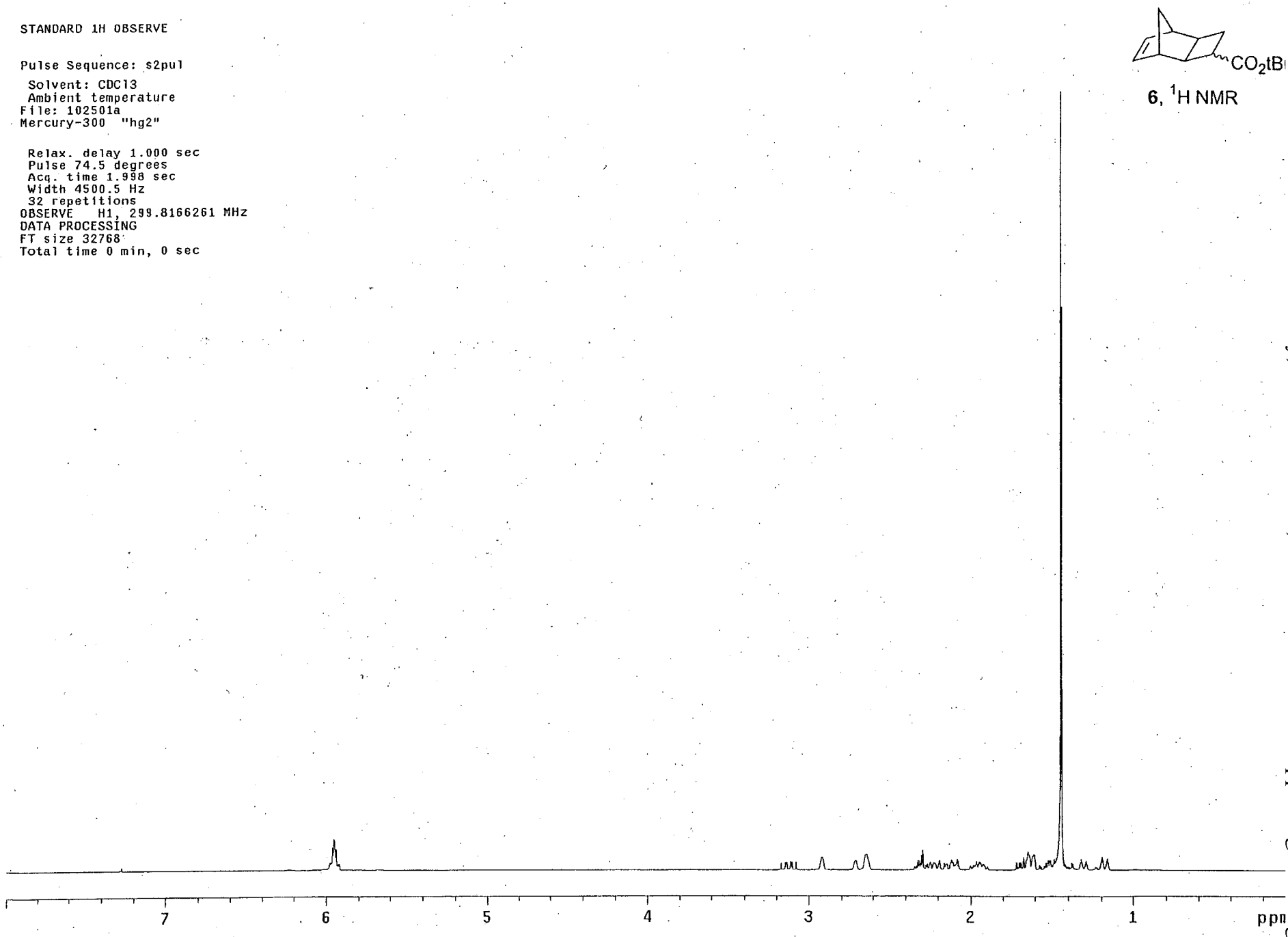


5, ^{13}C NMR

Archive directory: /export/home/grubds/vnmrsys/data
Sample directory:
File: CARBON

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300 "hg1"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.3 Hz
64 repetitions
OBSERVE C13, 75.4013662 MHz
DECOUPLE H1, 299.8675693 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 25 sec



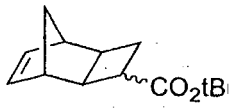
6, ^{1}H NMR

Archive directory: /export/home/grubds/vnmrsys/data
Sample directory:
File: CARBON

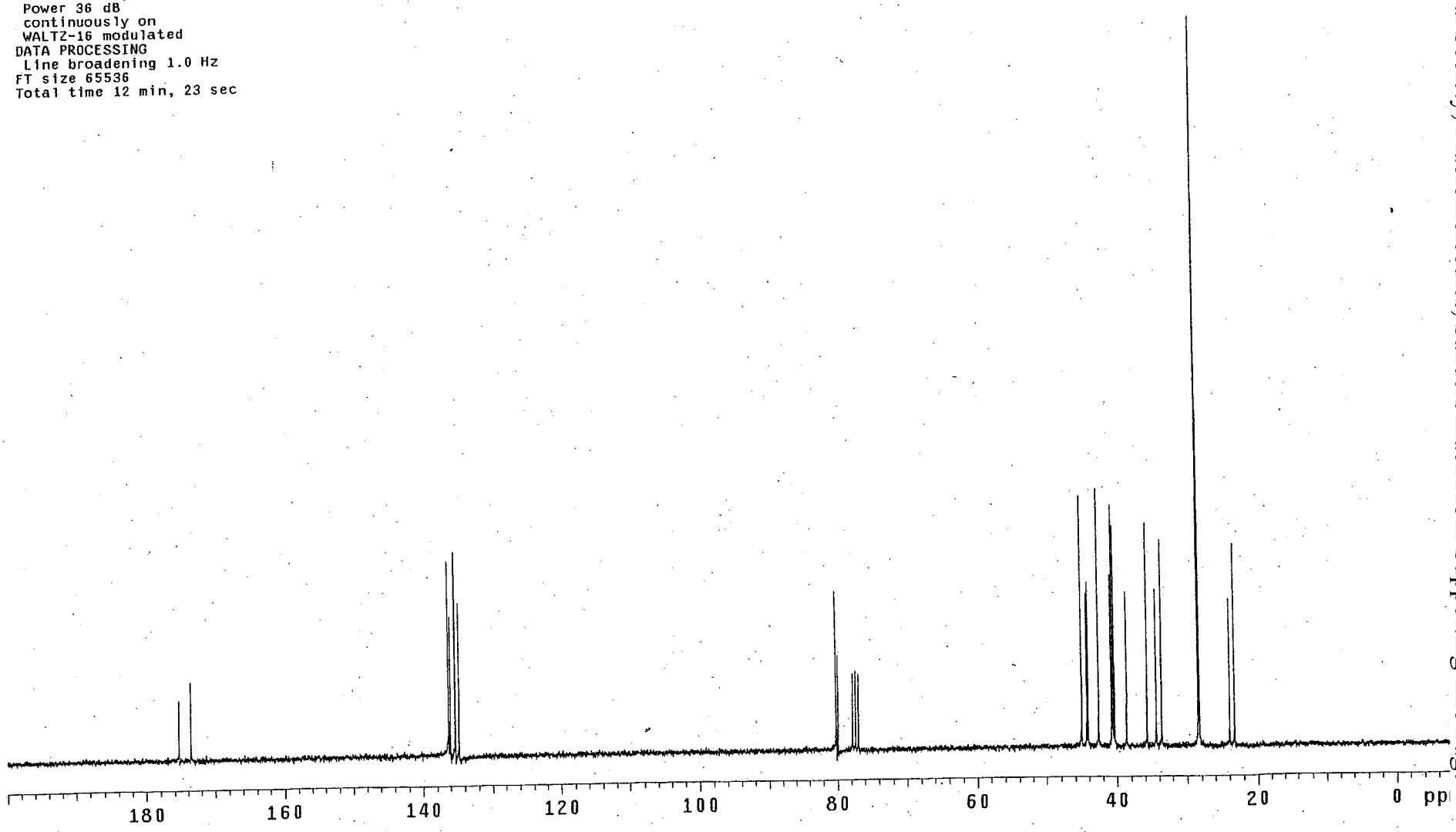
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-300 "hg1"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 15834.3 Hz
64 repetitions
OBSERVE C13, 75.4013662 MHz
DECOUPLE H1, 299.8675693 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 12 min, 23 sec



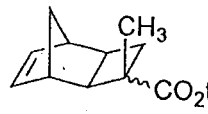
6, ^{13}C NMR



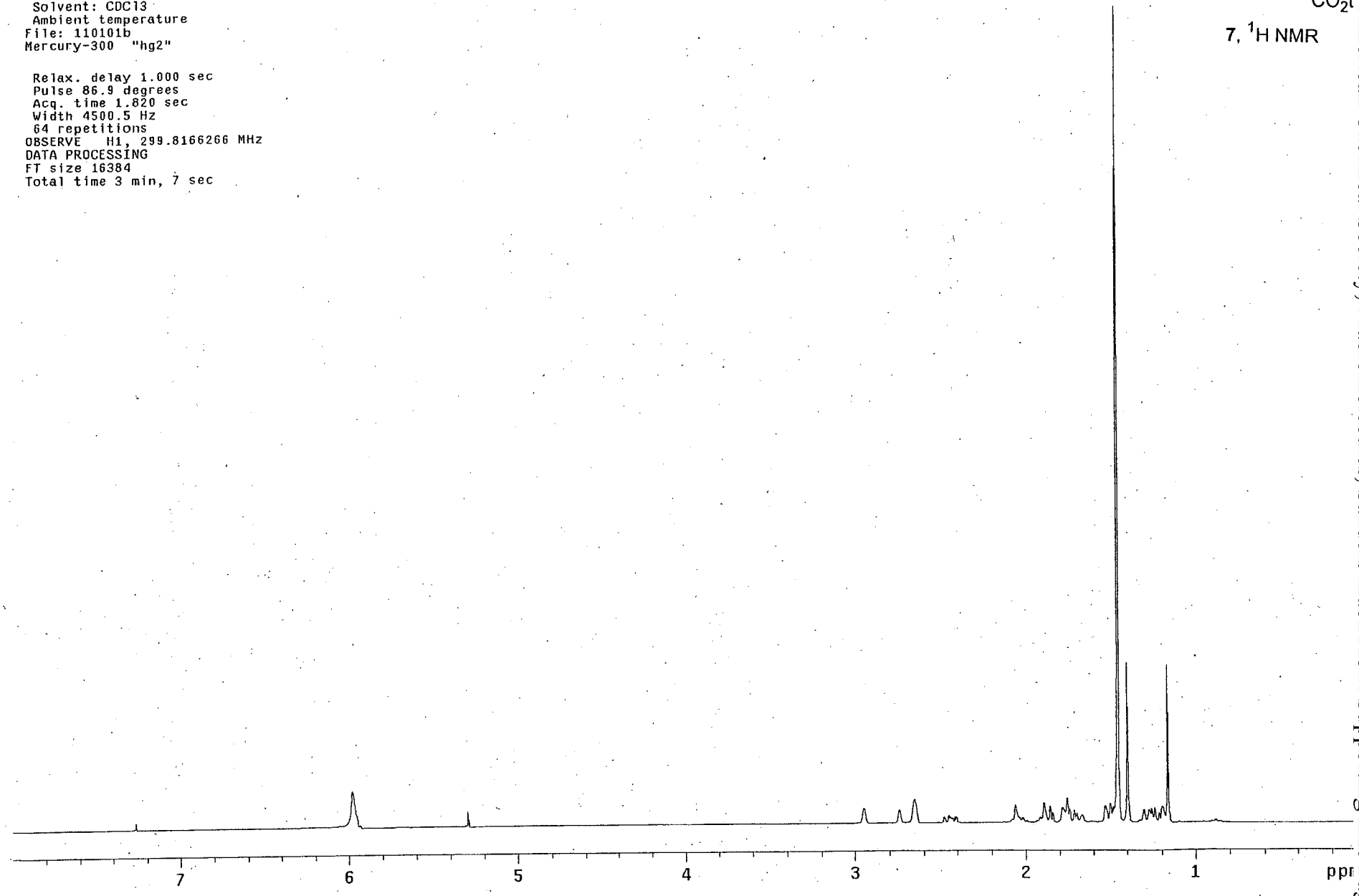
STANDARD 1H OBSERVE

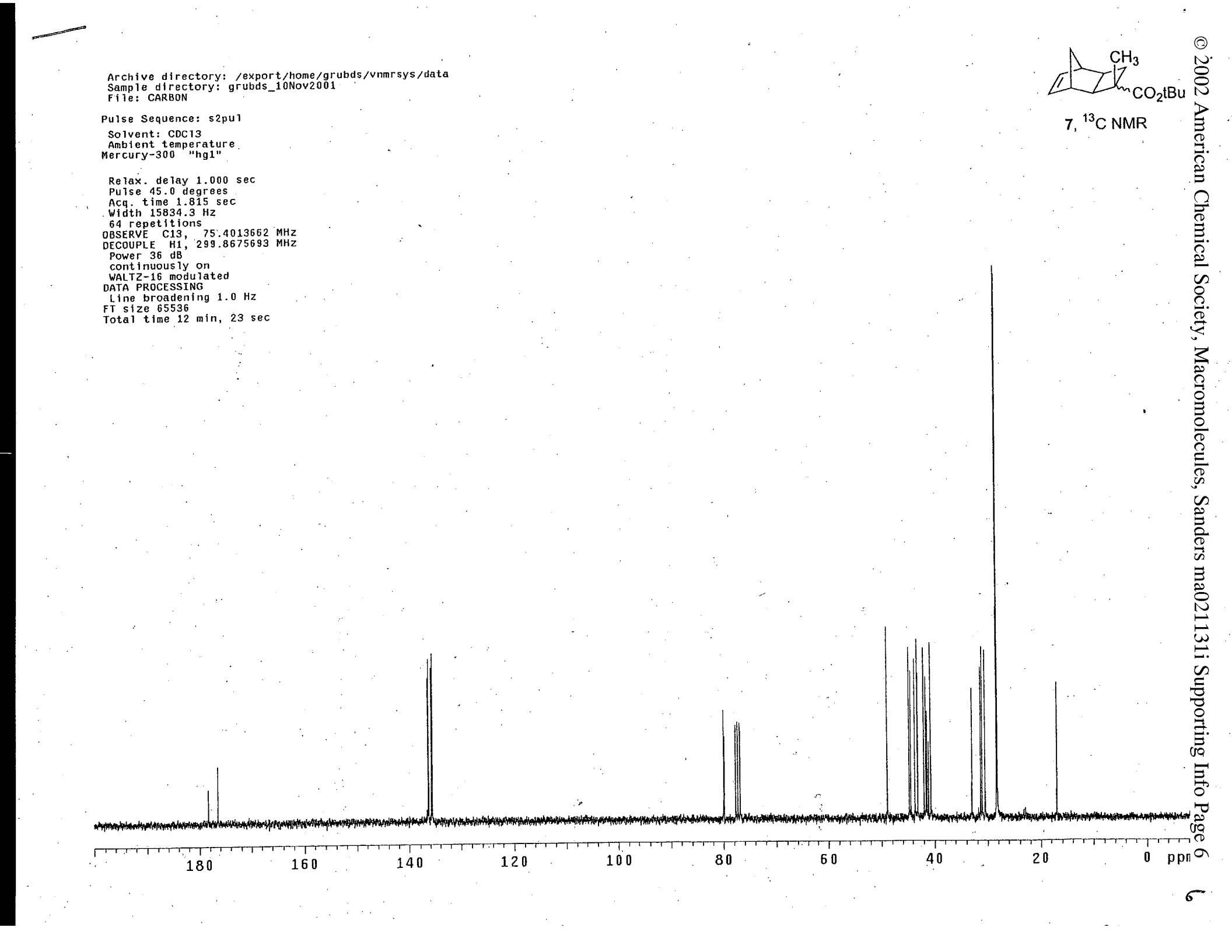
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 110101b
Mercury-300 "hg2"

Relax. delay 1.000 sec
Pulse 86.9 degrees
Acq. time 1.820 sec
Width 4500.5 Hz
64 repetitions
OBSERVE H1, 299.8166266 MHz
DATA PROCESSING
FT size 16384
Total time 3 min, 7 sec



7, ^{1}H NMR

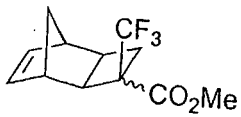




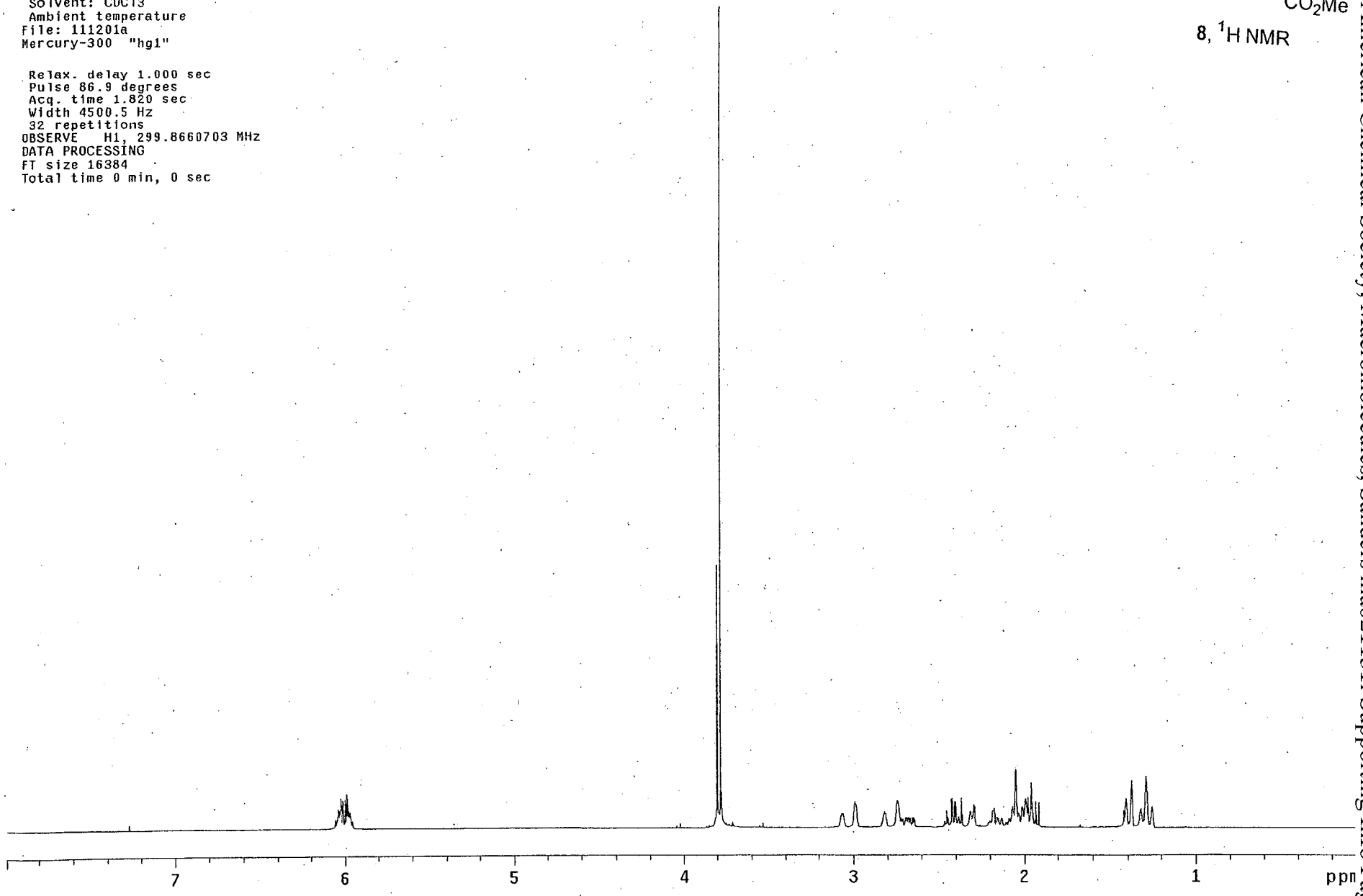
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 111201a
Mercury-300 "hg1"

Relax. delay 1.000 sec
Pulse 86.9 degrees
Acq. time 1.820 sec
Width 4500.5 Hz
32 repetitions
OBSERVE H1, 299.8660703 MHz
DATA PROCESSING
FT size 16384
Total time 0 min, 0 sec



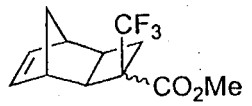
8, ^{1}H NMR



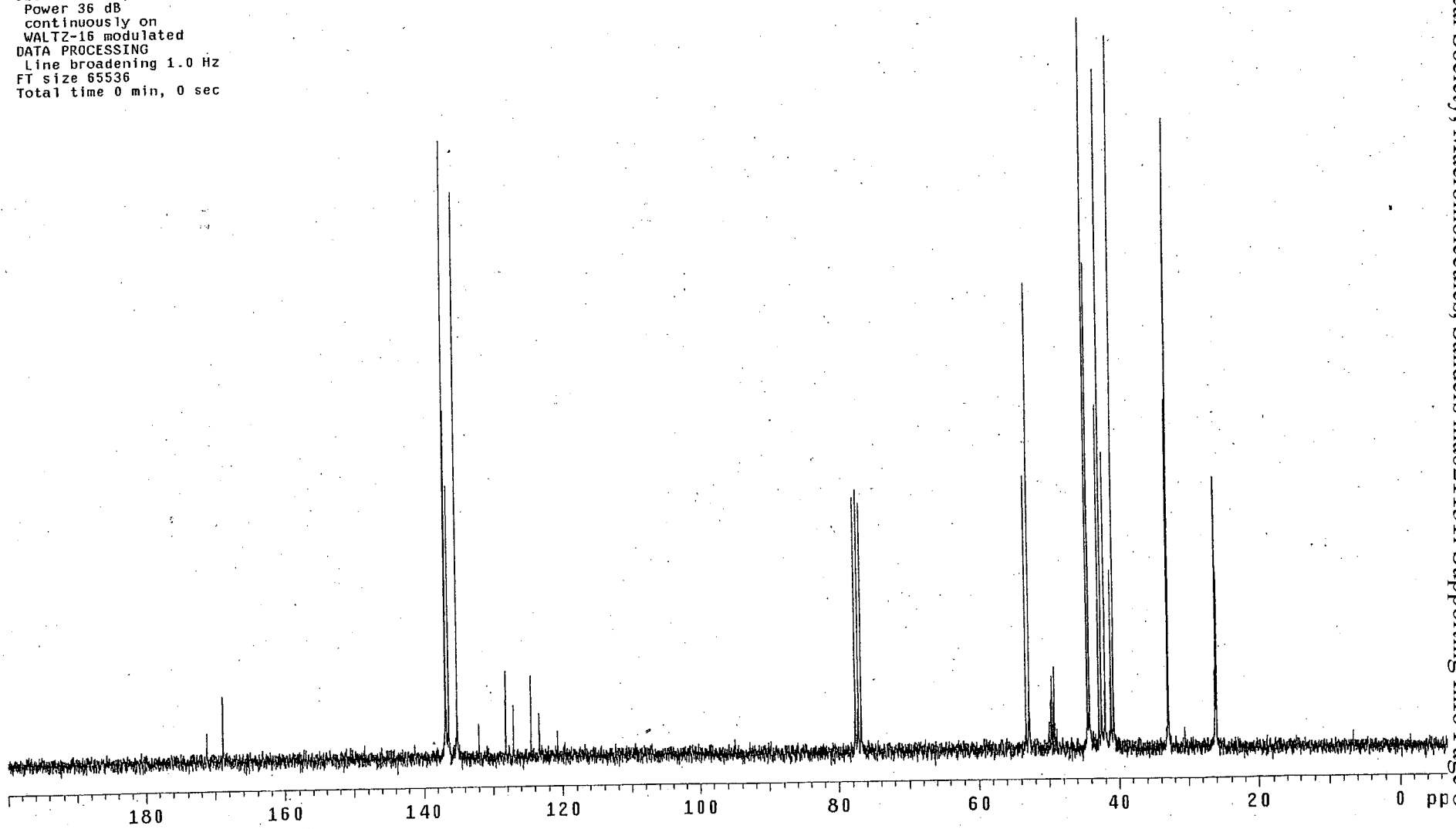
Archive directory: /export/home/grubds/vnmrsys/data
Sample directory:
File: CARBON

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300 "hg1"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 15834.3 Hz
256 repetitions
OBSERVE C13, 75.4013662 MHz
DECOUPLE H1, 299.8675693 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 0 min, 0 sec



8, ^{13}C NMR



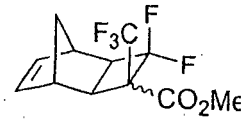
8, ^{19}F NMR

19F OBSERVE
STANDARD PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-300 "hg2"

Relax. delay 4.000 sec
Pulse 123.8 degrees
Acq. time 0.300 sec
Width 50000.0 Hz
8 repetitions
OBSERVE F19, 282.1096548 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 0 min, 0 sec



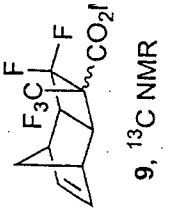
9, ^{1}H NMR

Archive directory: /export/home/grubds/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

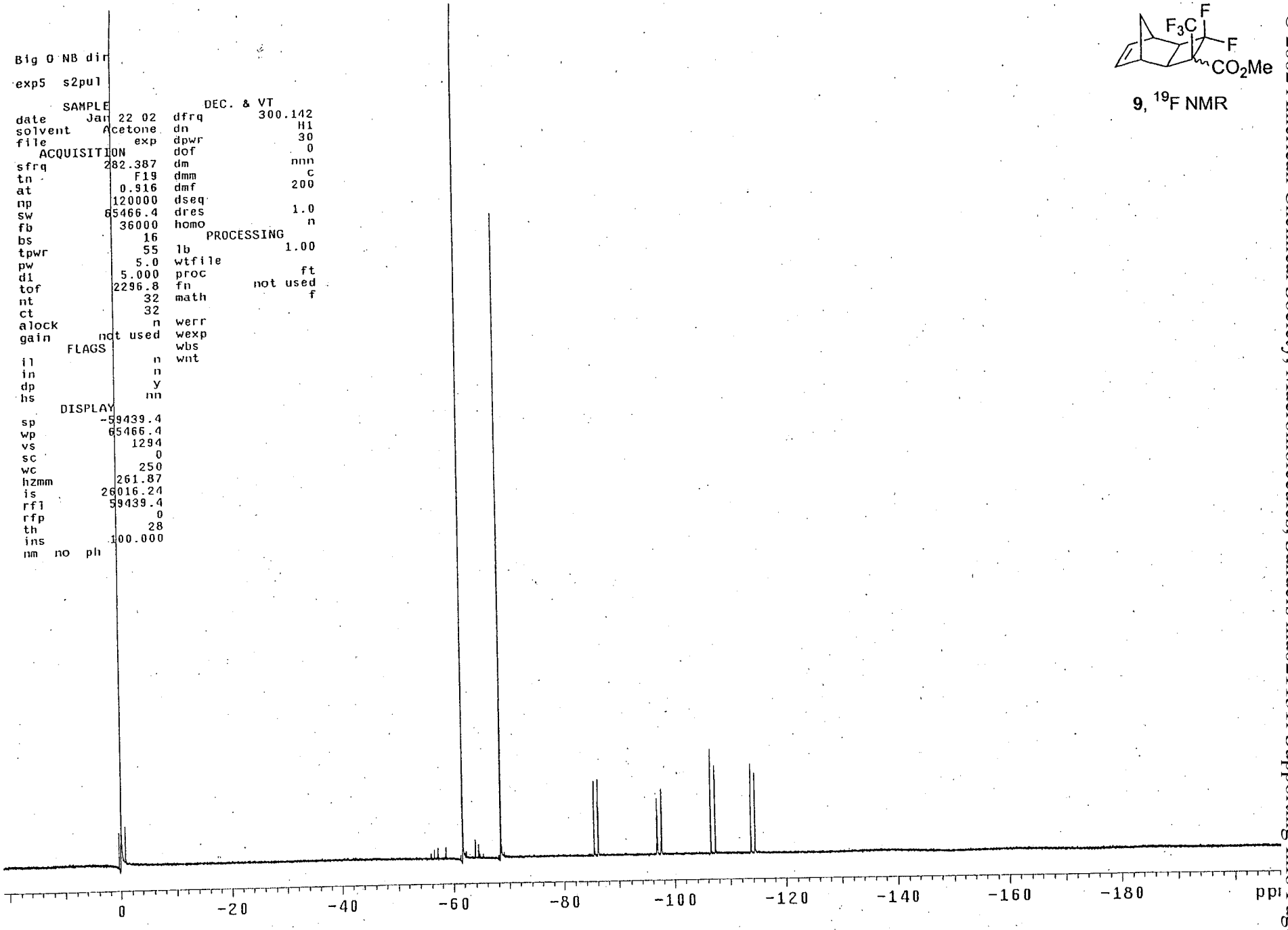
Solvent: CDCl3
Ambient temperature
File: 92800a
Mercury-300 "hg2"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4797.1 Hz
16 repetitions
OBSERVE H1, 299.8166284 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec

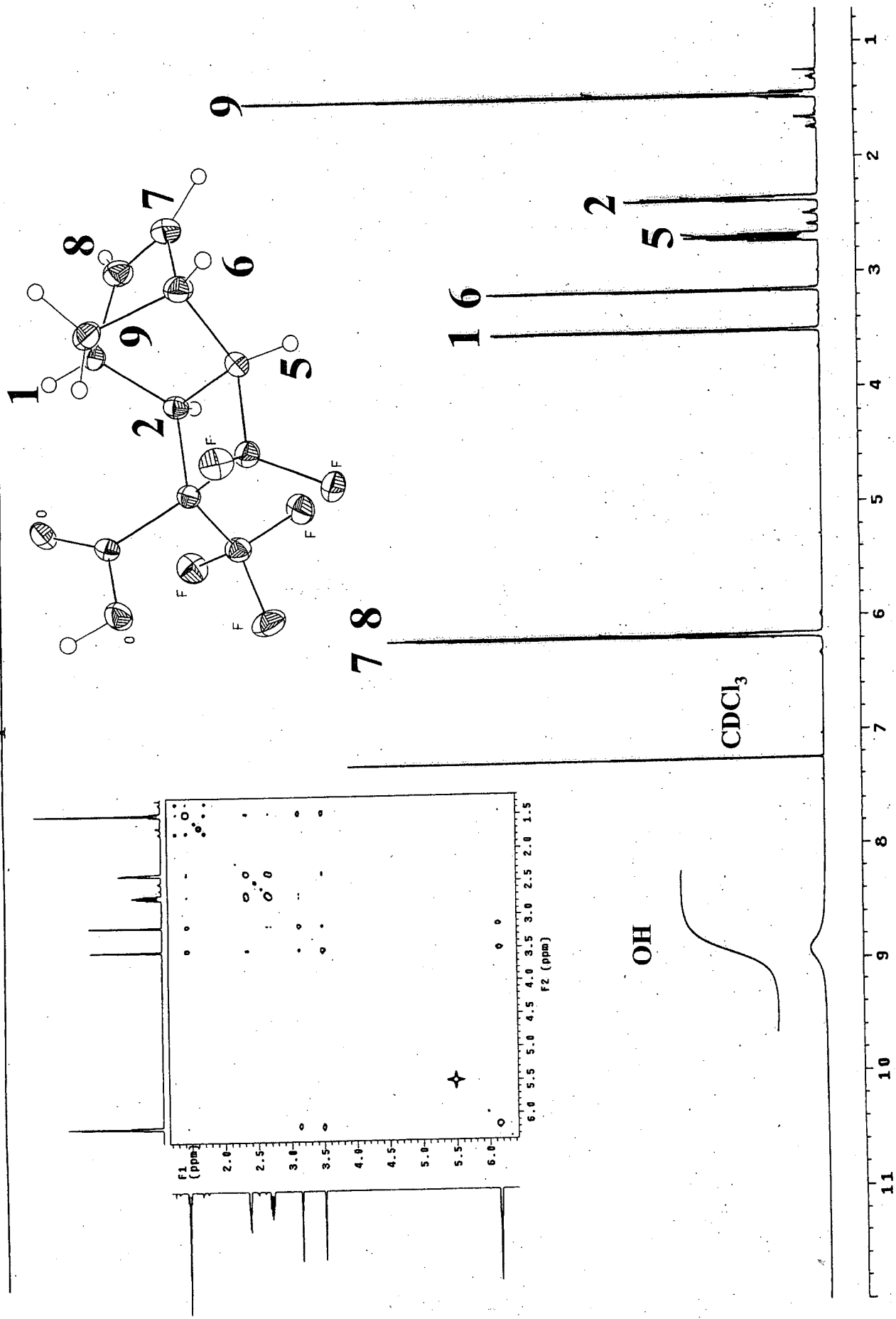


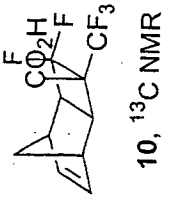
9, ^{13}C NMR

Brian Osborn//SF-TCN//B2-117
Solvent: Benzene
Ambient temperature
UNITYplus-300 "nmr2"
PULSE SEQUENCE
Relax. delay 2.000 sec
Pulse 36.0 degrees
Acq. time 1.777 sec
Width 18009.9 Hz
512 repetitions
OBSERVE C13, 75.4699983 MHz
DECOUPLE H1, 300.1409528 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
Single precision data
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 32 minutes



9, ^{19}F NMR





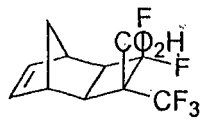
10, ^{13}C NMR

rh2-218

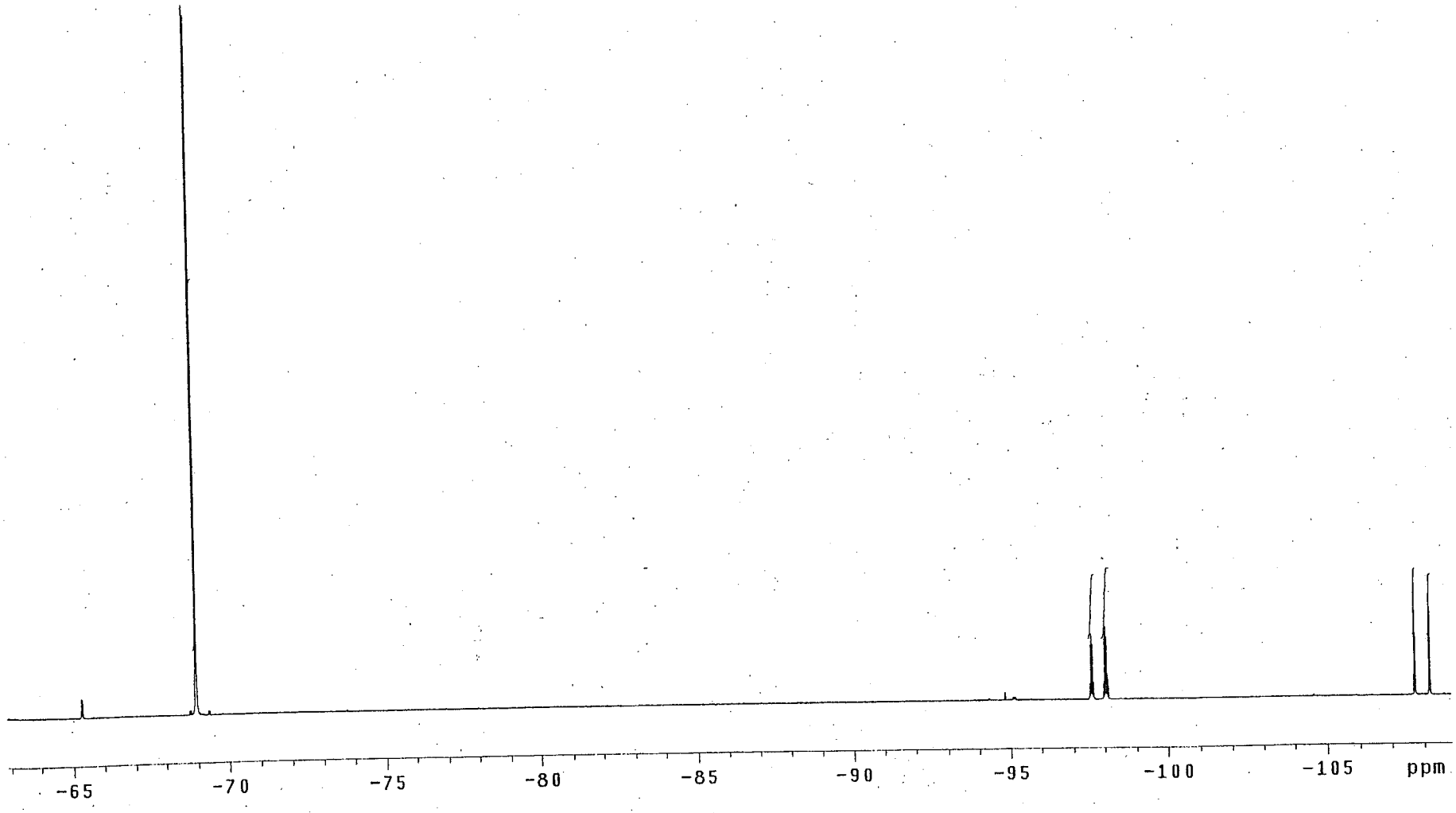
Pulse Sequence: s2pul

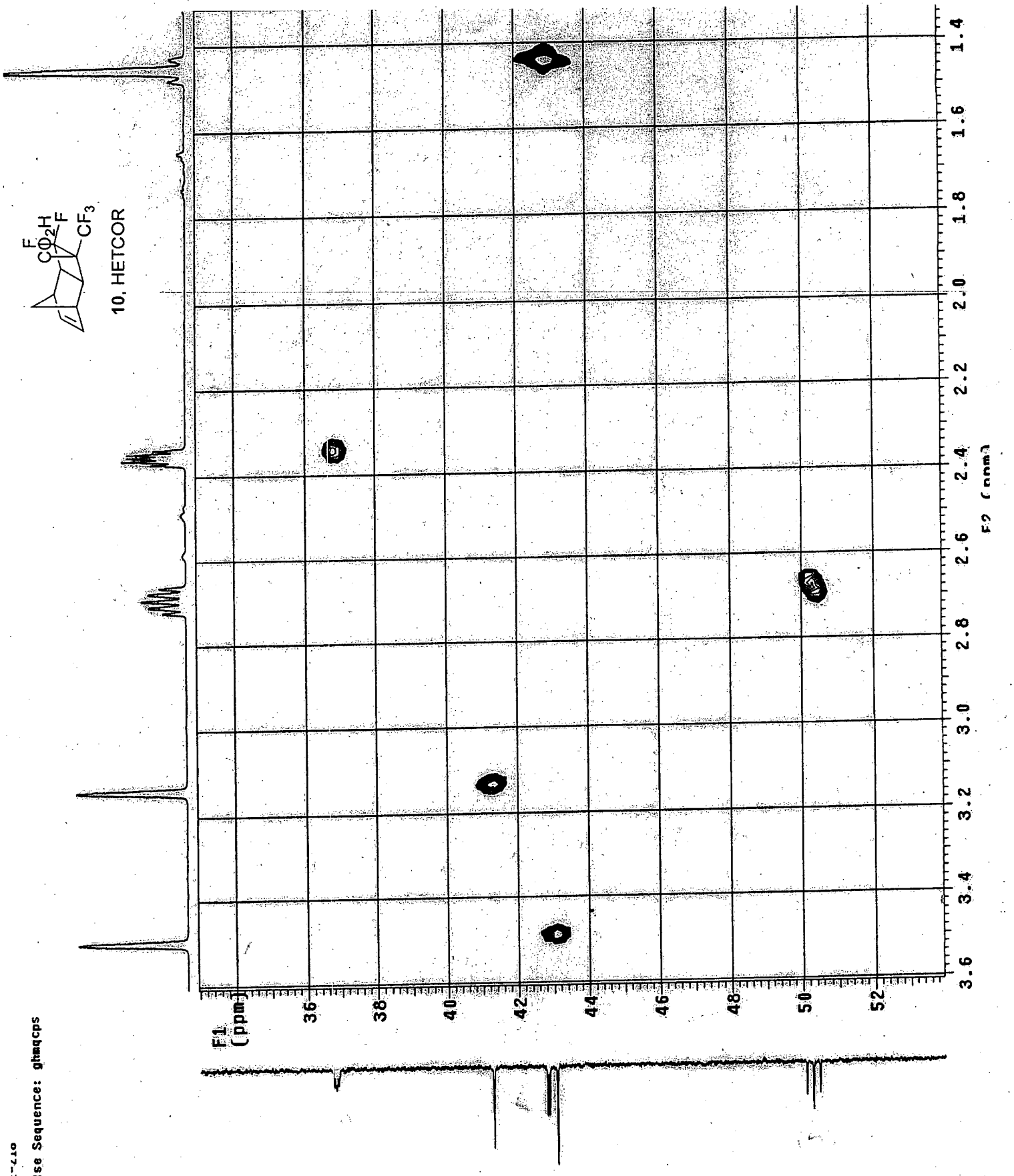
Solvent: CDCl3
Temp. 27.0 C / 300.1 K
INOVA-500 "nmr1"

PULSE SEQUENCE
Relax. delay 2.000 sec
Pulse 21.2 degrees
Acq. time 3.999 sec
Width 23902.0 Hz
16 repetitions
OBSERVE F19, 469.8554519 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 262144
Total time 1 min, 36 sec



10, ^{19}F NMR




10, HETCOR
F
CO2H
F
CF3
F1 (ppm)
36
38
40
42
44
46
48
50
52
3.6
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
Pulse Sequence: ghsqcps

X-ray Experimental for $C_{11}H_9F_5O_2$: Crystals grew as very large prisms by slow ???. The data crystal was cut from a larger crystal and had approximate dimensions; 0.48 x 0.24 x 0.19 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoKα radiation (λ = 0.71073Å). A total of 347 frames of data were collected using ω-scans with a scan range of 1° and a counting time of 39 seconds per frame. The data were collected at –120 °C using a Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using DENZO-SMN.[1] The structure was solved by direct methods using SIR92[2] and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.[3] The hydrogen atom positions were located in a ΔF and refined with isotropic displacement parameters. The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.0455*P)^2 + (0.4828*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.102, with R(F) equal to 0.0455 and a goodness of fit, S, = 0.998. Definitions used for calculating R(F), $R_w(F^2)$ and the goodness of fit, S, are given below.[4] The data were corrected for secondary extinction effects. The correction takes the form: $F_{corr} = kF_c/[1 + (6(4)x10^{-6})* F_c^2 \lambda^3/(\sin 2\theta)]^{0.25}$ where k is the overall scale factor. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[5] All figures were generated using SHELXTL/PC.[6] Tables of positional and thermal parameters, bond lengths and angles, figures and lists of observed and calculated structure factors are located in tables 1 through 6.

References

1) DENZO-SMN. (1997). Z. Otwinowski and W. Minor, Methods in Enzymology, **276**: Macromolecular Crystallography, part A, 307 – 326, C. W. Carter, Jr. and R. M. Sweets, Editors, Academic Press.

2) SIR92. (1993). A program for crystal structure solution. Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. J. Appl. Cryst. 26, 343-350.

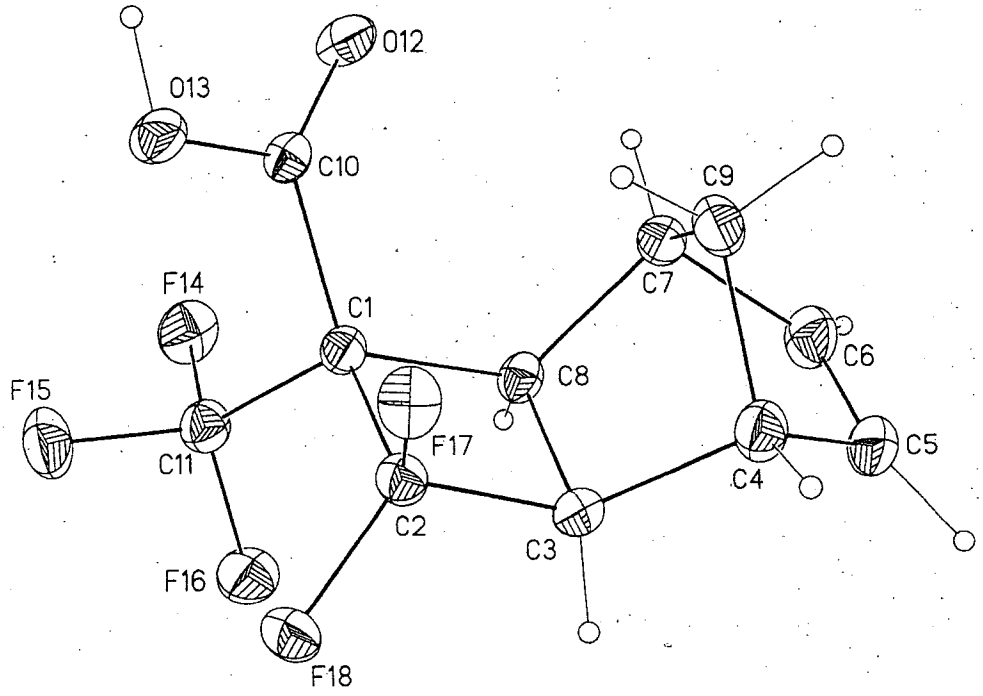
3) Sheldrick, G. M. (1994). SHELXL97. Program for the Refinement of Crystal Structures. University of Gottingen, Germany.

4) $R_w(F^2) = \{\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w(|F_o|)^4\}^{1/2}$ where w is the weight given each reflection.
$R(F) = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|\}$ for reflections with $F_o > 4(\sigma(F_o))$.
$S = [\Sigma w(|F_o|^2 - |F_c|^2)^2/(n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.

5) International Tables for X-ray Crystallography (1992). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.

6) Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Structure **1**. View of **10** showing the atom labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. Hydrogen atoms are drawn to an arbitrary size.



Structure **2**. View of the H-bound dimers formed by **10**. The dimer lies around a crystallographic inversion center at 0, 1/2, 1/2. The geometry of this interaction is: O13-H13····O12 (related by –x, 1-y, 1-z), O····O 2.697(2)Å, H····O 1.85(3)Å, O-H····O 174(2)°. Thermal ellipsoids are scaled to the 30% probability level. Hydrogen atoms are drawn to an arbitrary size.

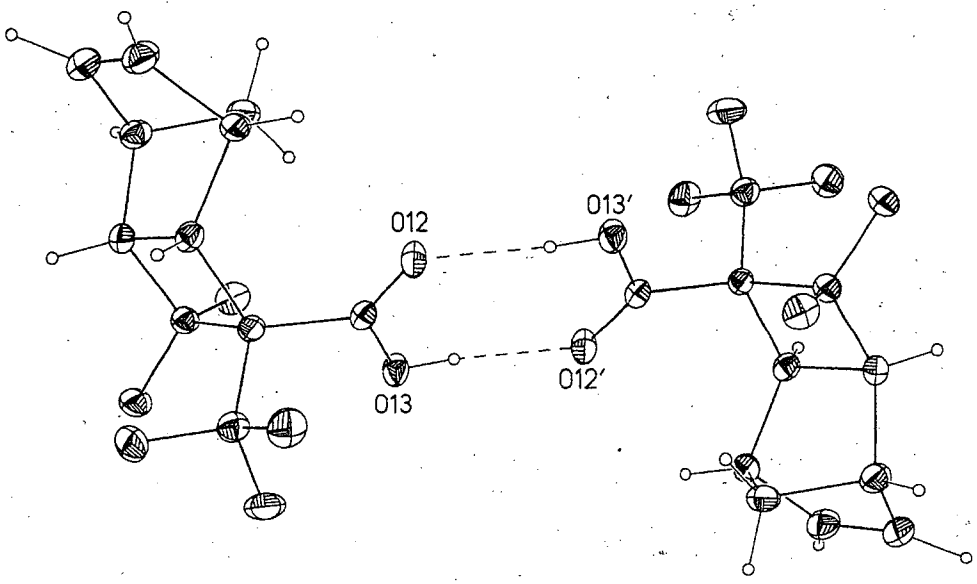


Table 1. Crystal data and structure refinement for 1.

Empirical formula	C11 H9 F5 O2	
Formula weight	268.18	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P21/n	
Unit cell dimensions	a = 6.7504(2) Å	α= 90°.
	b = 19.0686(5) Å	β= 110.485(2)°.
	c = 8.9486(2) Å	γ = 90°.
Volume	1079.03(5) $Å^3$	
Z	4	
Density (calculated)	1.651 Mg/m^3	
Absorption coefficient	0.169 mm^{-1}	
F(000)	544	
Crystal size	0.48 x 0.24 x 0.19 mm	
Theta range for data collection	3.29 to 27.49°.	
Index ranges	-8<=h<=8, -22<=k<=24, -11<=l<=11	
Reflections collected	4338	
Independent reflections	2414 [R(int) = 0.0188]	
Completeness to theta = 27.49°	97.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2414 / 0 / 200	
Goodness-of-fit on F^2	0.998	
Final R indices [I>2sigma(I)]	R1 = 0.0392, wR2 = 0.0915	
R indices (all data)	R1 = 0.0585, wR2 = 0.1016	
Extinction coefficient	6(4)x10x10^{-6}	
Largest diff. peak and hole	0.382 and -0.251 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 1. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2305(2)	3607(1)	3784(2)	23(1)
C(2)	4730(2)	3586(1)	4102(2)	26(1)
C(3)	4345(3)	3470(1)	2335(2)	28(1)
C(4)	4800(3)	4081(1)	1362(2)	32(1)
C(5)	3661(3)	3859(1)	-365(2)	38(1)
C(6)	1613(3)	3916(1)	-667(2)	38(1)
C(7)	1299(3)	4179(1)	847(2)	30(1)
C(8)	1891(3)	3531(1)	1956(2)	26(1)
C(9)	3263(3)	4642(1)	1510(2)	33(1)
C(10)	1379(2)	4246(1)	4334(2)	24(1)
C(11)	1523(3)	2961(1)	4411(2)	31(1)
O(12)	-191(2)	4541(1)	3460(1)	33(1)
O(13)	2395(2)	4413(1)	5826(1)	32(1)
F(14)	-595(2)	2961(1)	3925(1)	42(1)
F(15)	2237(2)	2922(1)	6001(1)	44(1)
F(16)	2084(2)	2362(1)	3878(1)	41(1)
F(17)	5809(1)	4172(1)	4781(1)	33(1)
F(18)	5764(2)	3050(1)	5069(1)	37(1)

Table 3. Bond lengths [Å] and angles [°] for 1.

C(1)-C(11)	1.523(2)	C(6)-C(7)	1.527(3)
C(1)-C(10)	1.526(2)	C(6)-H(6)	1.01(2)
C(1)-C(2)	1.561(2)	C(7)-C(9)	1.529(3)
C(1)-C(8)	1.567(2)	C(7)-C(8)	1.548(2)
C(2)-F(17)	1.3563(19)	C(7)-H(7)	0.95(2)
C(2)-F(18)	1.3632(18)	C(8)-H(8)	0.981(19)
C(2)-C(3)	1.526(2)	C(9)-H(9A)	0.98(2)
C(3)-C(4)	1.549(2)	C(9)-H(9B)	1.00(2)
C(3)-C(8)	1.574(2)	C(10)-O(12)	1.2125(19)
C(3)-H(3)	0.96(2)	C(10)-O(13)	1.3094(19)
C(4)-C(5)	1.525(3)	C(11)-F(15)	1.335(2)
C(4)-C(9)	1.529(3)	C(11)-F(14)	1.341(2)
C(4)-H(4)	0.988(19)	C(11)-F(16)	1.341(2)
C(5)-C(6)	1.317(3)	O(13)-H(13)	0.85(3)
C(5)-H(5)	1.03(2)		

C(11)-C(1)-C(10)	107.03(13)	C(5)-C(4)-C(9)	99.51(14)
C(11)-C(1)-C(2)	112.84(13)	C(5)-C(4)-C(3)	103.46(15)
C(10)-C(1)-C(2)	118.94(13)	C(9)-C(4)-C(3)	101.77(13)
C(11)-C(1)-C(8)	110.56(13)	C(5)-C(4)-H(4)	118.8(11)
C(10)-C(1)-C(8)	117.86(13)	C(9)-C(4)-H(4)	118.6(12)
C(2)-C(1)-C(8)	88.86(11)	C(3)-C(4)-H(4)	112.2(11)
F(17)-C(2)-F(18)	104.97(12)	C(6)-C(5)-C(4)	107.82(16)
F(17)-C(2)-C(3)	117.02(13)	C(6)-C(5)-H(5)	127.3(13)
F(18)-C(2)-C(3)	114.35(14)	C(4)-C(5)-H(5)	124.3(13)
F(17)-C(2)-C(1)	115.12(13)	C(5)-C(6)-C(7)	107.84(16)
F(18)-C(2)-C(1)	113.99(13)	C(5)-C(6)-H(6)	127.9(12)
C(3)-C(2)-C(1)	91.57(11)	C(7)-C(6)-H(6)	124.0(12)
C(2)-C(3)-C(4)	118.56(15)	C(6)-C(7)-C(9)	99.42(15)
C(2)-C(3)-C(8)	89.86(12)	C(6)-C(7)-C(8)	102.78(14)
C(4)-C(3)-C(8)	102.91(13)	C(9)-C(7)-C(8)	102.60(13)
C(2)-C(3)-H(3)	112.1(12)	C(6)-C(7)-H(7)	118.1(11)
C(4)-C(3)-H(3)	113.6(12)	C(9)-C(7)-H(7)	116.6(12)
C(8)-C(3)-H(3)	117.6(11)	C(8)-C(7)-H(7)	114.9(11)

C(7)-C(8)-C(1)	120.45(14)	H(9A)-C(9)-H(9B)	108.6(16)
C(7)-C(8)-C(3)	102.34(13)	O(12)-C(10)-O(13)	124.93(15)
C(1)-C(8)-C(3)	89.55(11)	O(12)-C(10)-C(1)	121.73(14)
C(7)-C(8)-H(8)	113.0(11)	O(13)-C(10)-C(1)	113.28(14)
C(1)-C(8)-H(8)	111.1(11)	F(15)-C(11)-F(14)	106.96(13)
C(3)-C(8)-H(8)	118.4(11)	F(15)-C(11)-F(16)	106.85(14)
C(4)-C(9)-C(7)	94.83(14)	F(14)-C(11)-F(16)	106.65(14)
C(4)-C(9)-H(9A)	114.3(11)	F(15)-C(11)-C(1)	113.28(14)
C(7)-C(9)-H(9A)	115.1(11)	F(14)-C(11)-C(1)	110.38(14)
C(4)-C(9)-H(9B)	112.7(11)	F(16)-C(11)-C(1)	112.35(13)
C(7)-C(9)-H(9B)	110.8(11)	C(10)-O(13)-H(13)	106.8(16)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 1. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	25(1)	23(1)	23(1)	-1(1)	10(1)	0(1)
C(2)	25(1)	28(1)	26(1)	3(1)	9(1)	2(1)
C(3)	30(1)	30(1)	28(1)	2(1)	13(1)	7(1)
C(4)	28(1)	41(1)	30(1)	5(1)	13(1)	2(1)
C(5)	45(1)	46(1)	28(1)	4(1)	18(1)	7(1)
C(6)	41(1)	48(1)	24(1)	2(1)	11(1)	3(1)
C(7)	28(1)	35(1)	26(1)	5(1)	10(1)	8(1)
C(8)	28(1)	26(1)	24(1)	-4(1)	11(1)	-3(1)
C(9)	40(1)	30(1)	29(1)	5(1)	13(1)	-1(1)
C(10)	24(1)	26(1)	24(1)	-2(1)	11(1)	-2(1)
C(11)	34(1)	29(1)	32(1)	0(1)	16(1)	-2(1)
O(12)	28(1)	39(1)	28(1)	-8(1)	6(1)	9(1)
O(13)	34(1)	36(1)	23(1)	-5(1)	7(1)	8(1)
F(14)	35(1)	43(1)	54(1)	-1(1)	23(1)	-10(1)
F(15)	60(1)	41(1)	33(1)	9(1)	21(1)	-4(1)
F(16)	54(1)	23(1)	53(1)	-1(1)	27(1)	-2(1)
F(17)	27(1)	39(1)	30(1)	-2(1)	7(1)	-7(1)
F(18)	33(1)	42(1)	36(1)	13(1)	12(1)	13(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 1.

	x	y	z	U(eq)
H(3)	4880(30)	3027(11)	2140(20)	35(5)
H(4)	6320(30)	4195(10)	1710(20)	38(5)
H(5)	4390(40)	3634(12)	-1080(30)	55(6)
H(6)	420(30)	3772(11)	-1660(30)	43(6)
H(7)	-20(30)	4390(10)	750(20)	34(5)
H(8)	1000(30)	3121(10)	1510(20)	30(5)
H(9A)	3560(30)	4814(10)	2600(20)	37(5)
H(9B)	3170(30)	5056(11)	800(20)	40(5)
H(13)	1750(40)	4764(14)	6040(30)	63(7)