Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Synthetic Transformation of 1,3-Diarylisobenzofuran-DMAD Adducts: A Facile Preparation of Tri-substituted α-Naphthols

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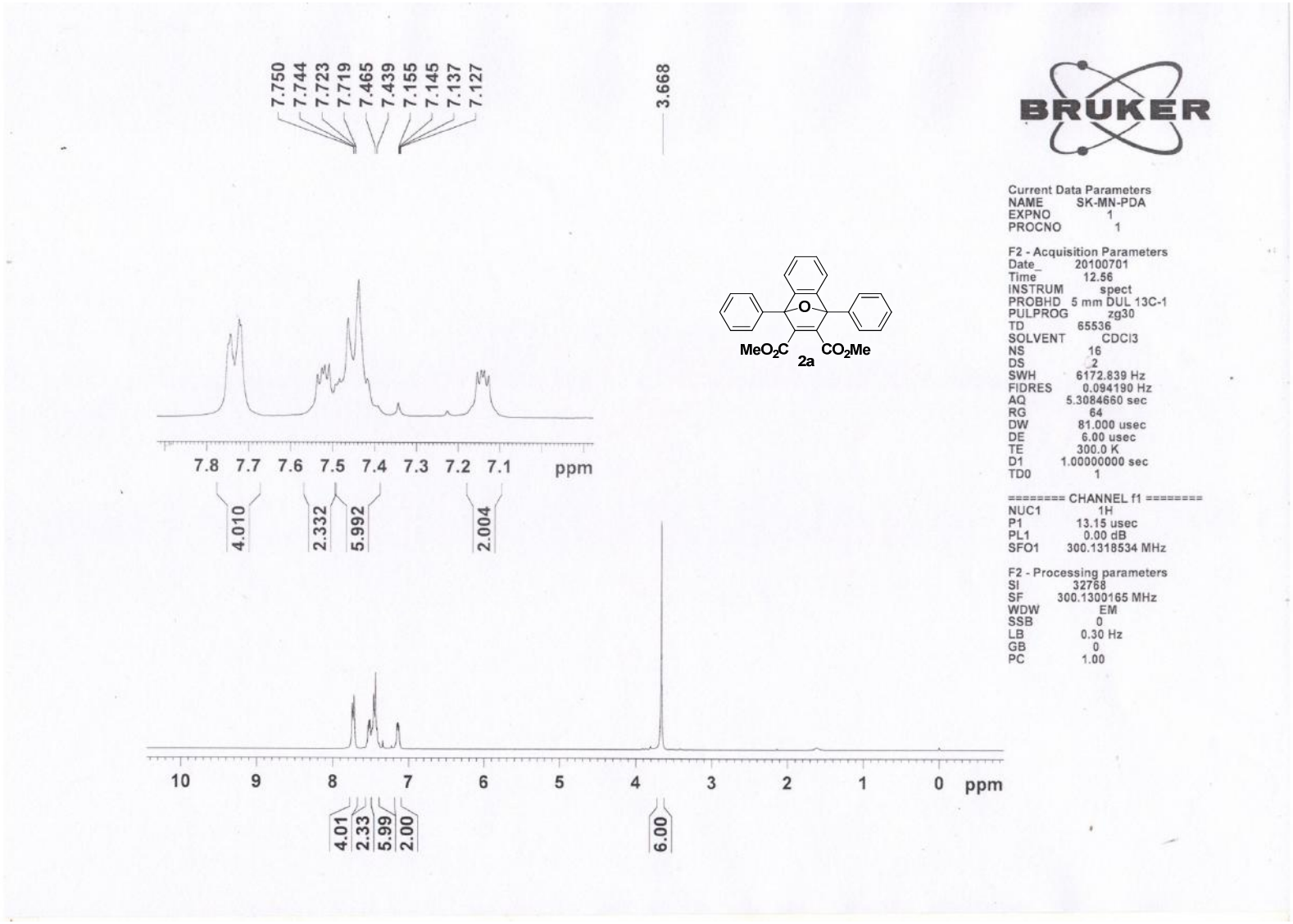
Supporting Information

Contents:



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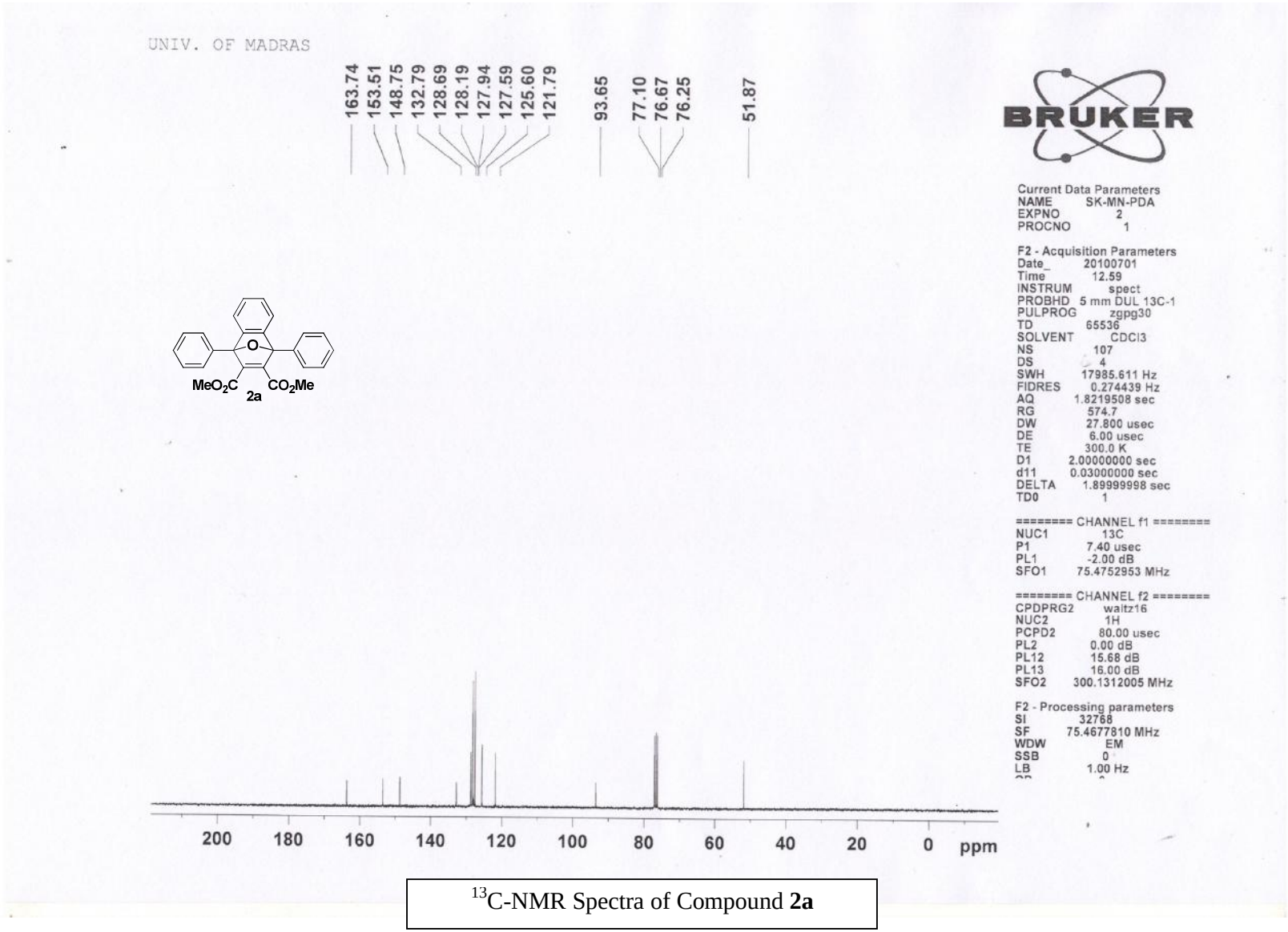

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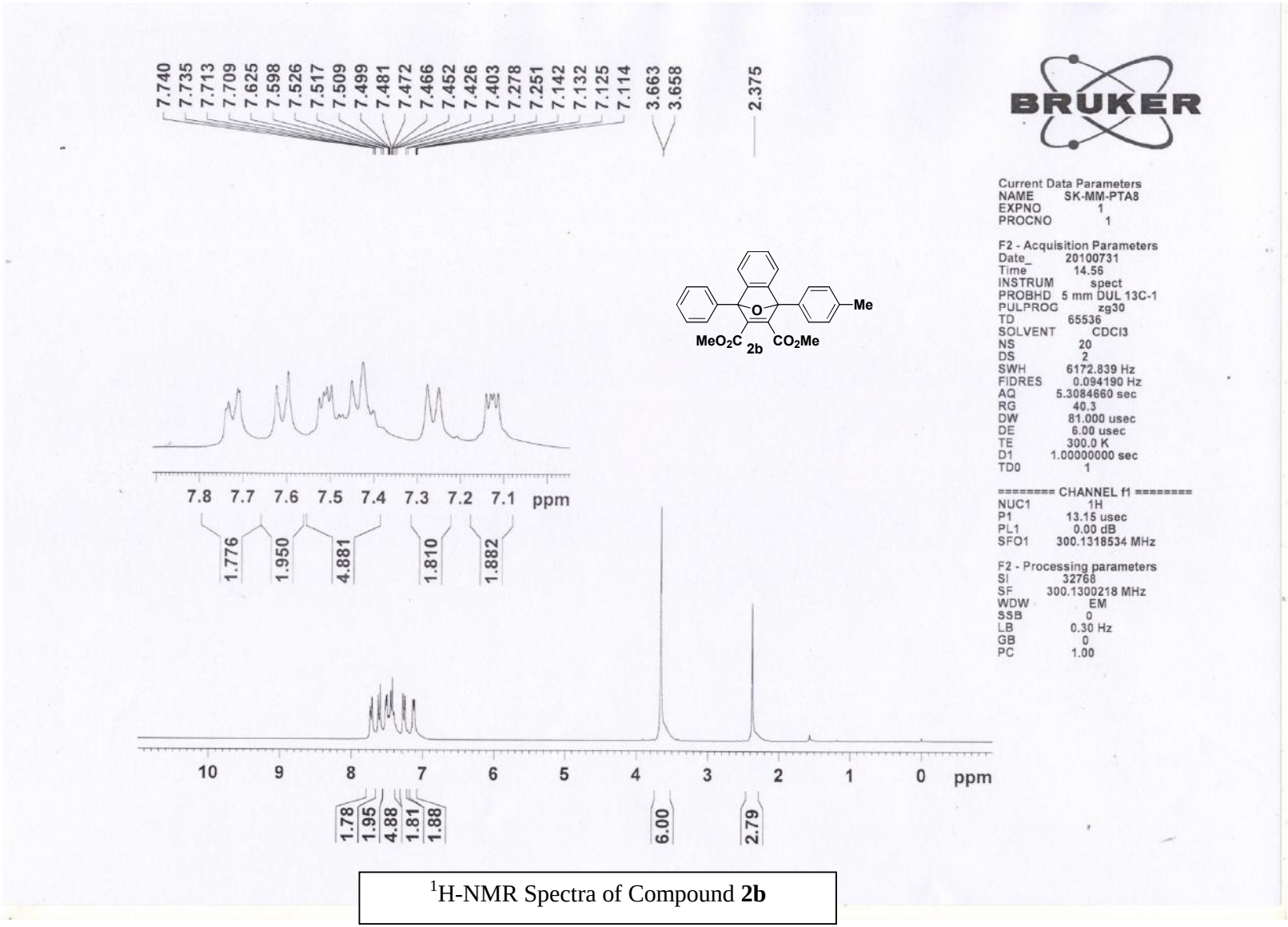

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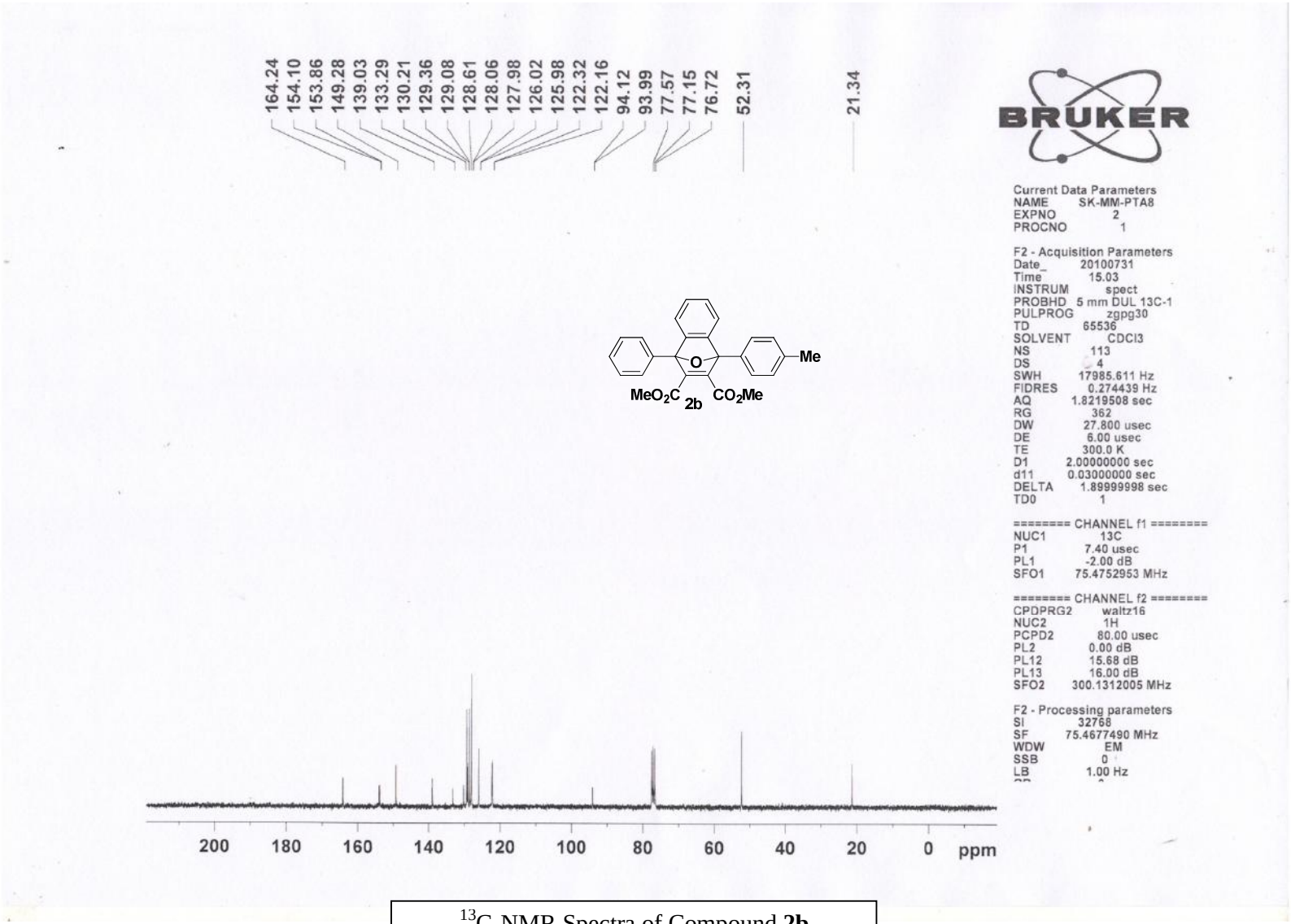

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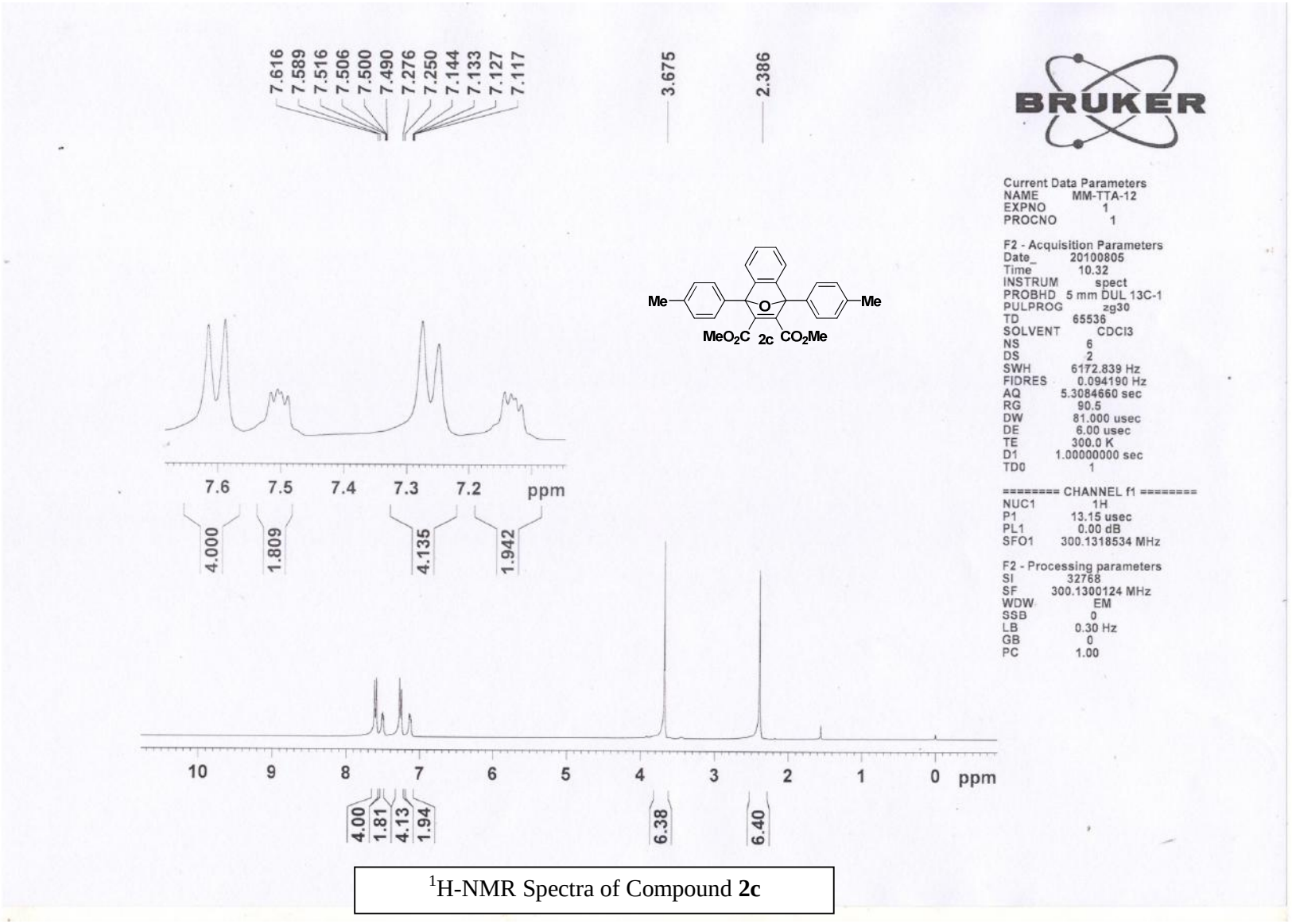

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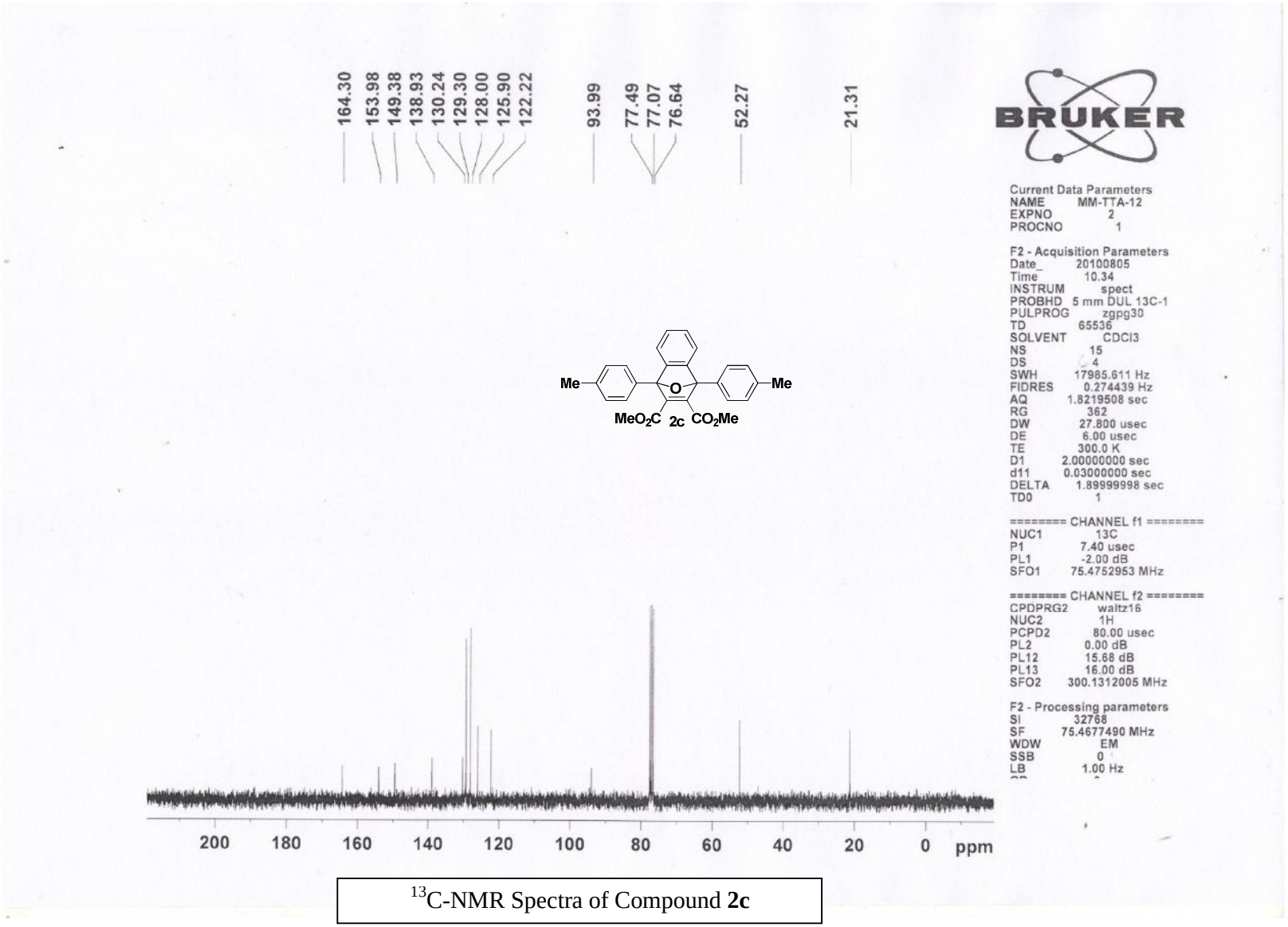

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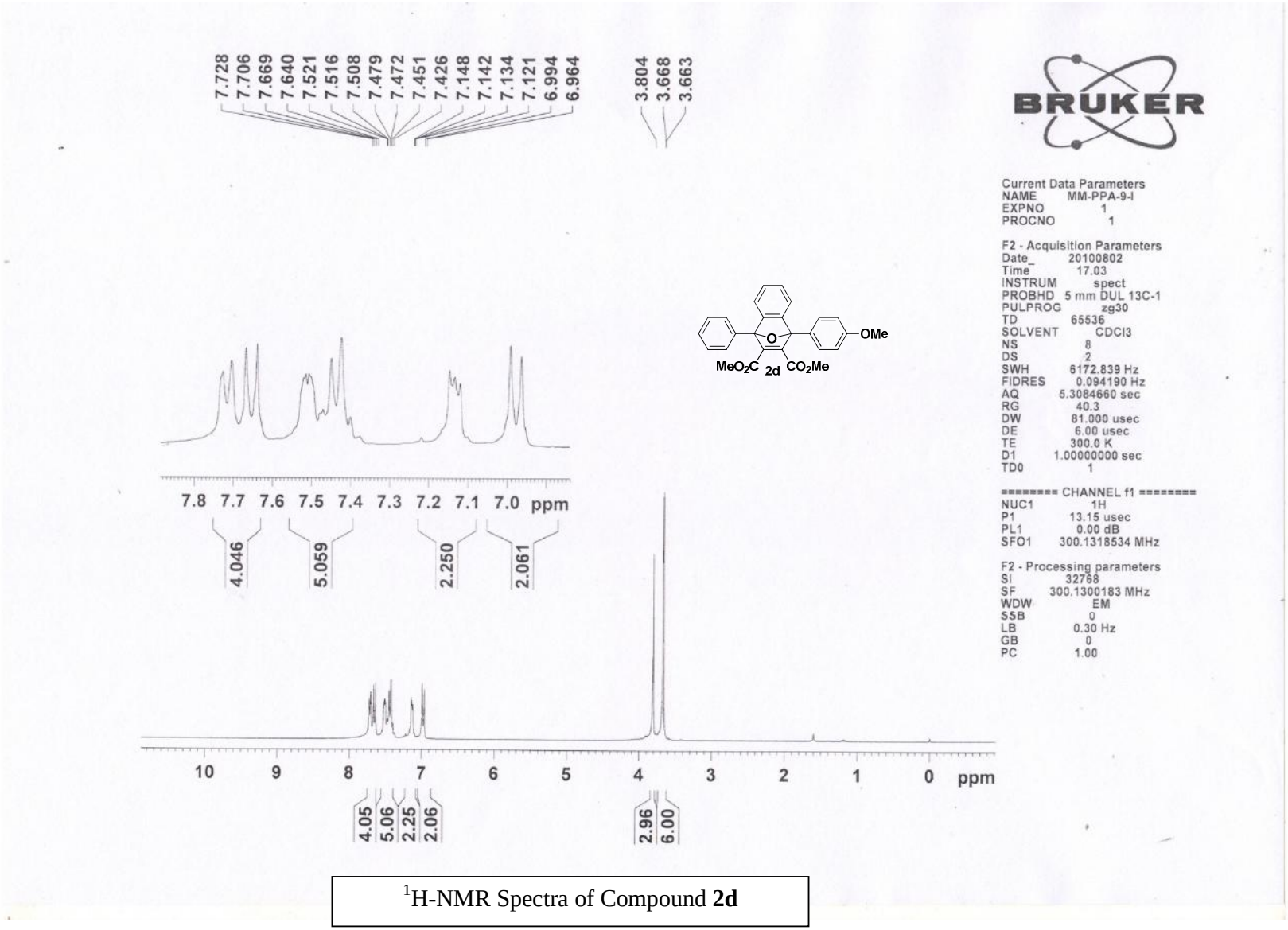

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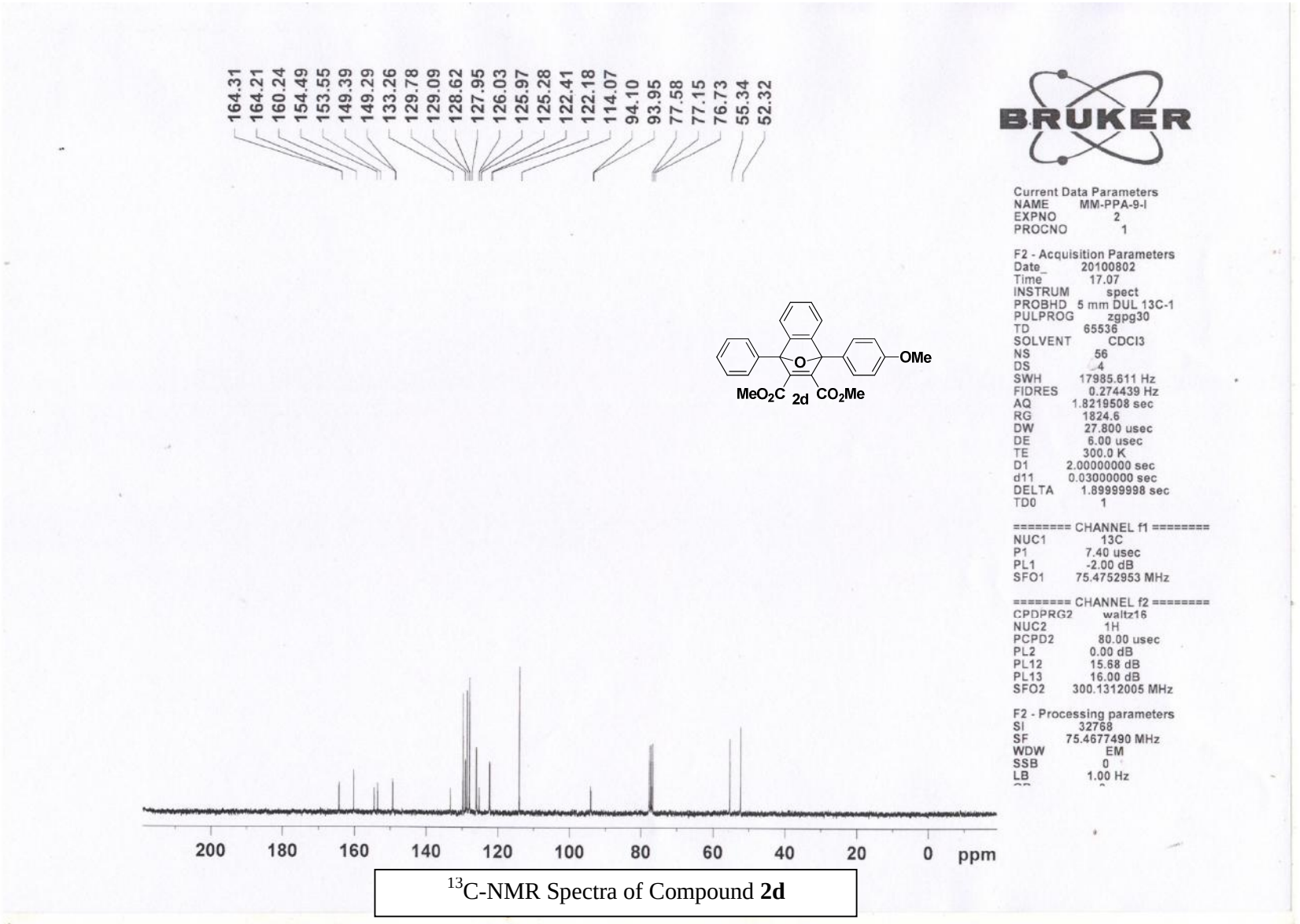

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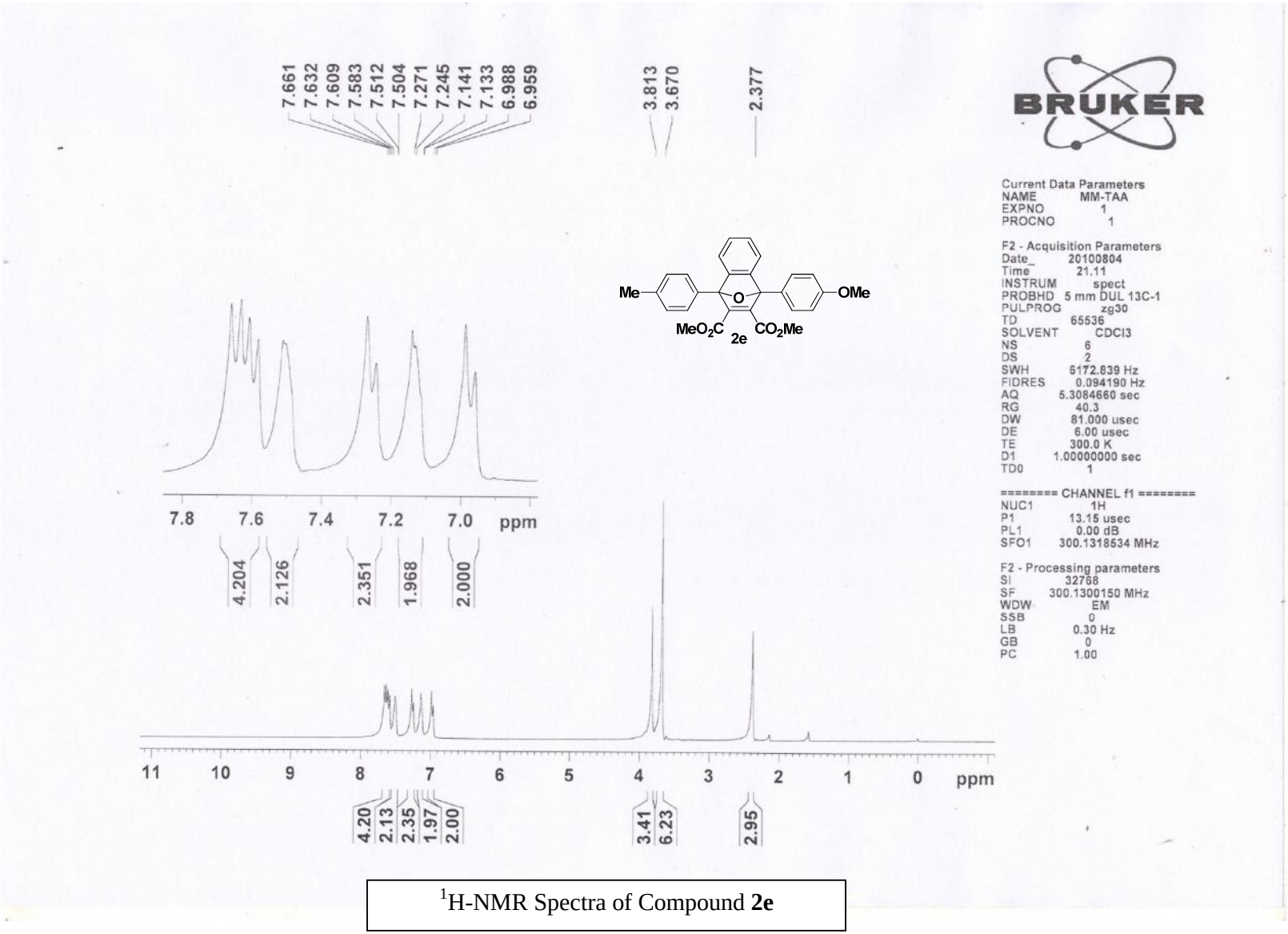

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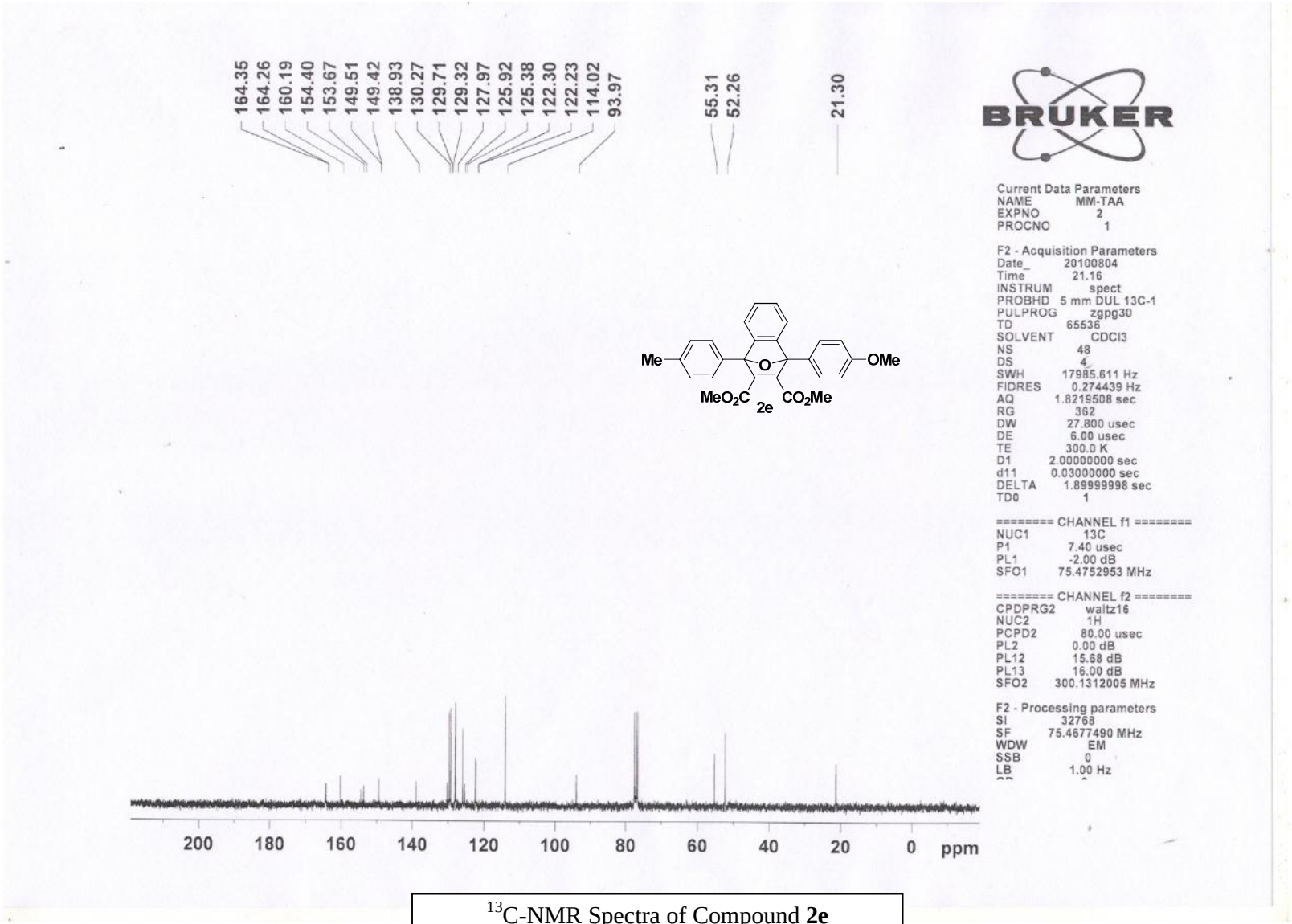

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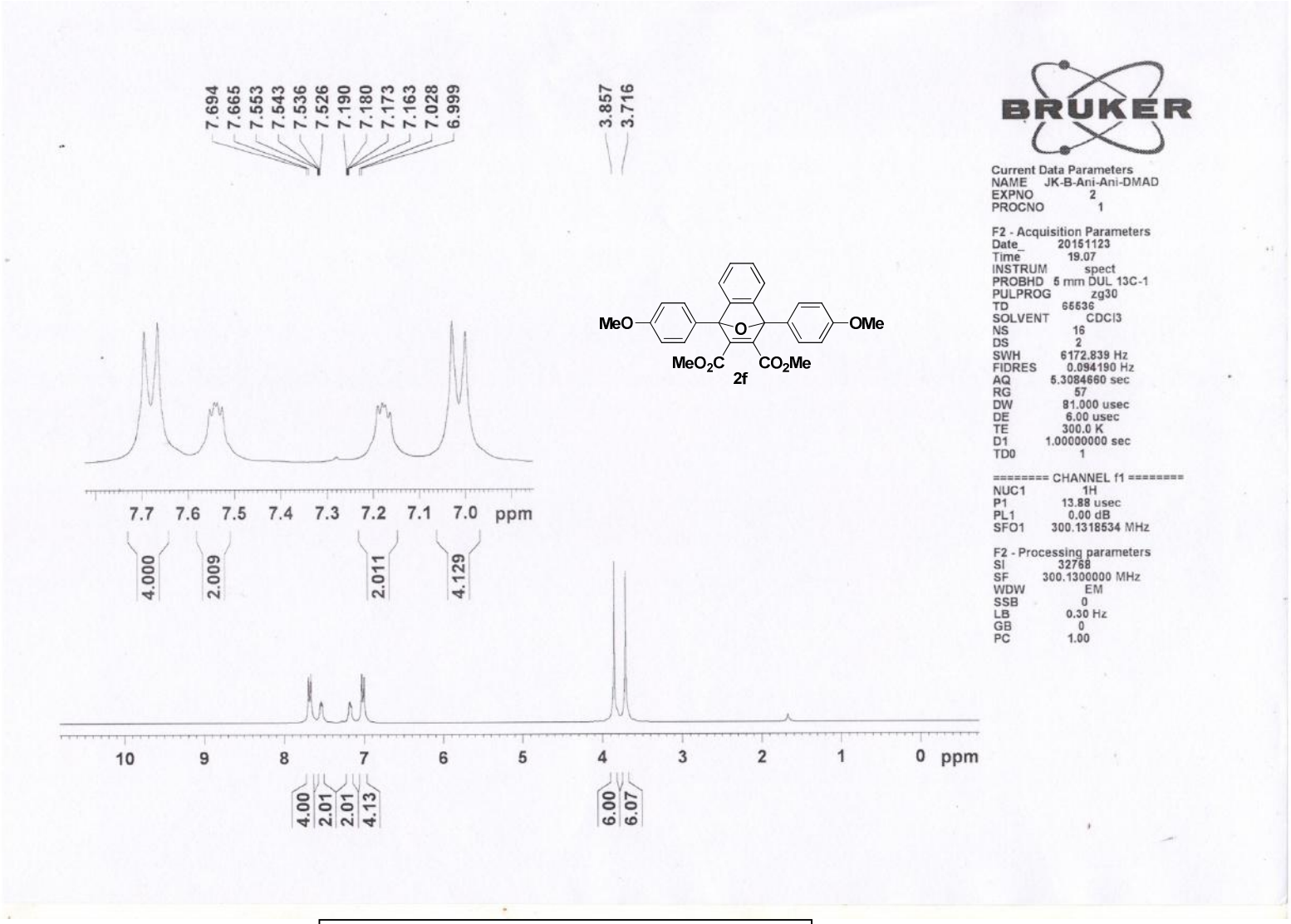

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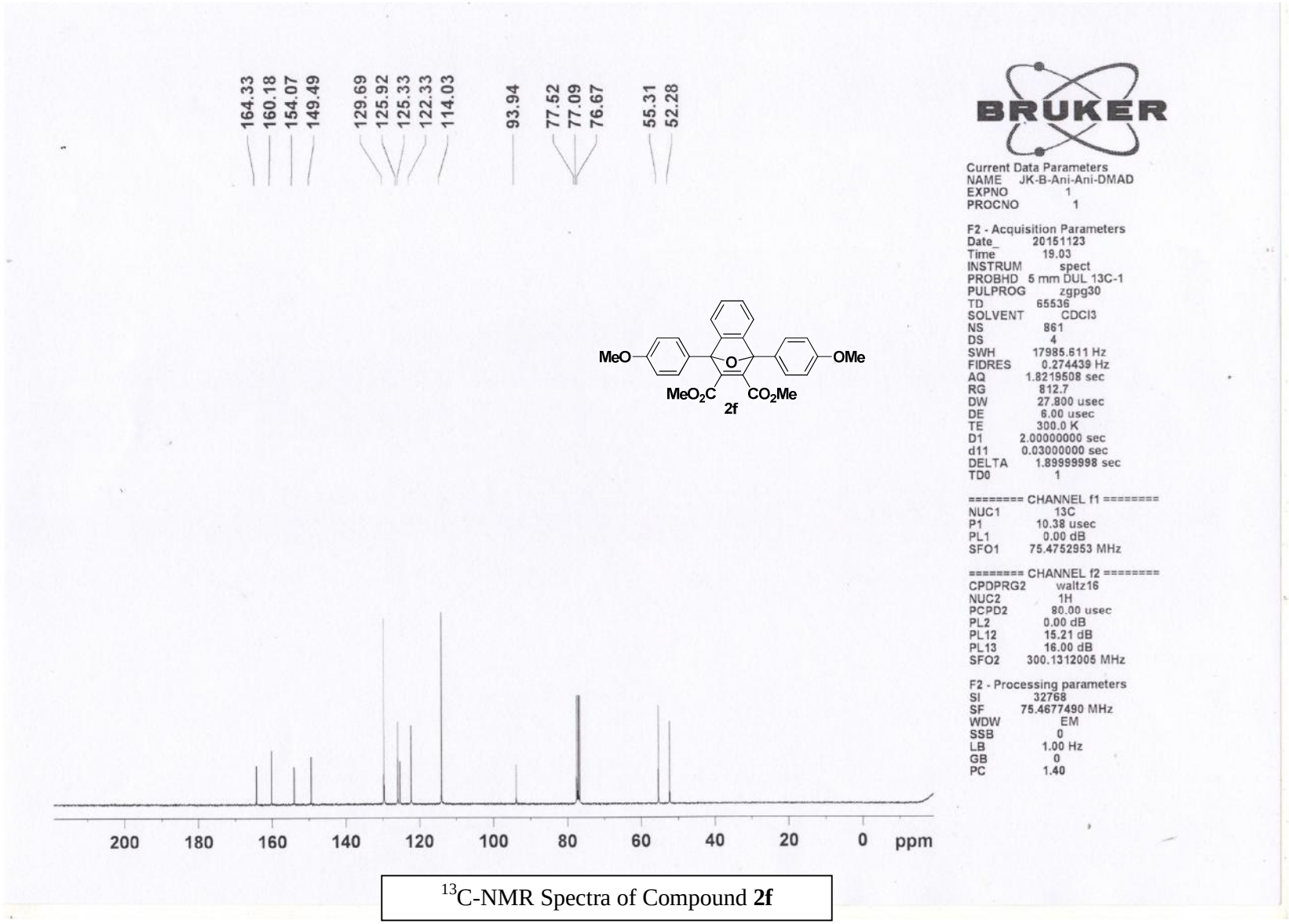

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PROCNO 1

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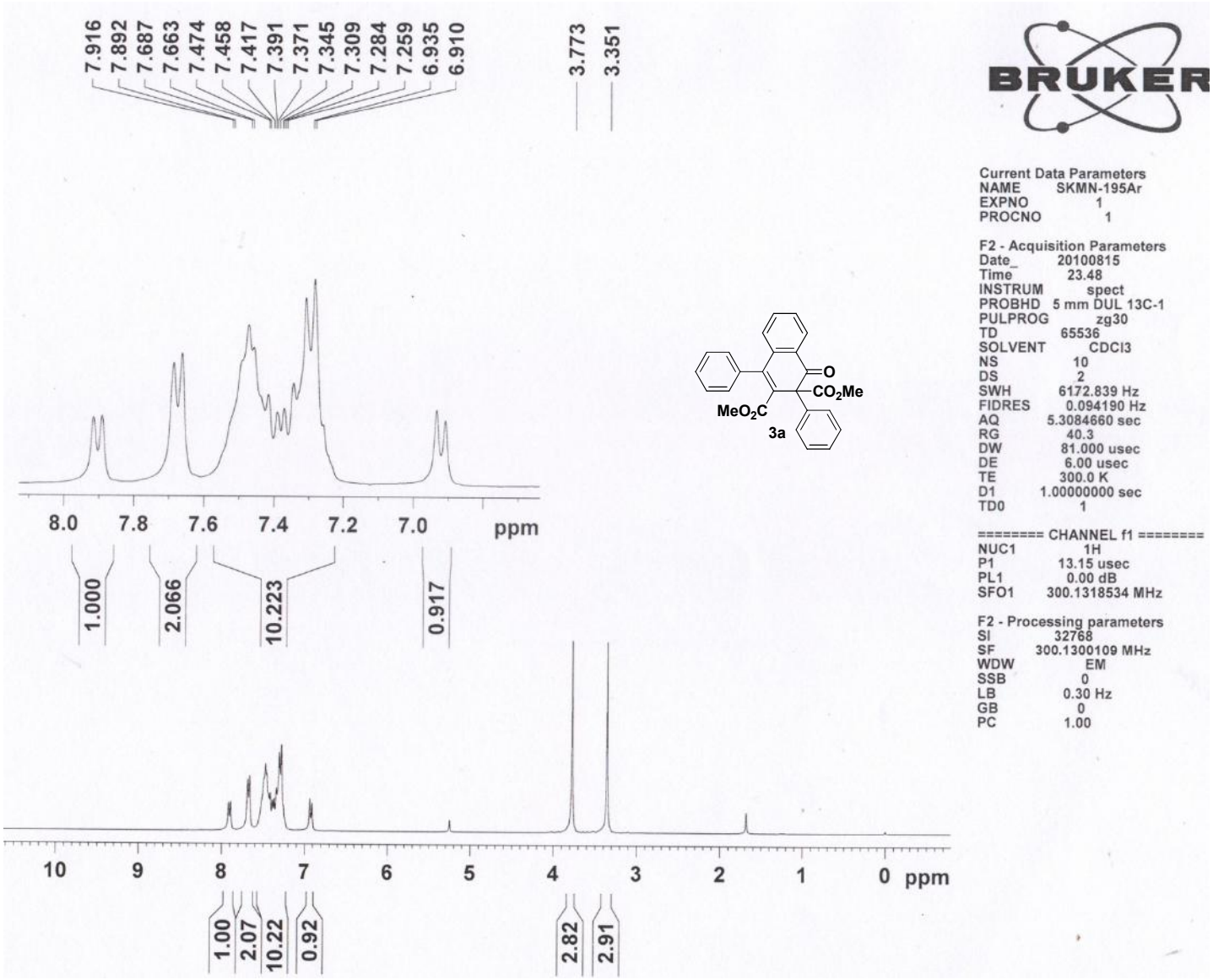

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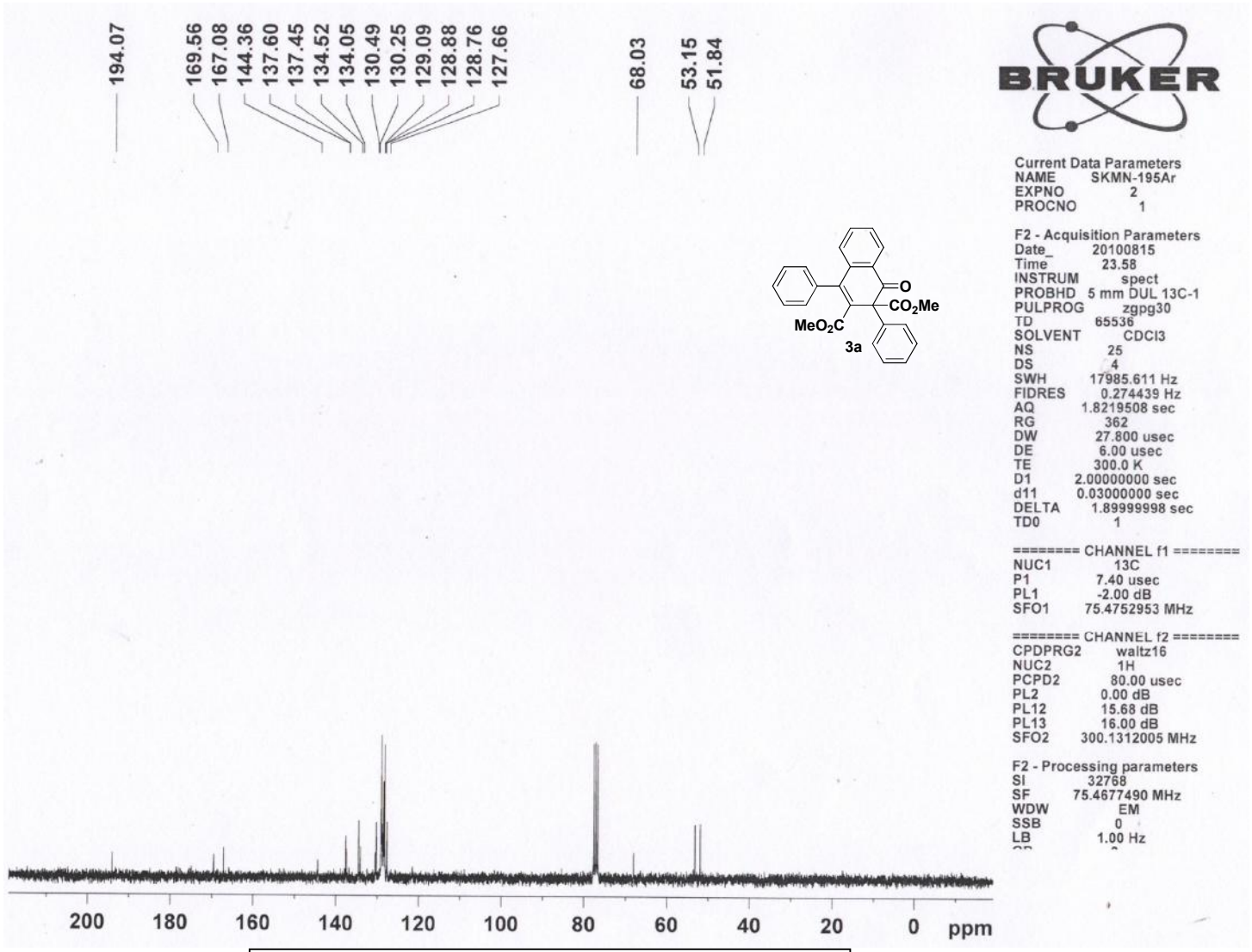

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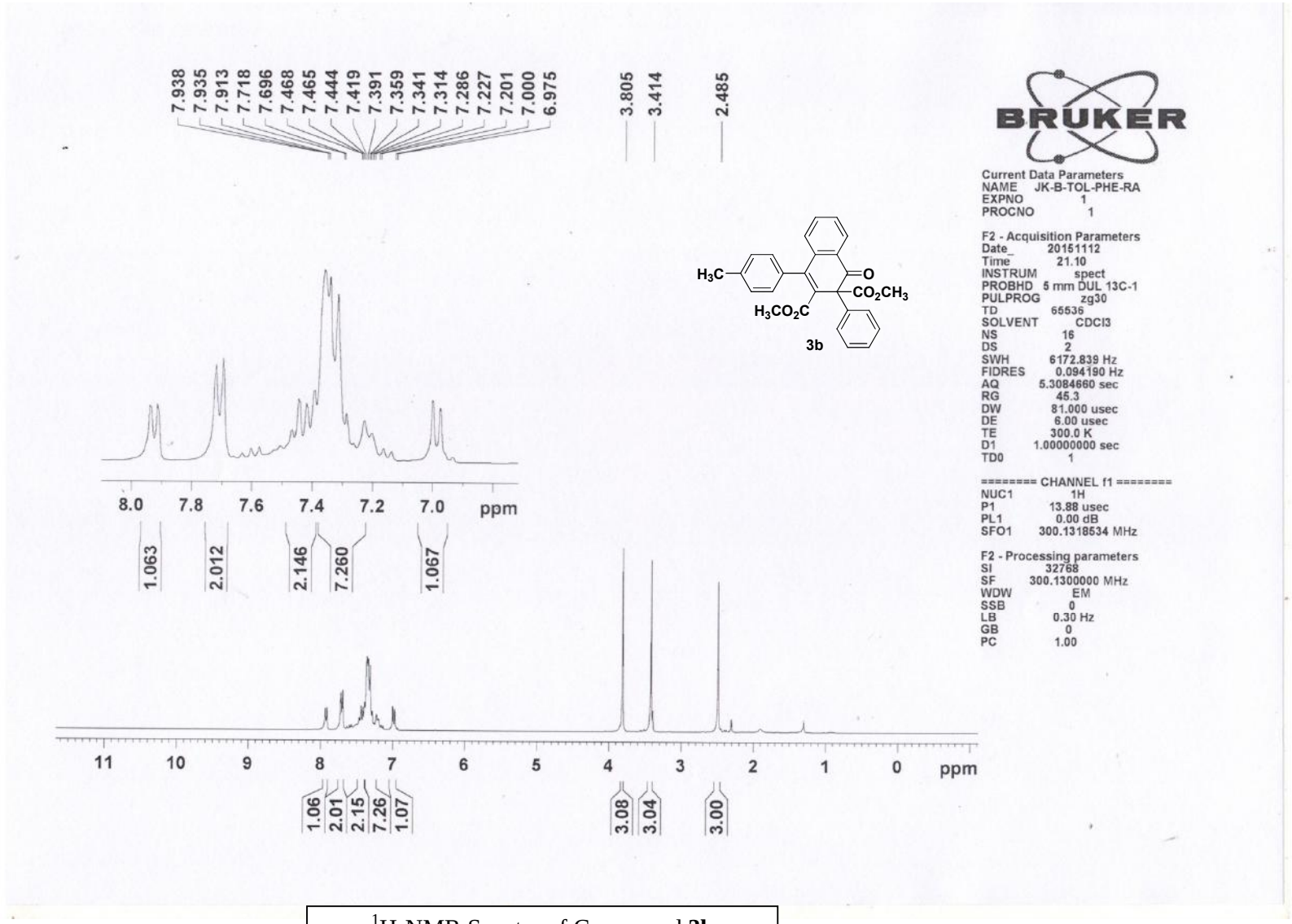

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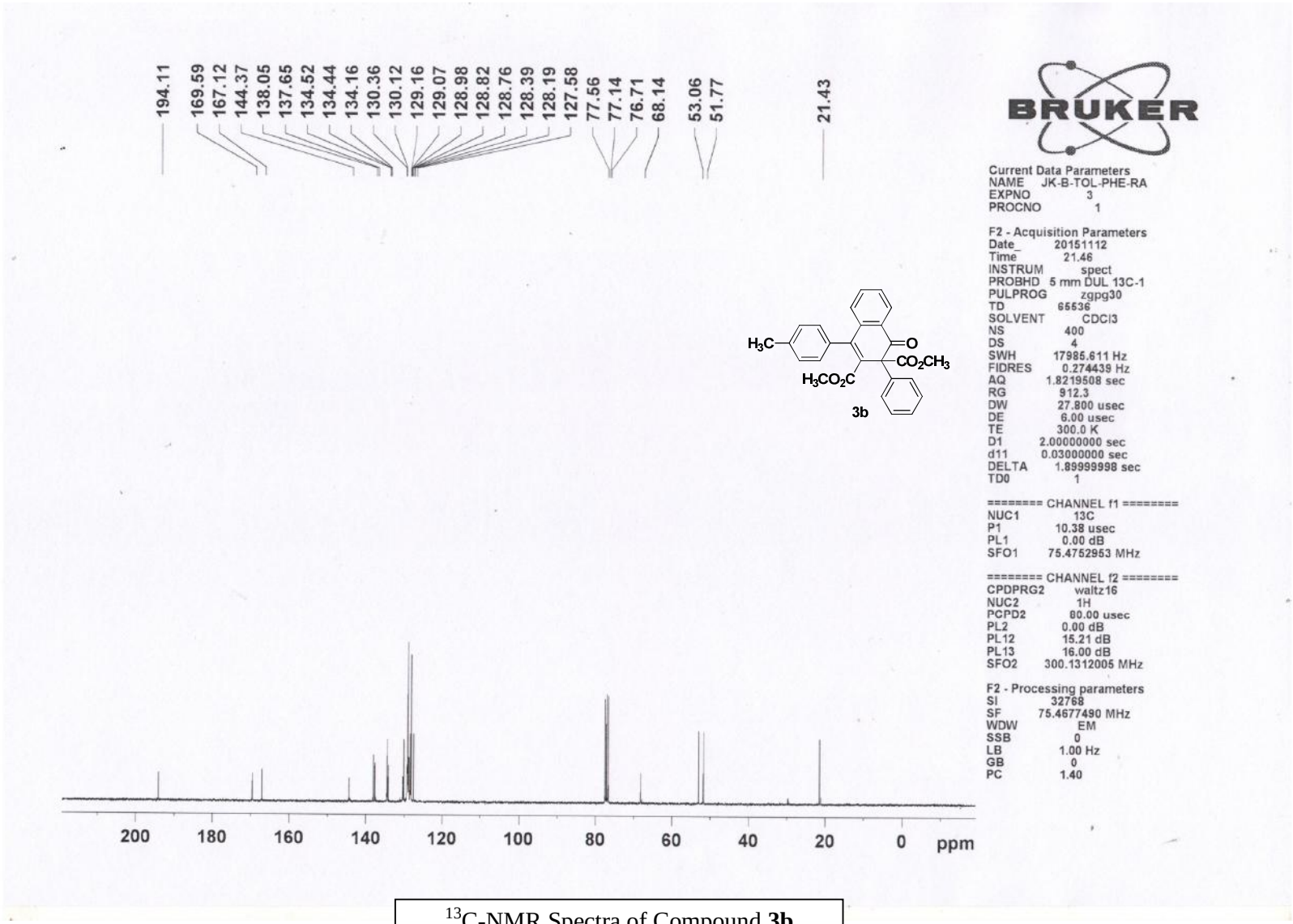

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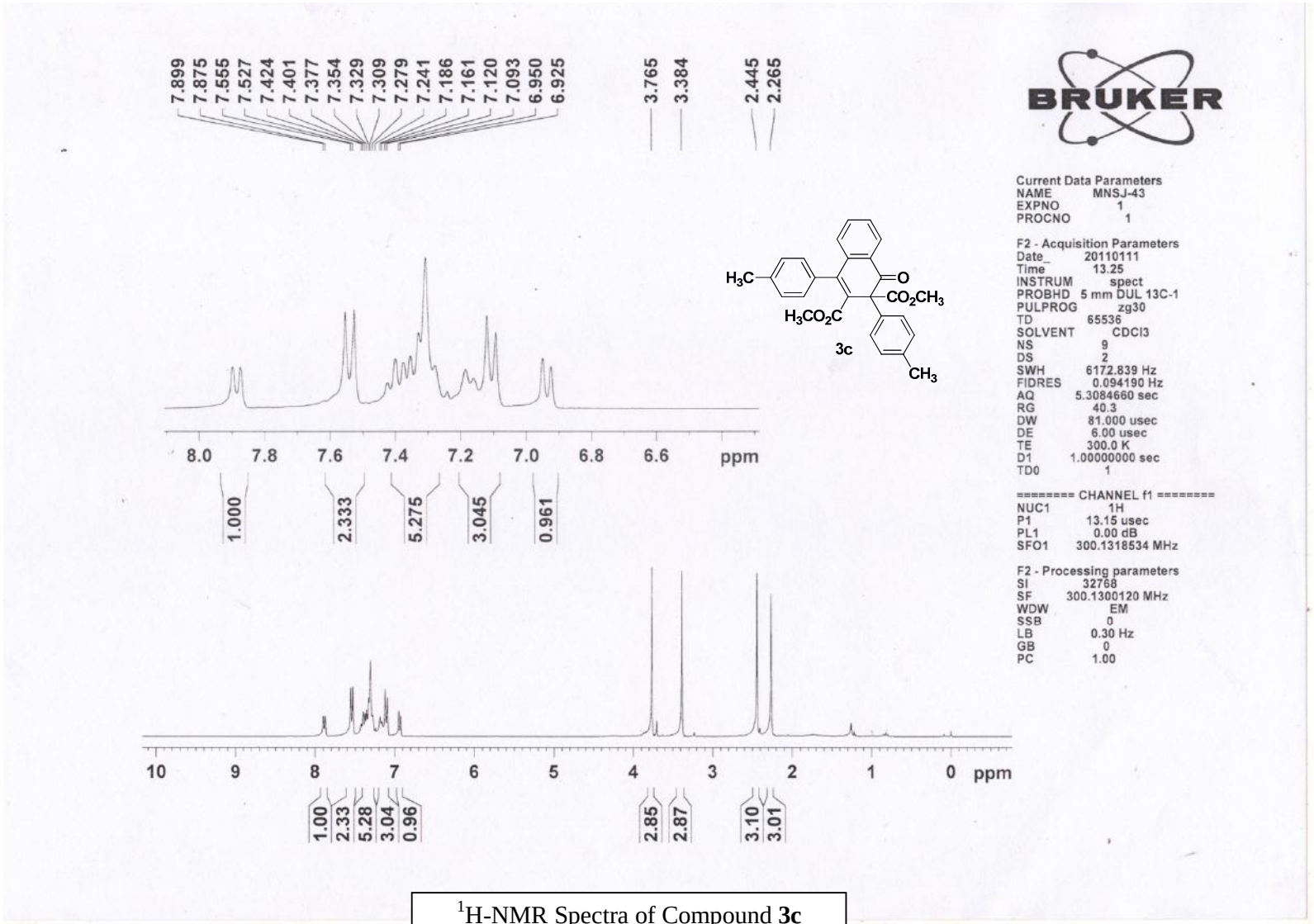

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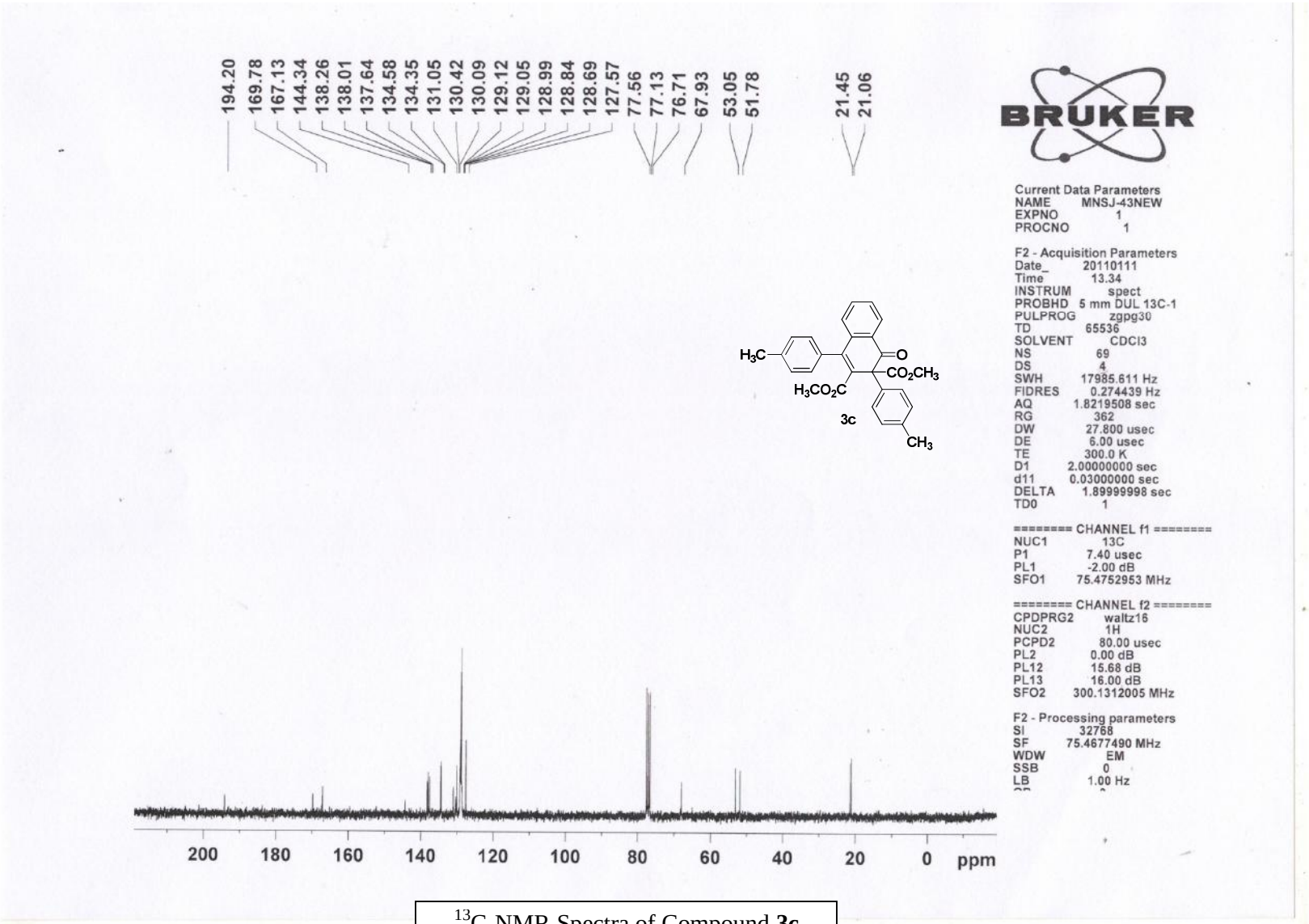

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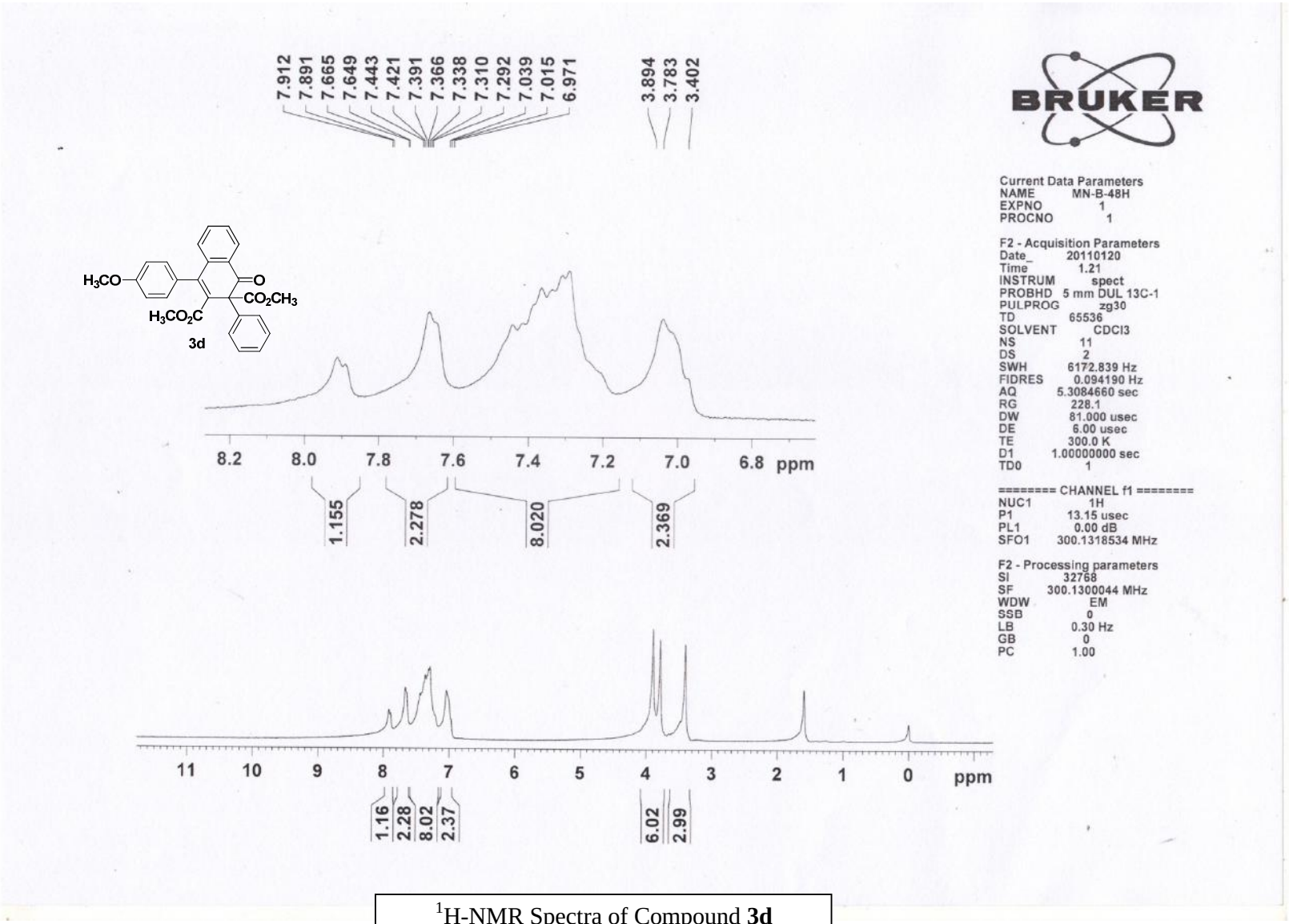

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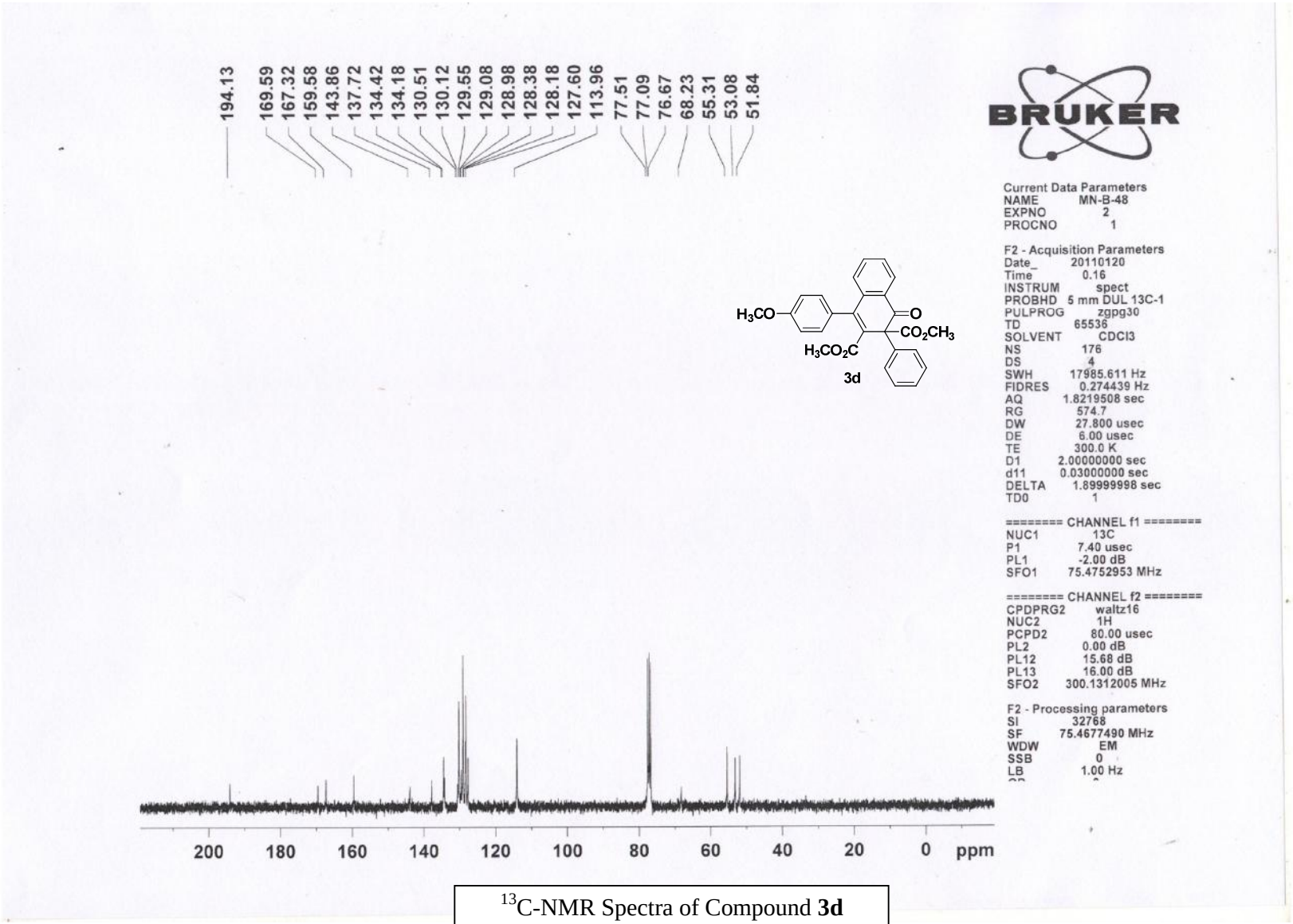

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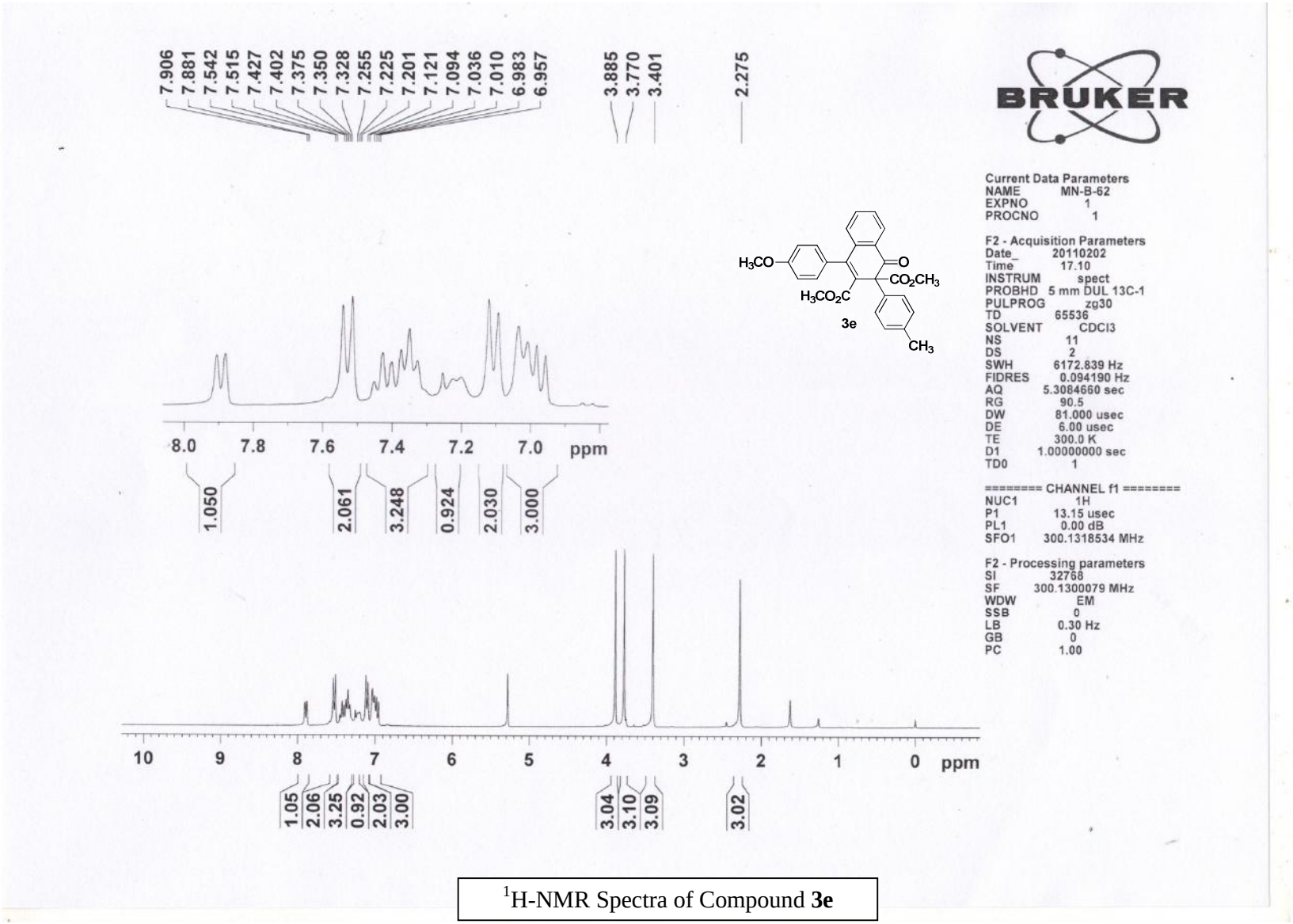

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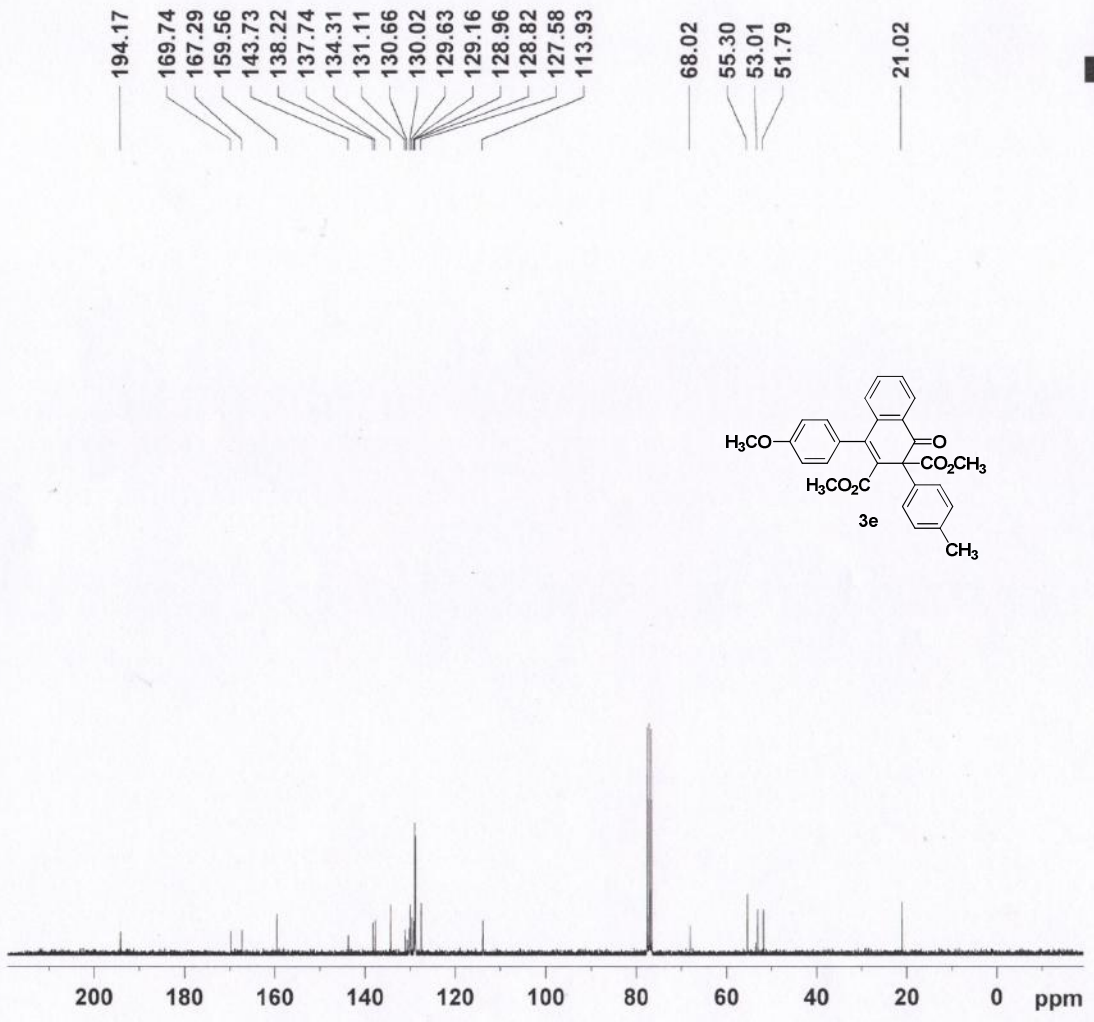

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RG 362
DW 27.800 usec
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TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
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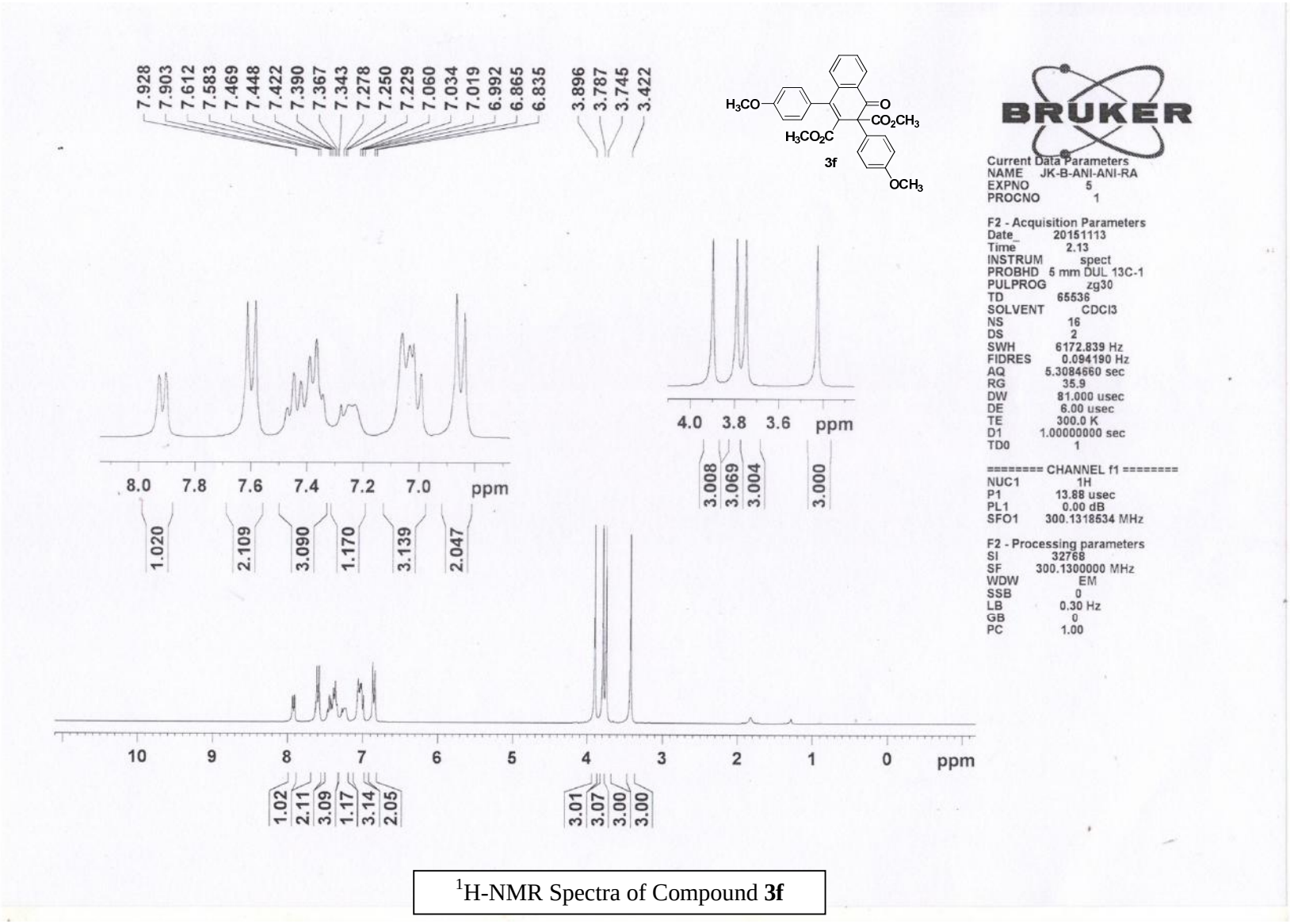

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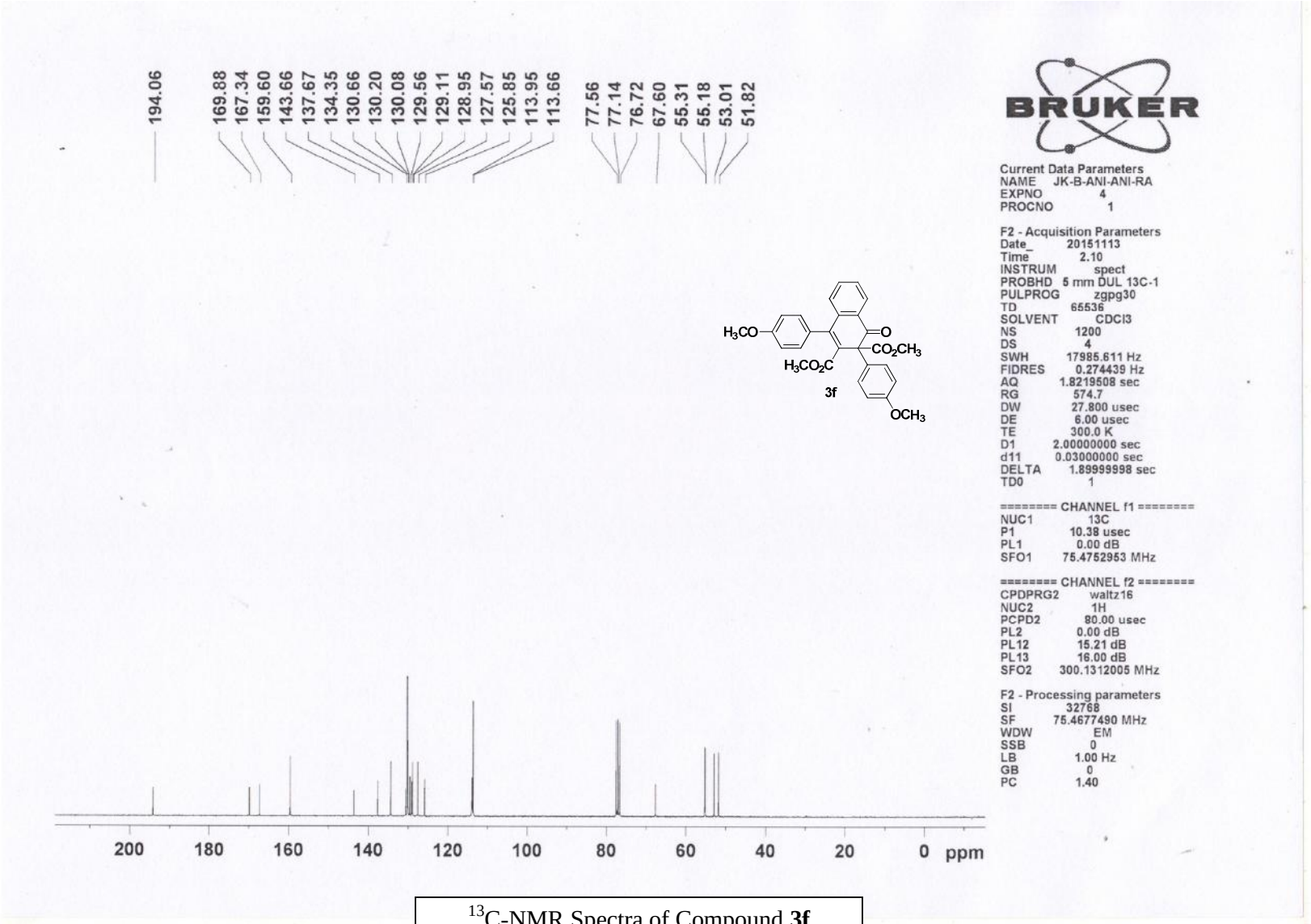

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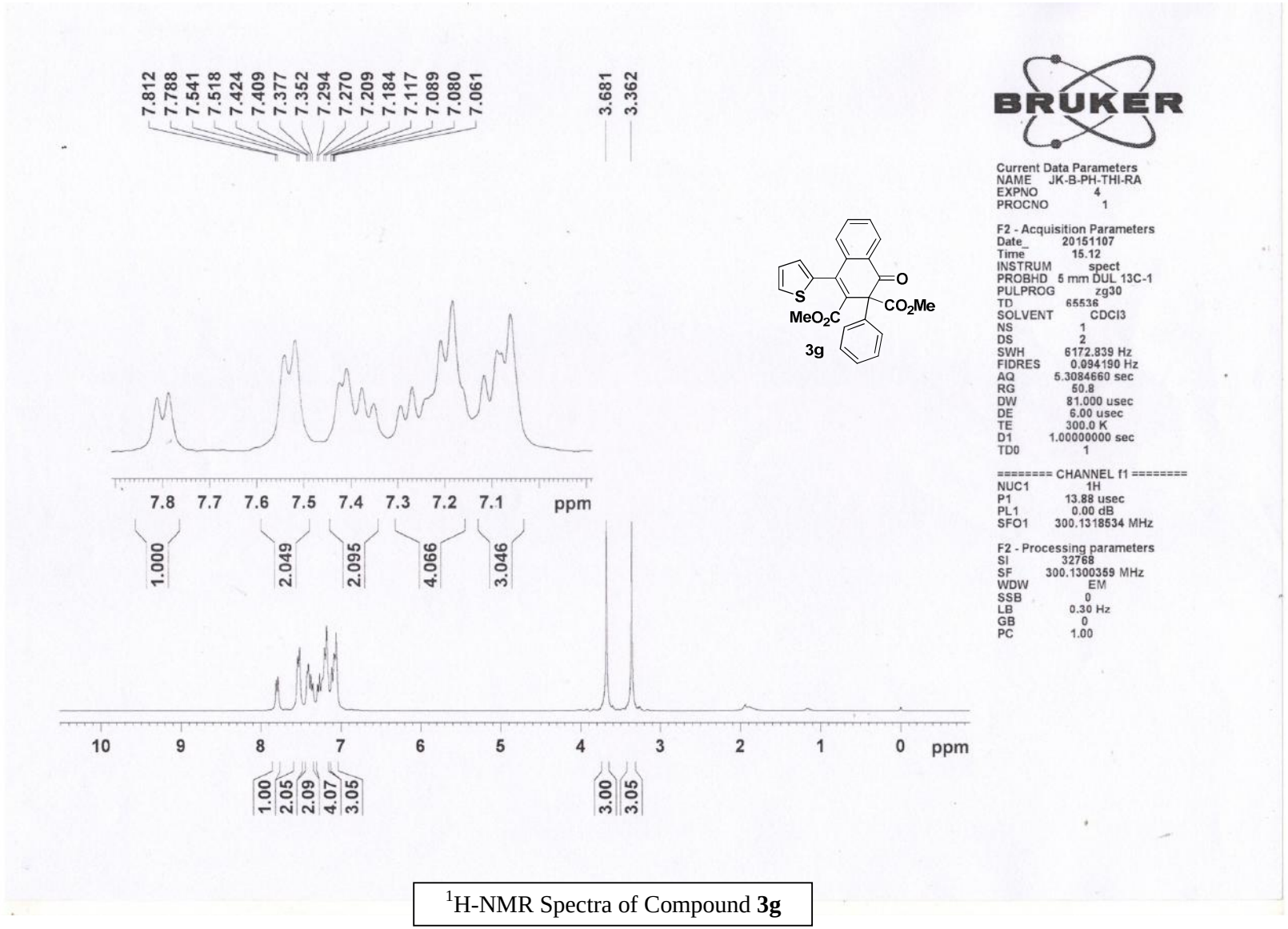

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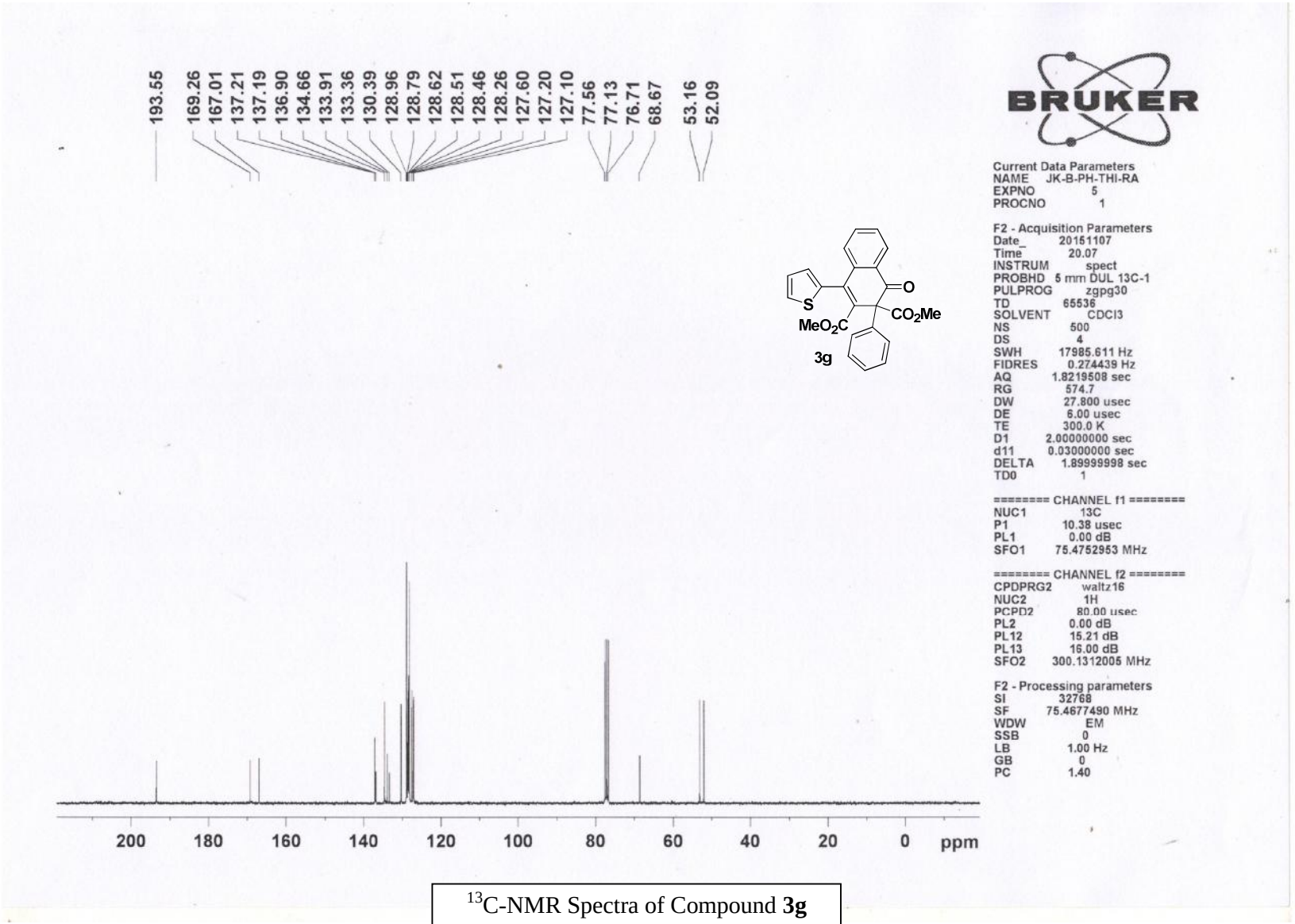

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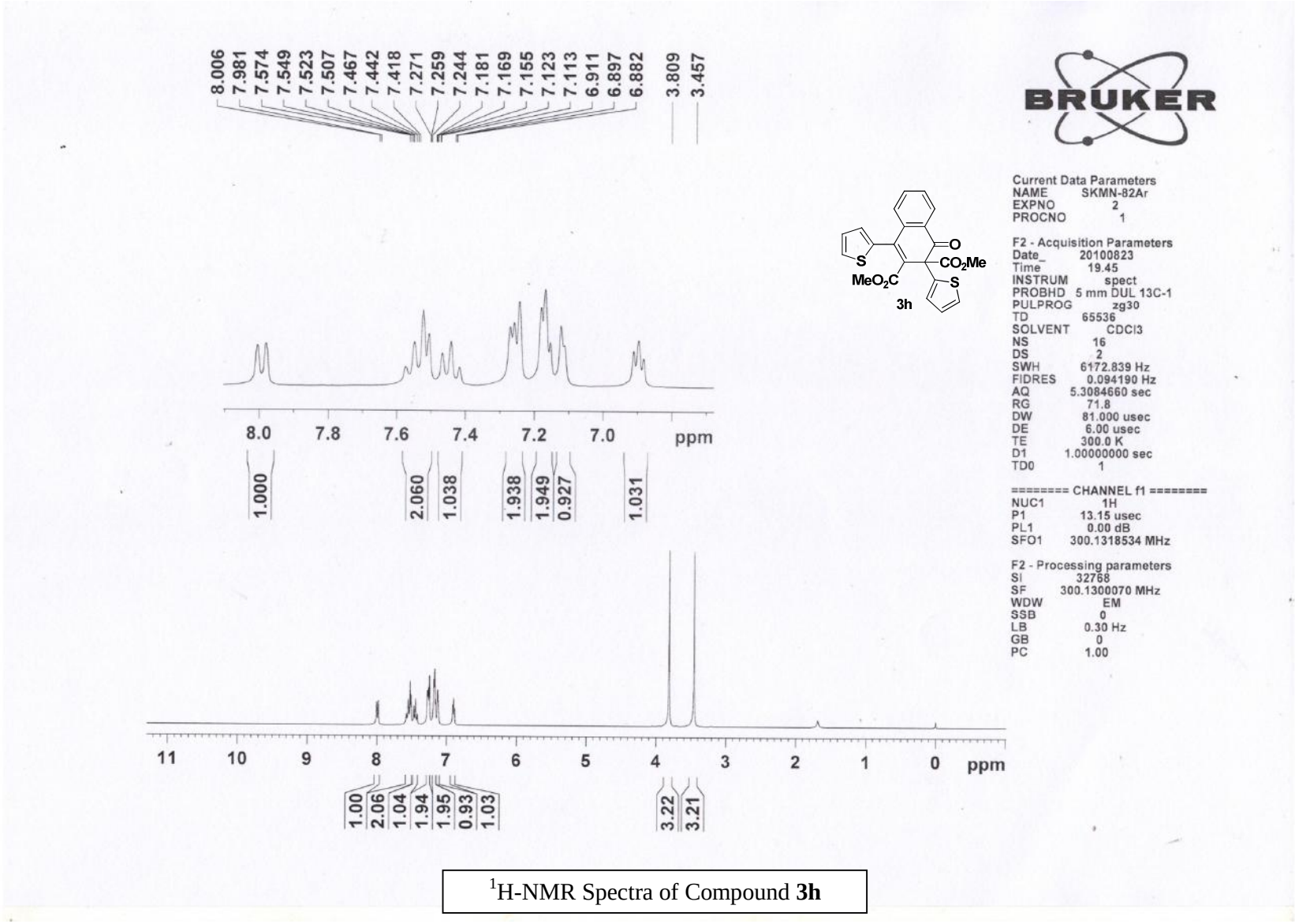

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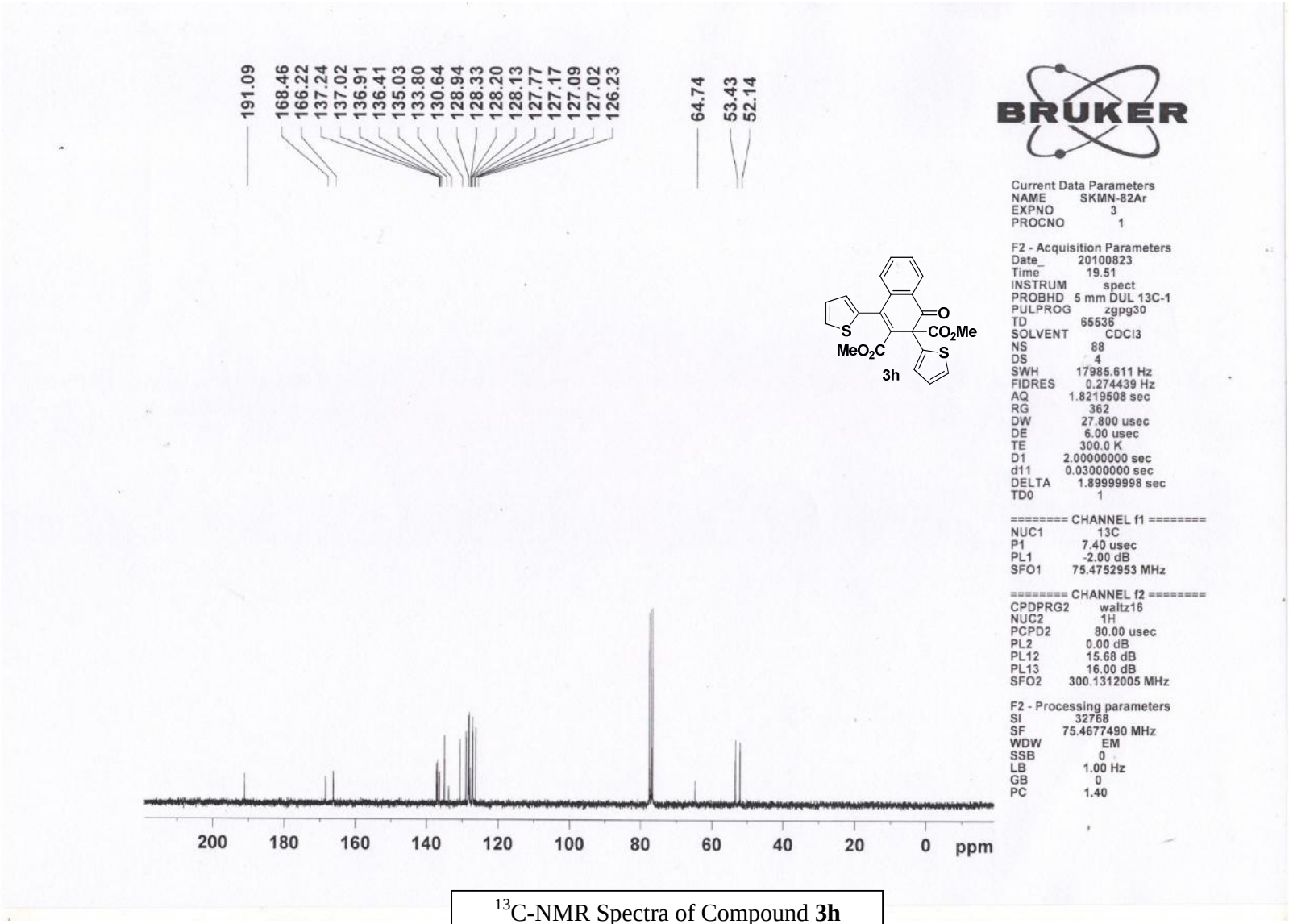

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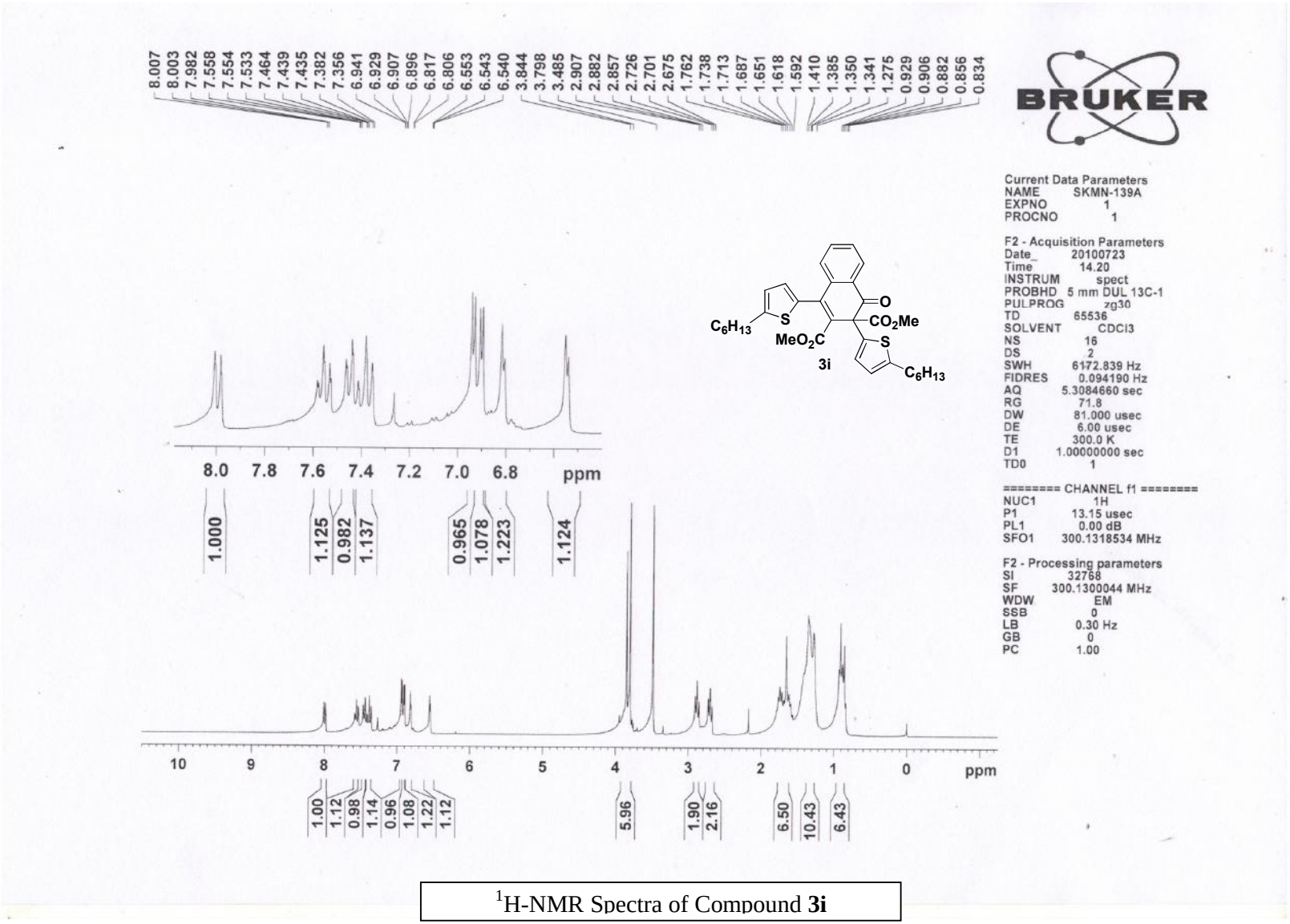

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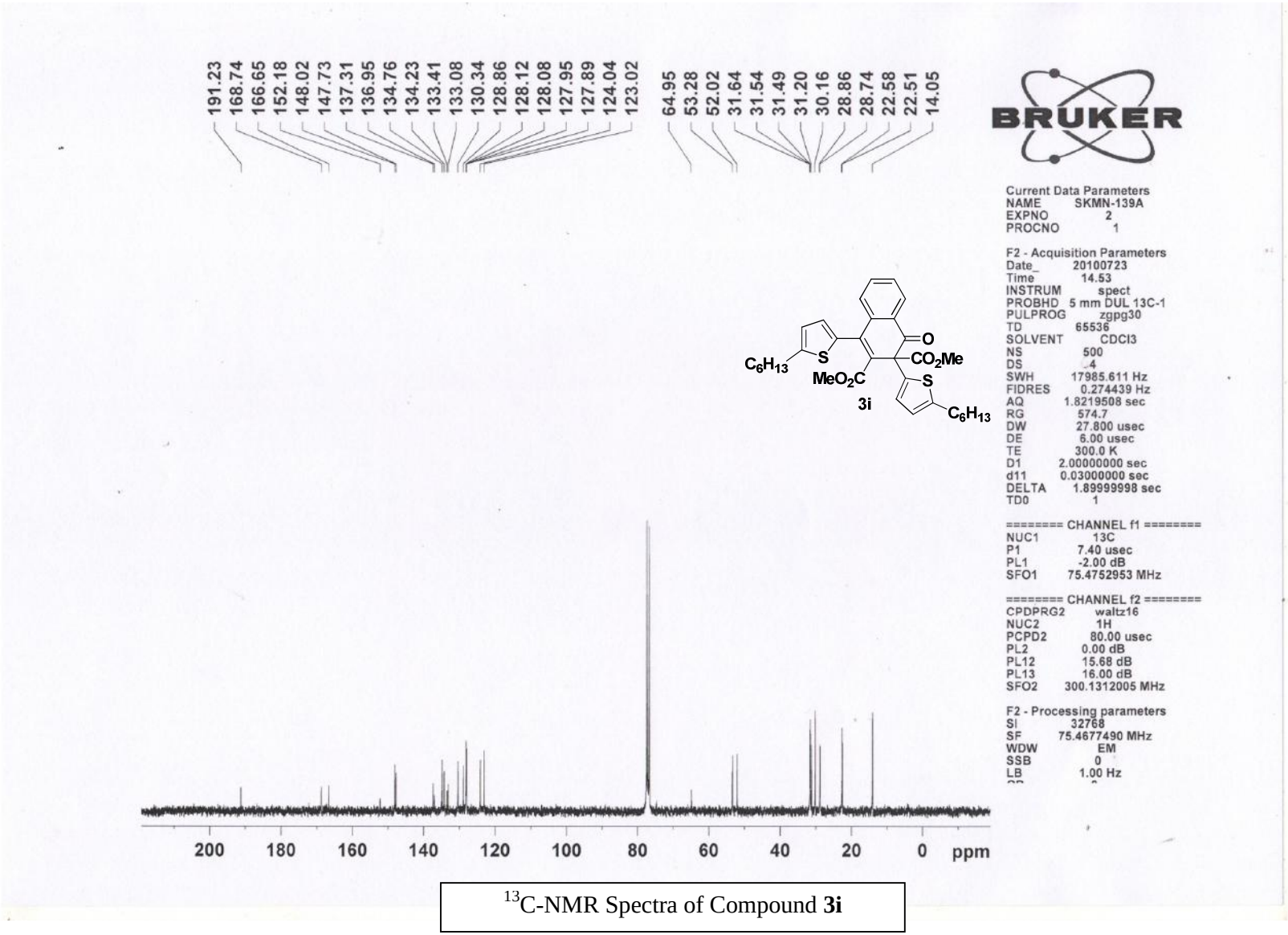

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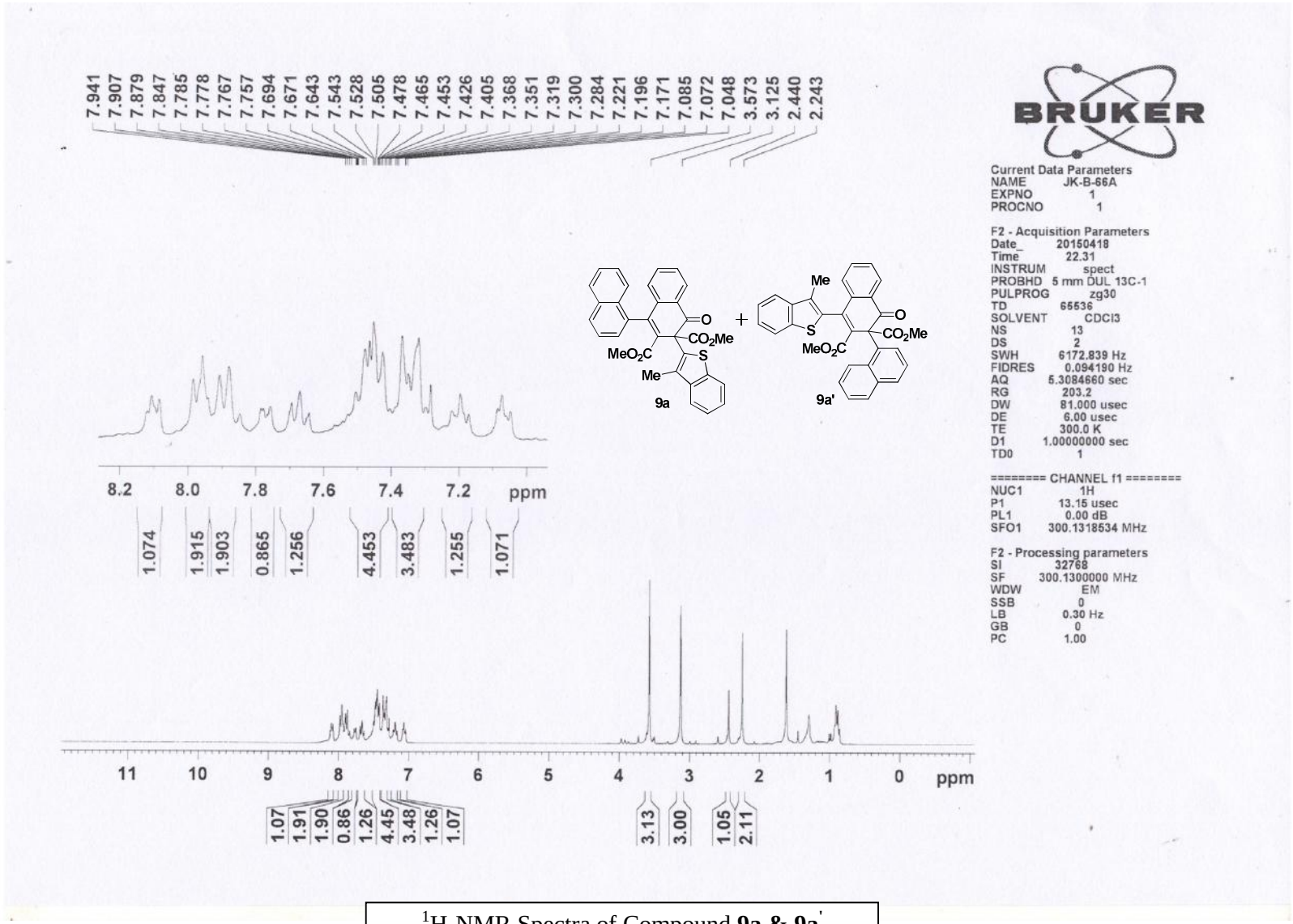

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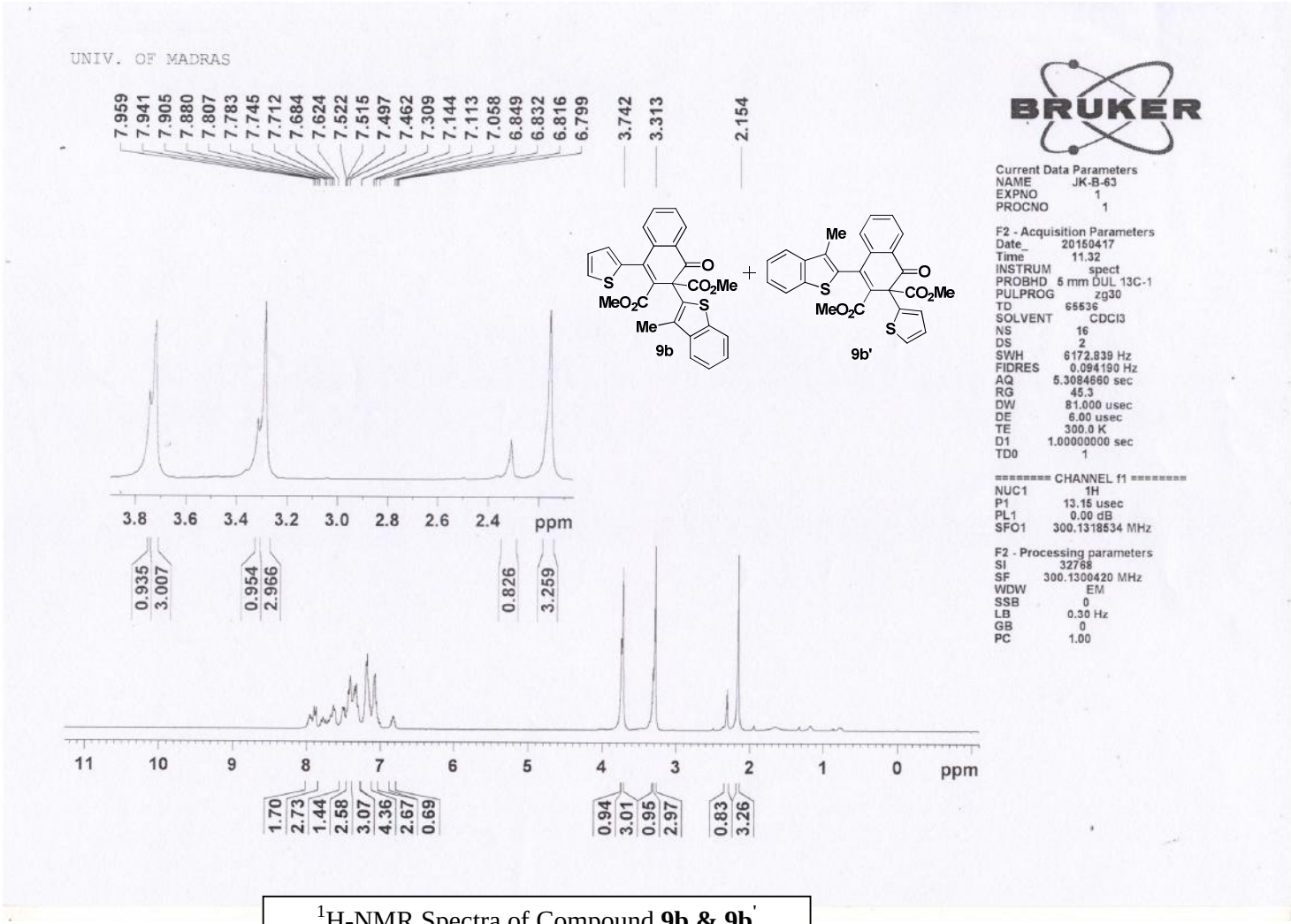

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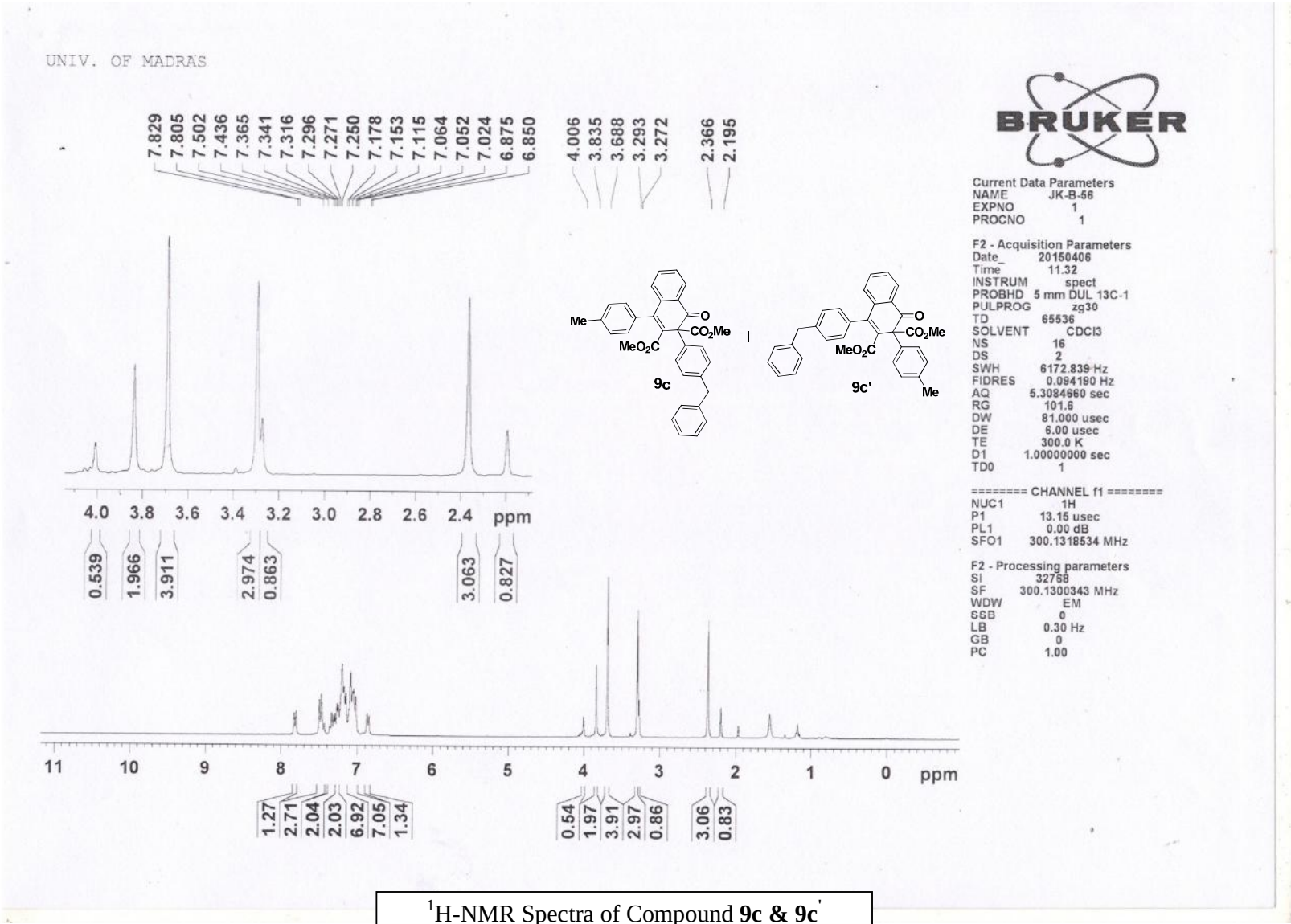

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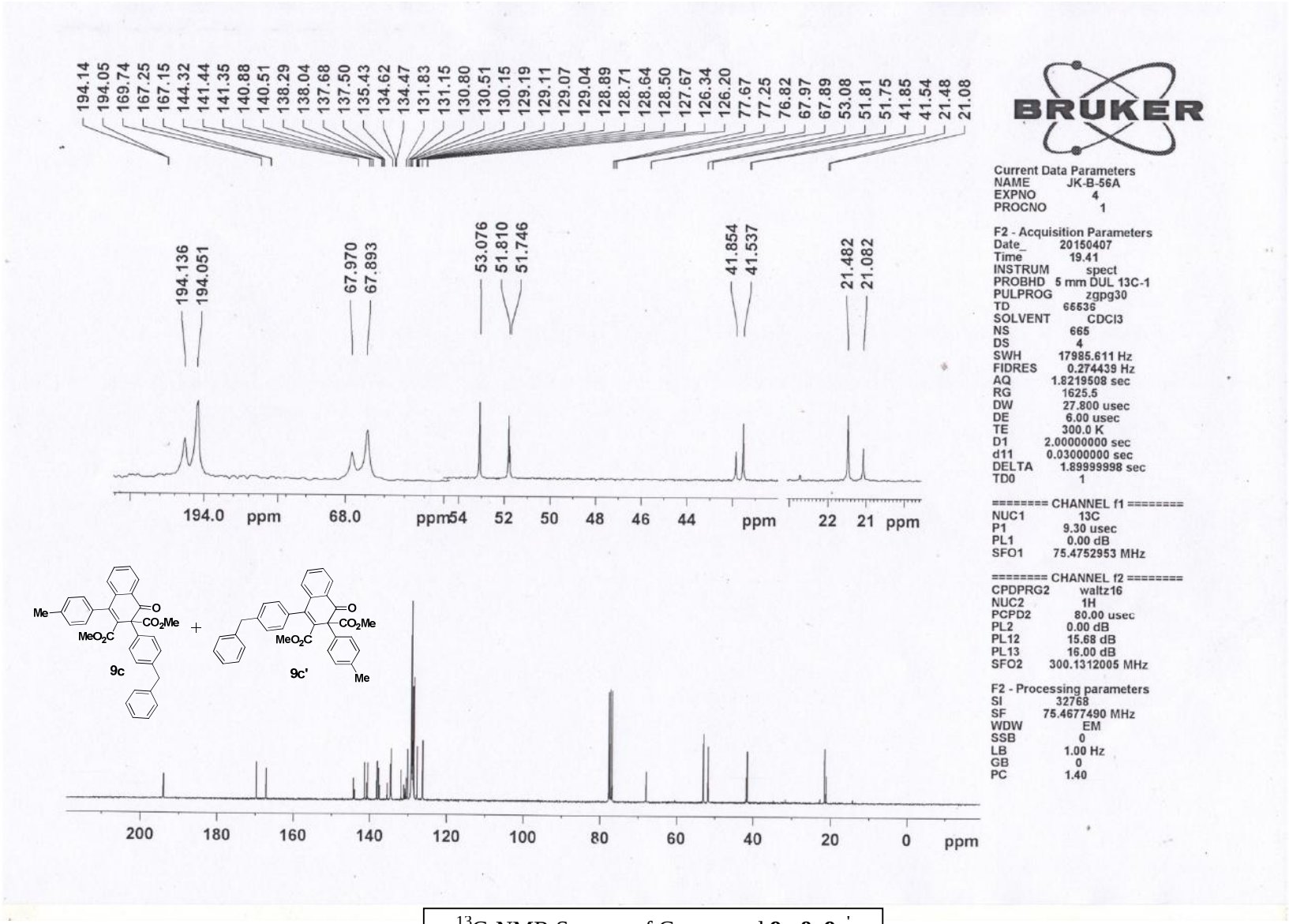

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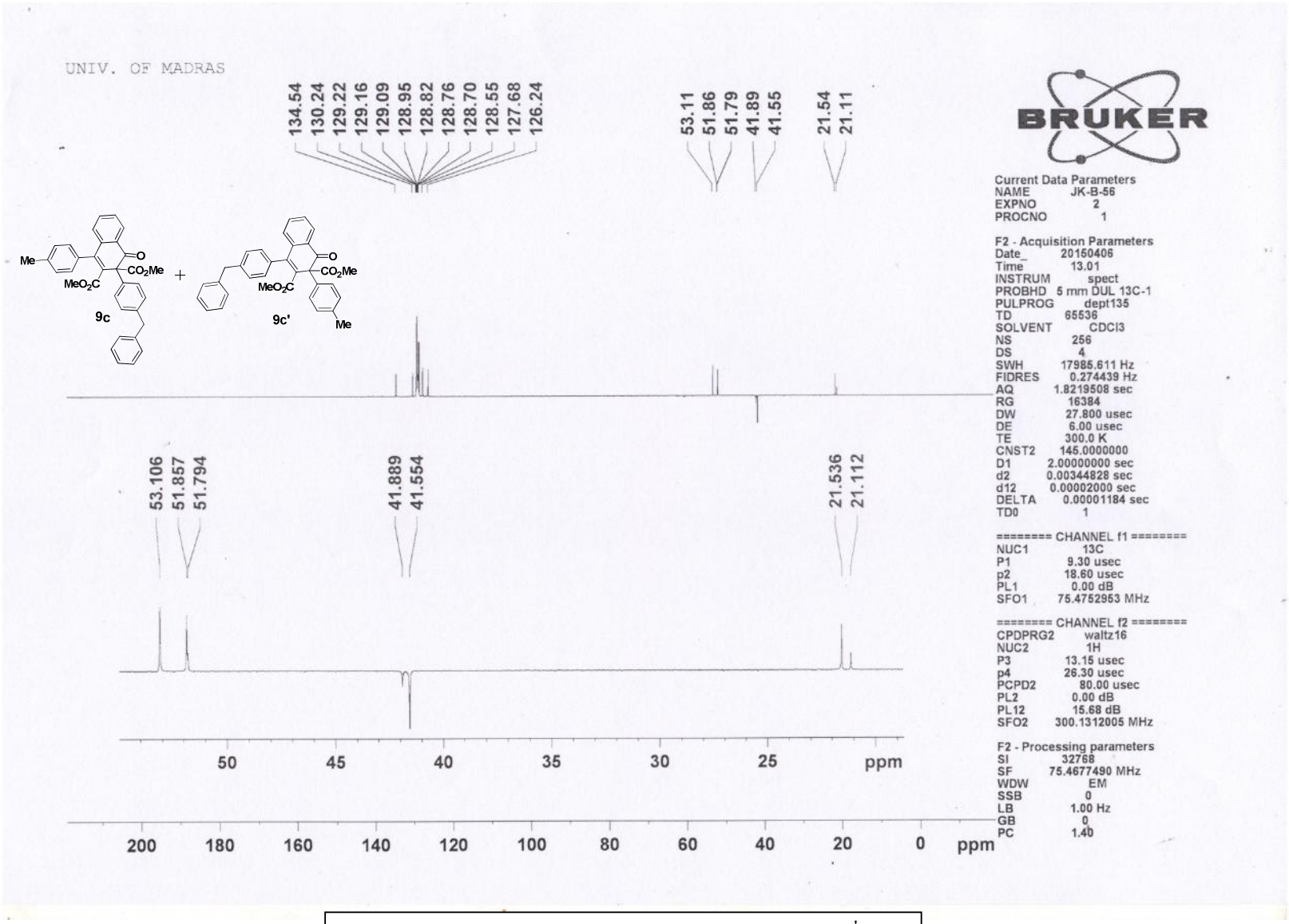

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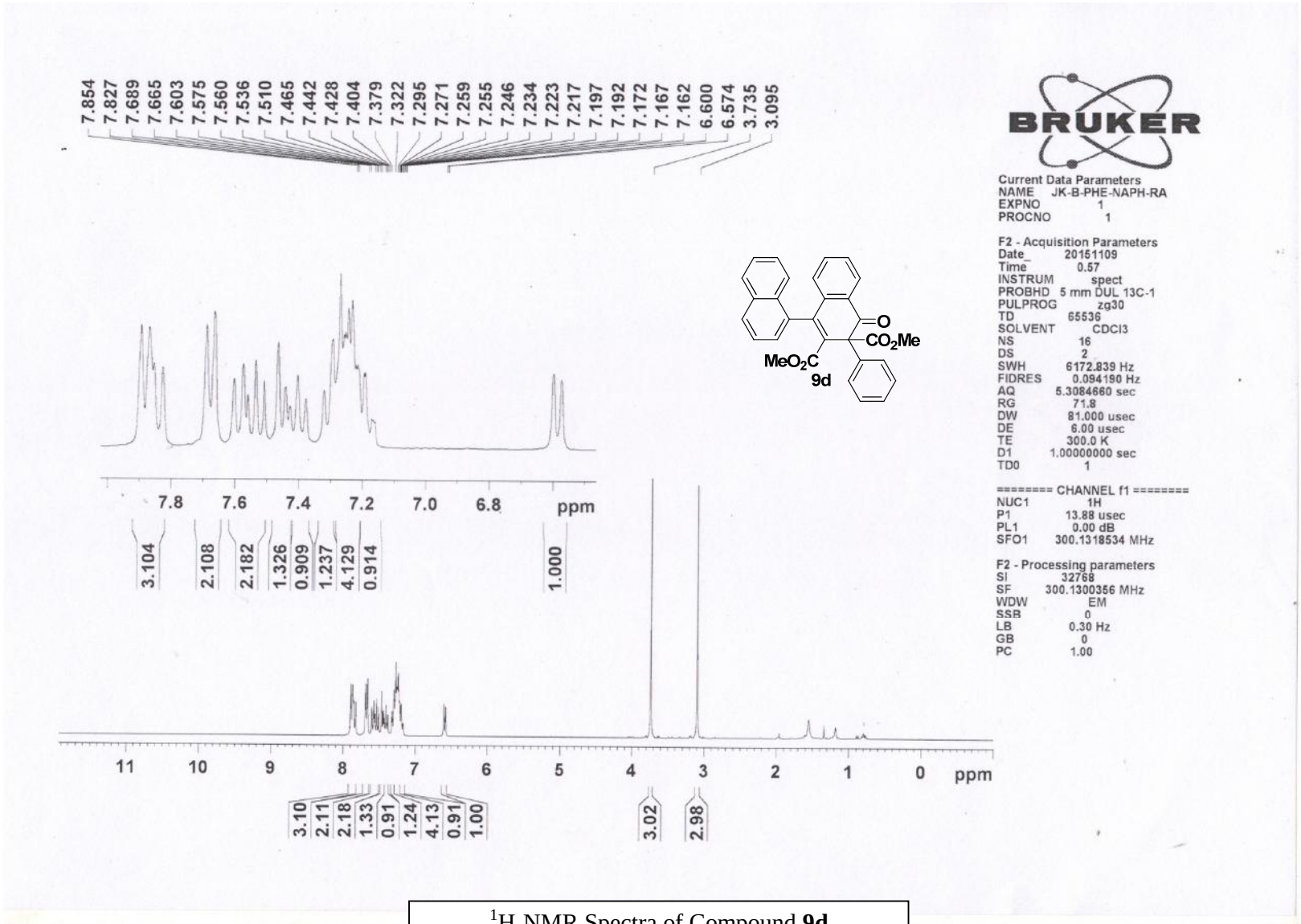

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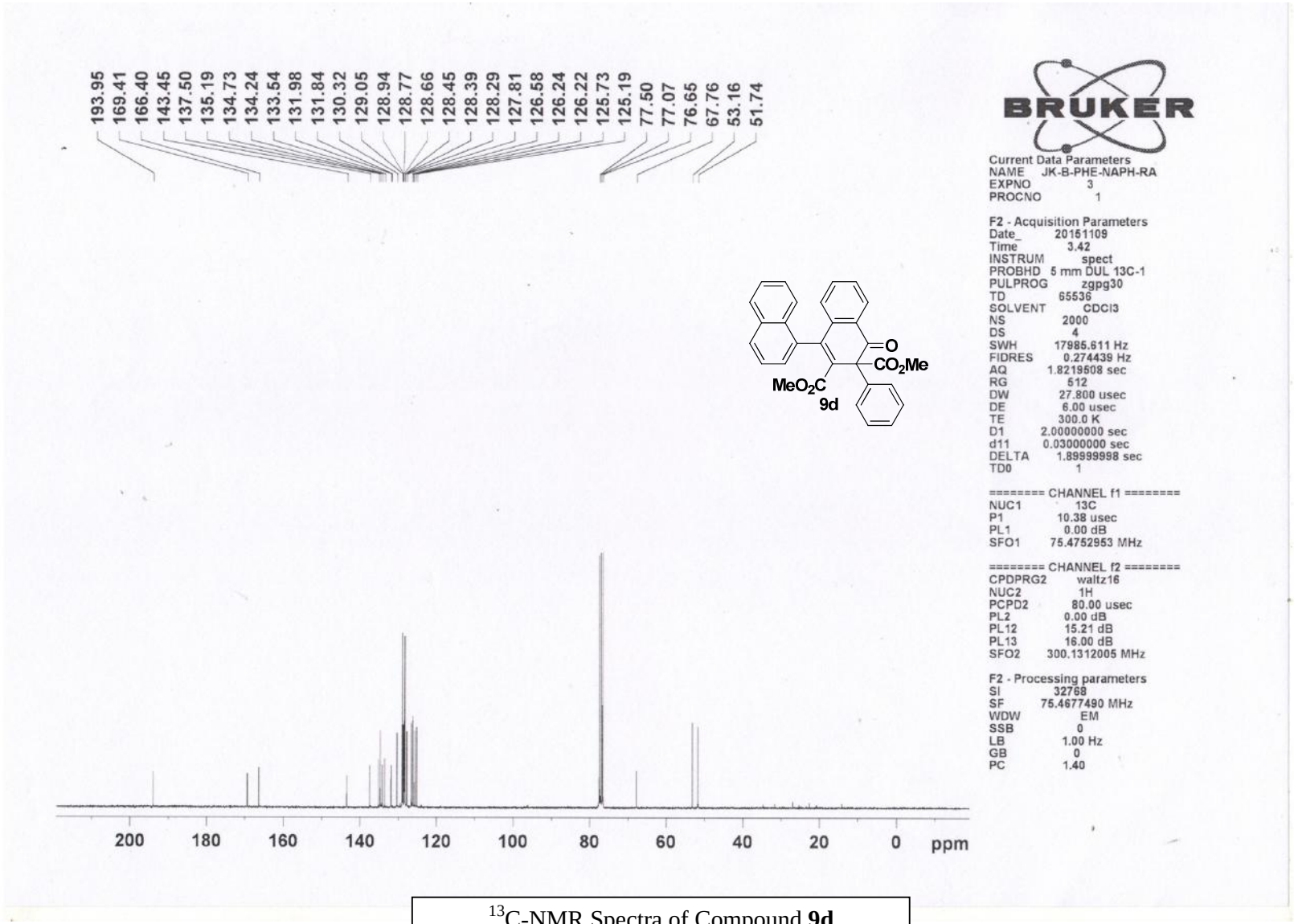

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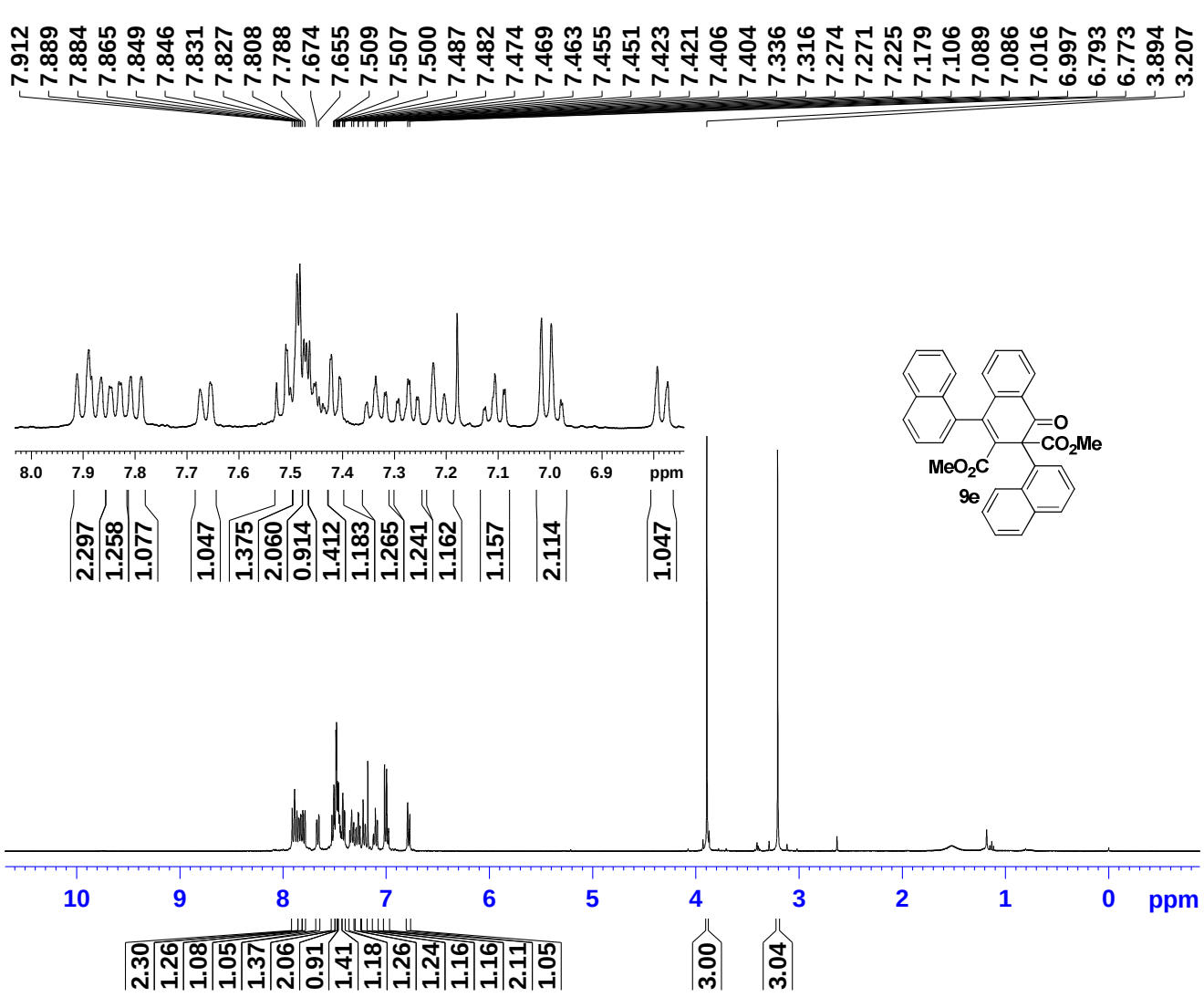

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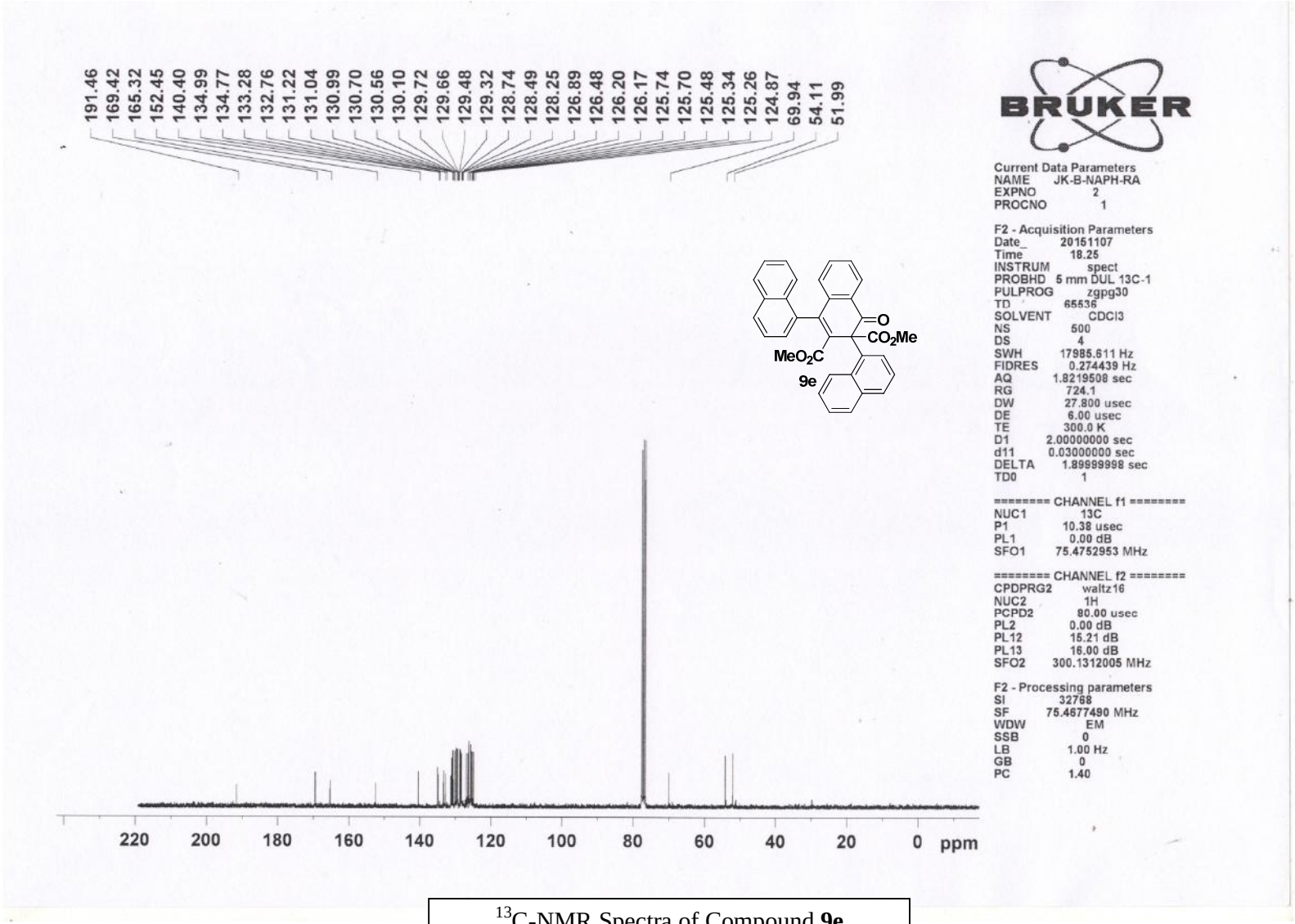

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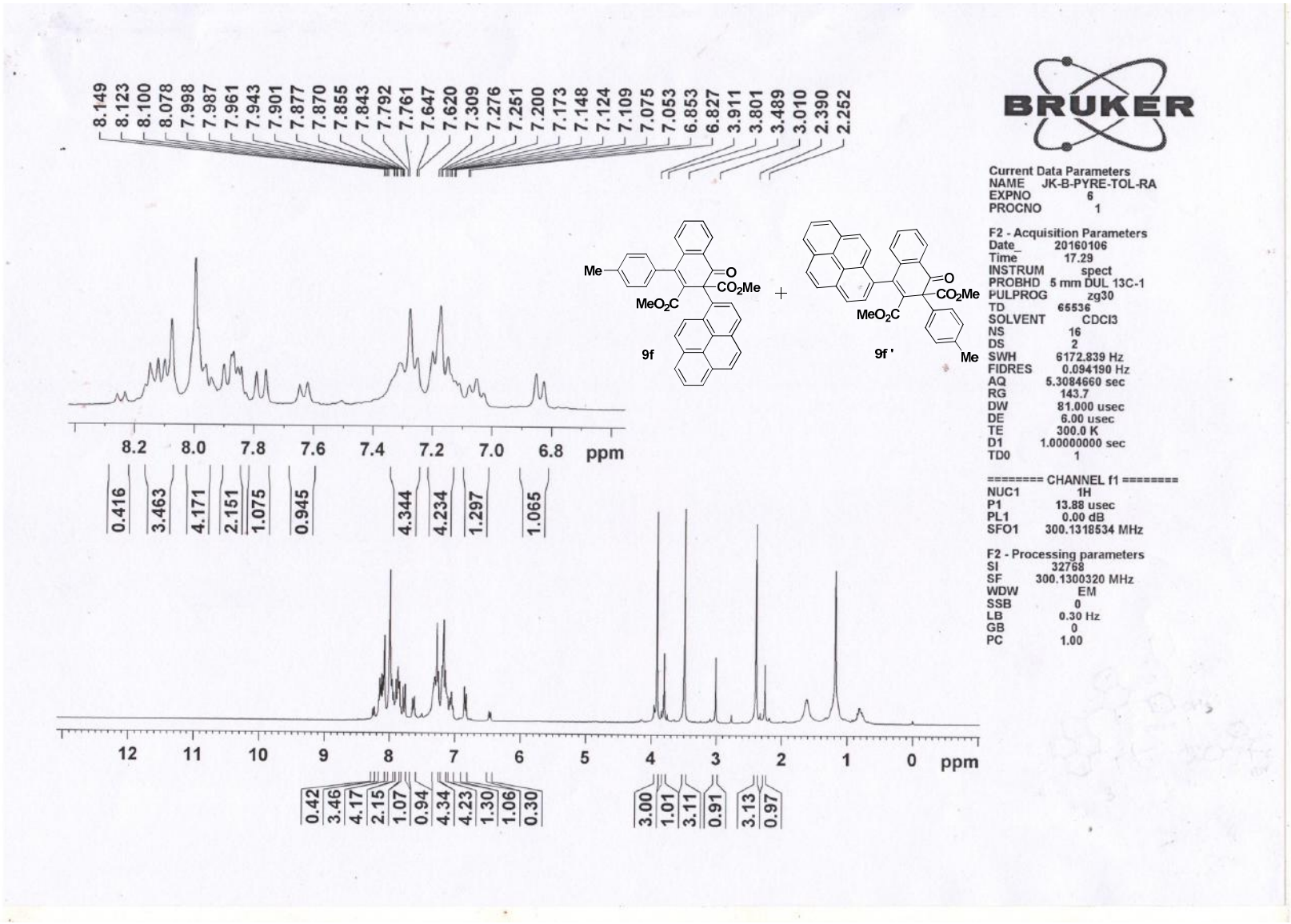

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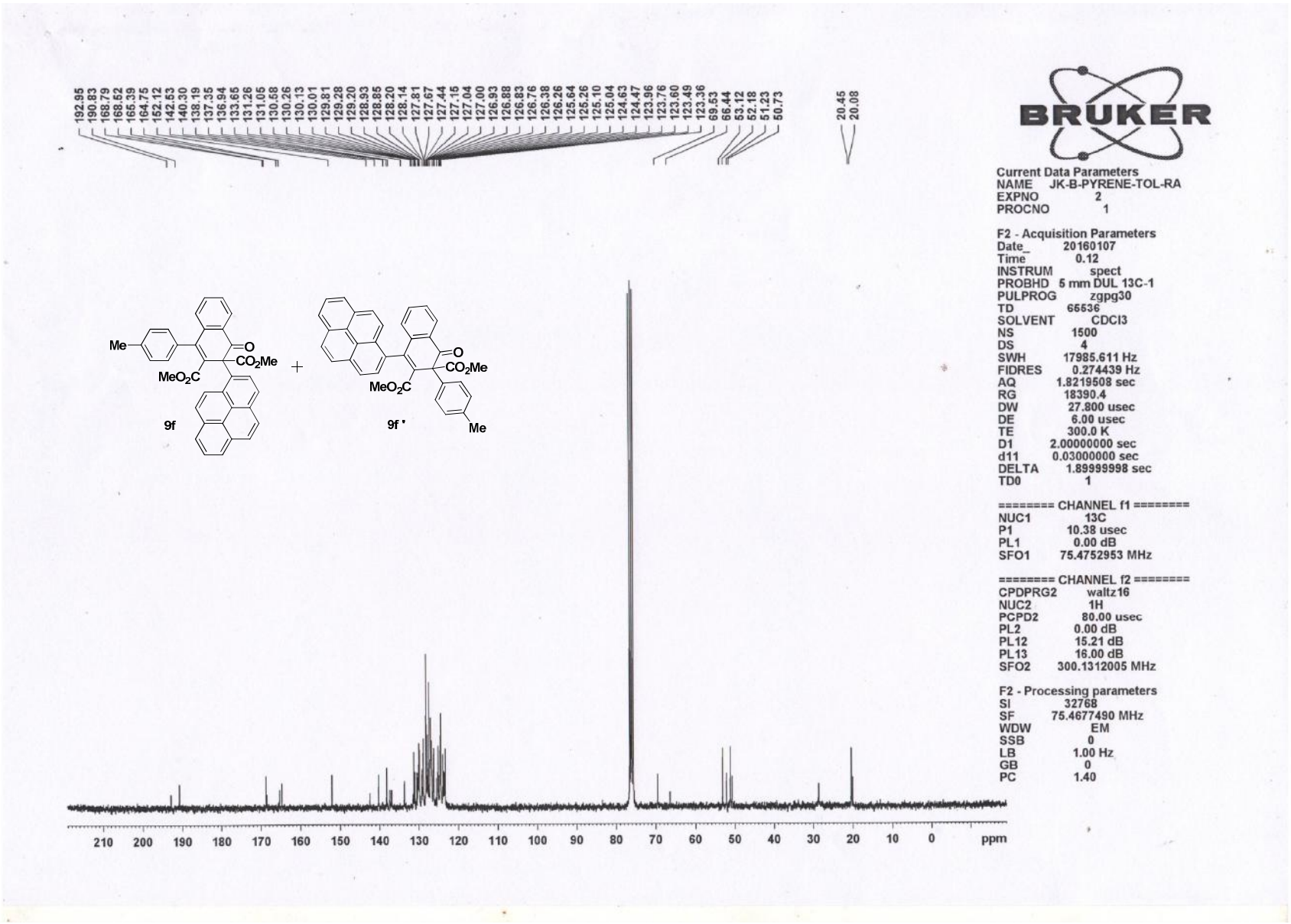

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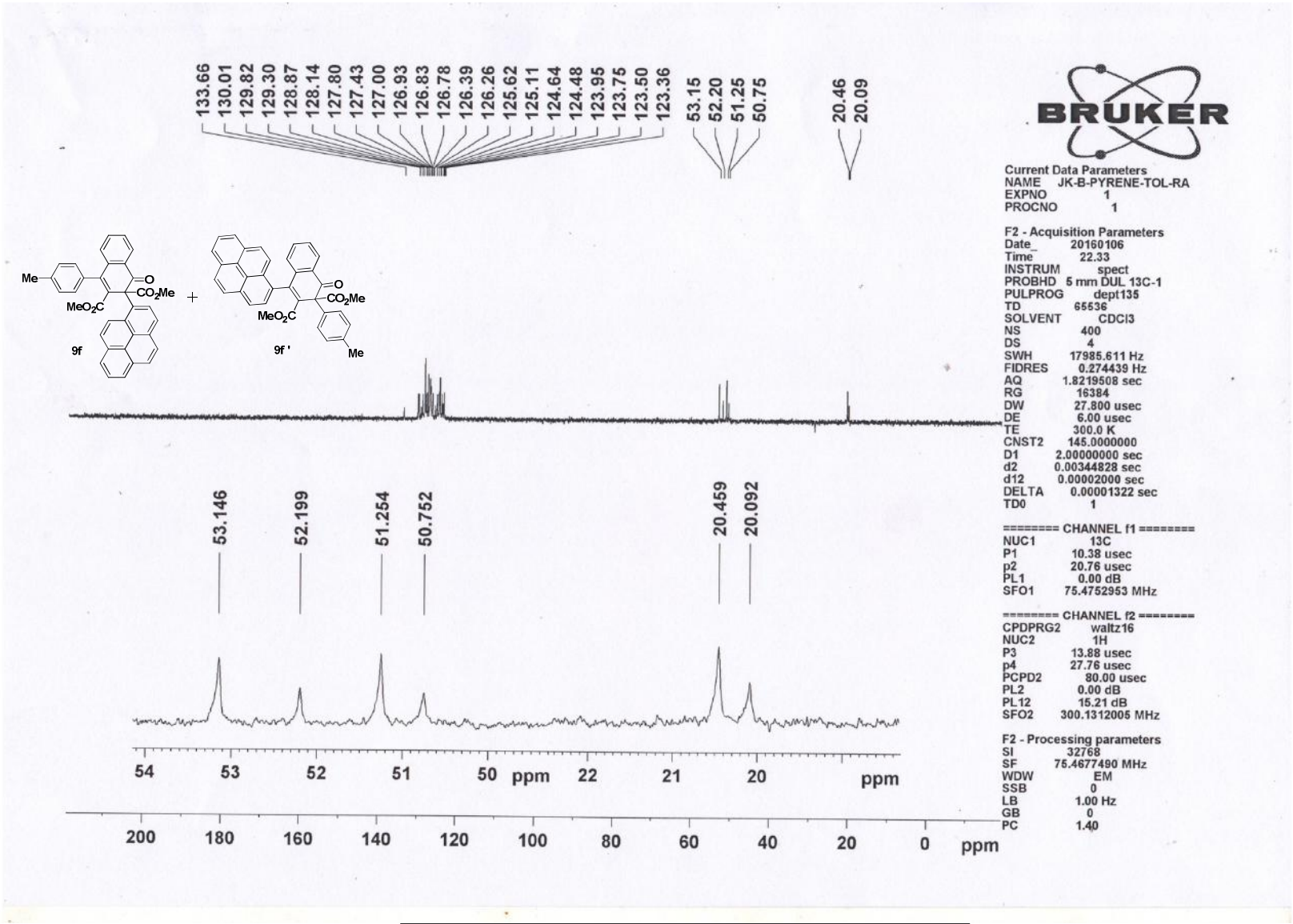

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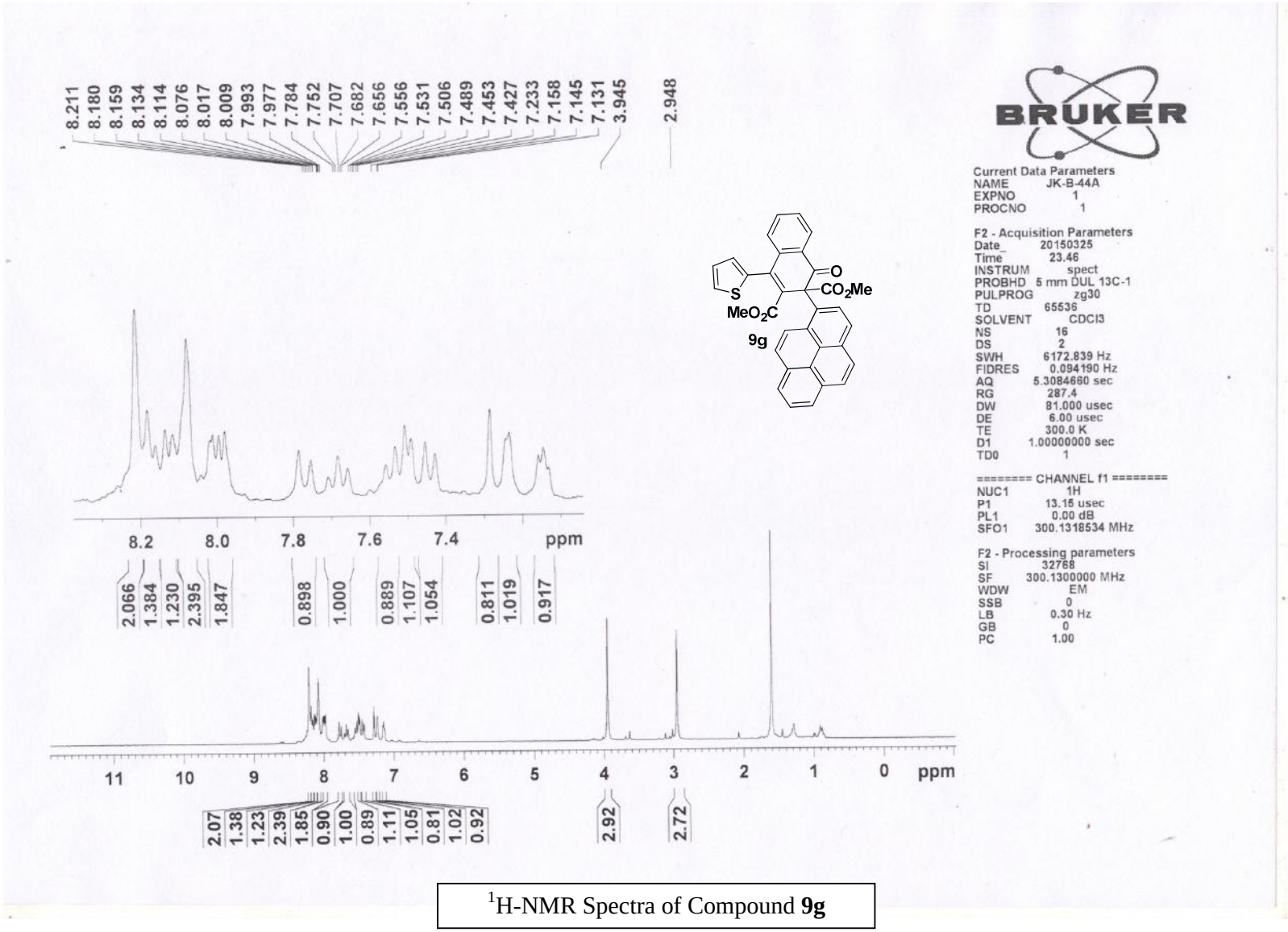

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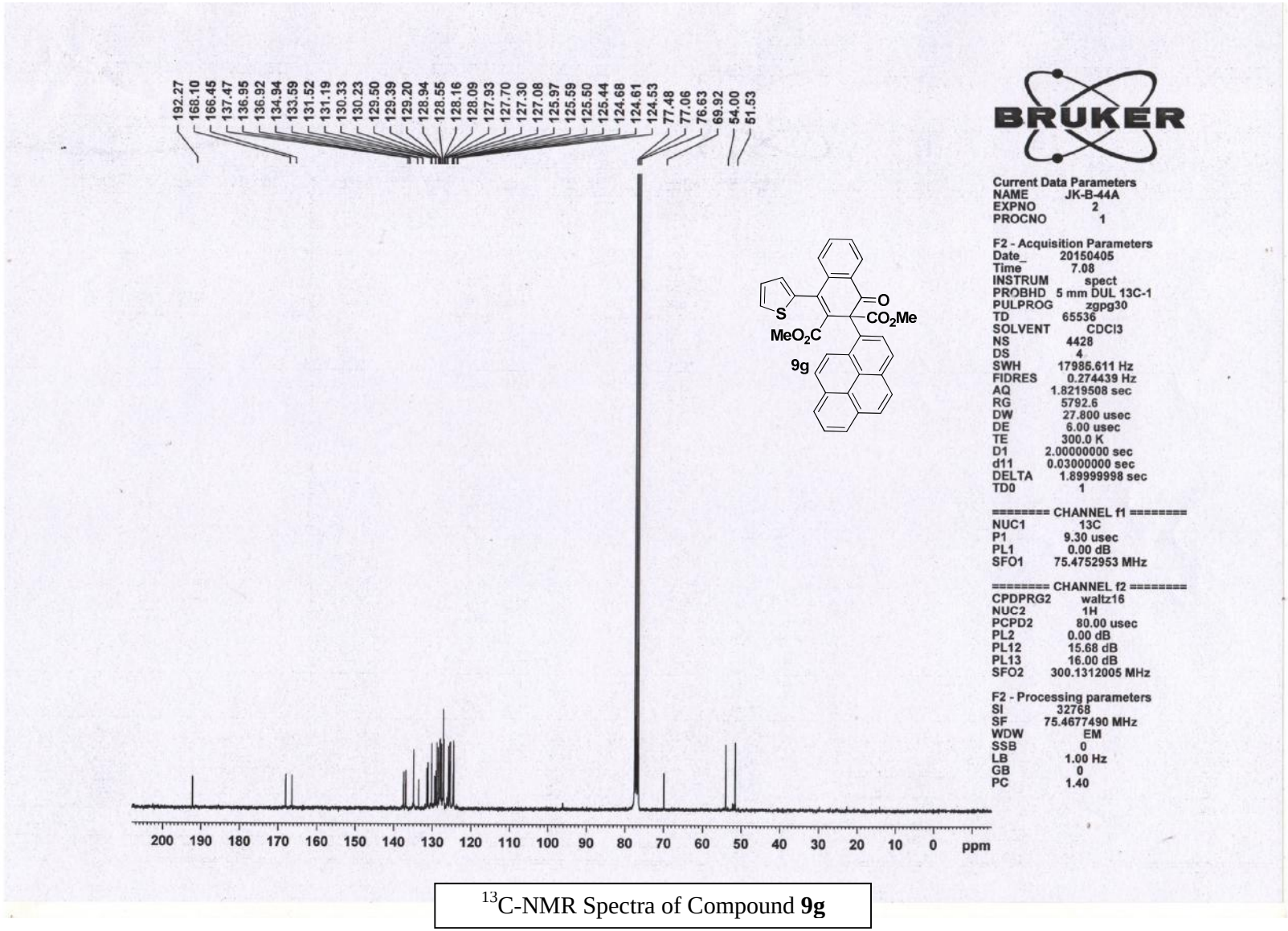

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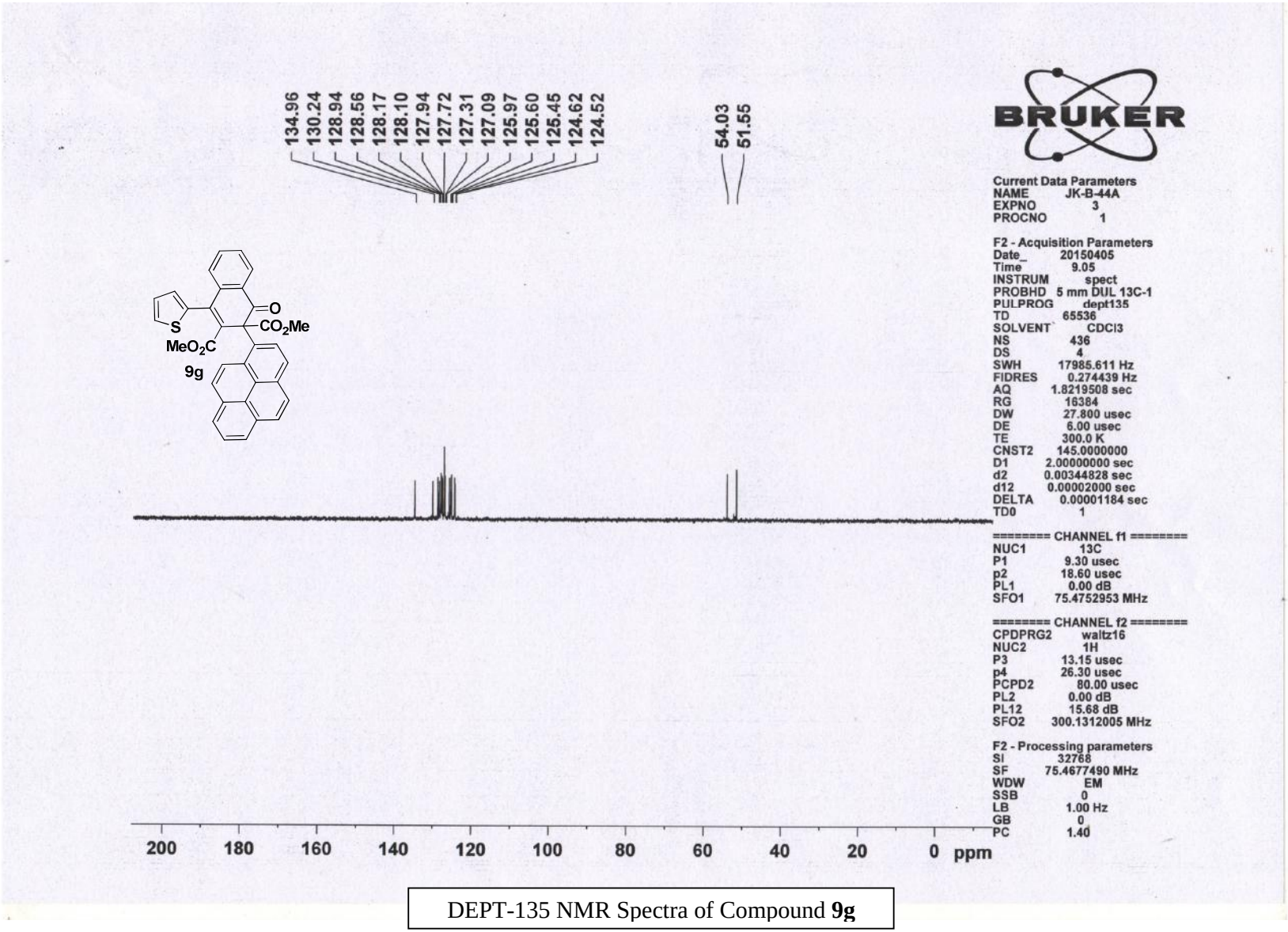

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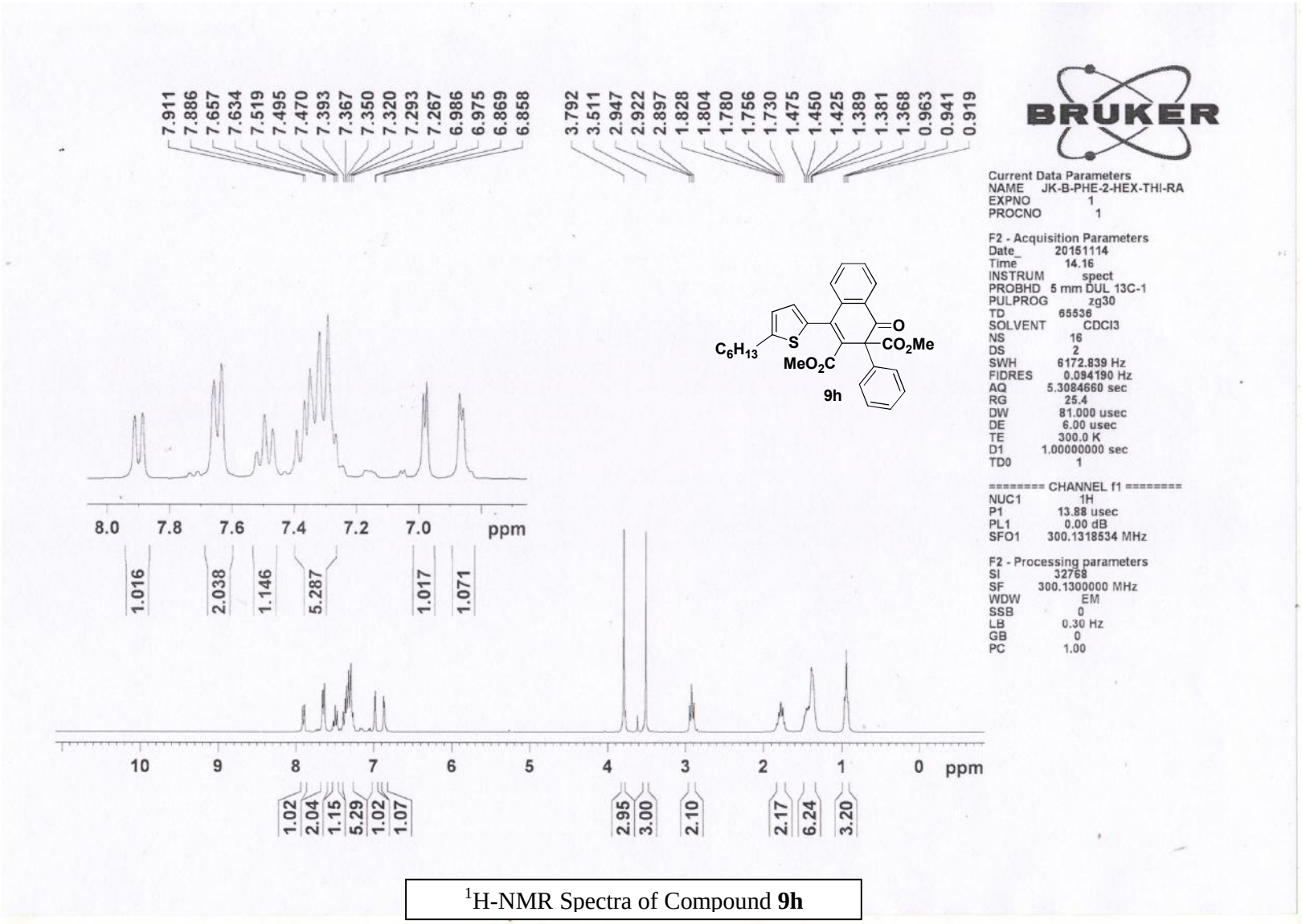

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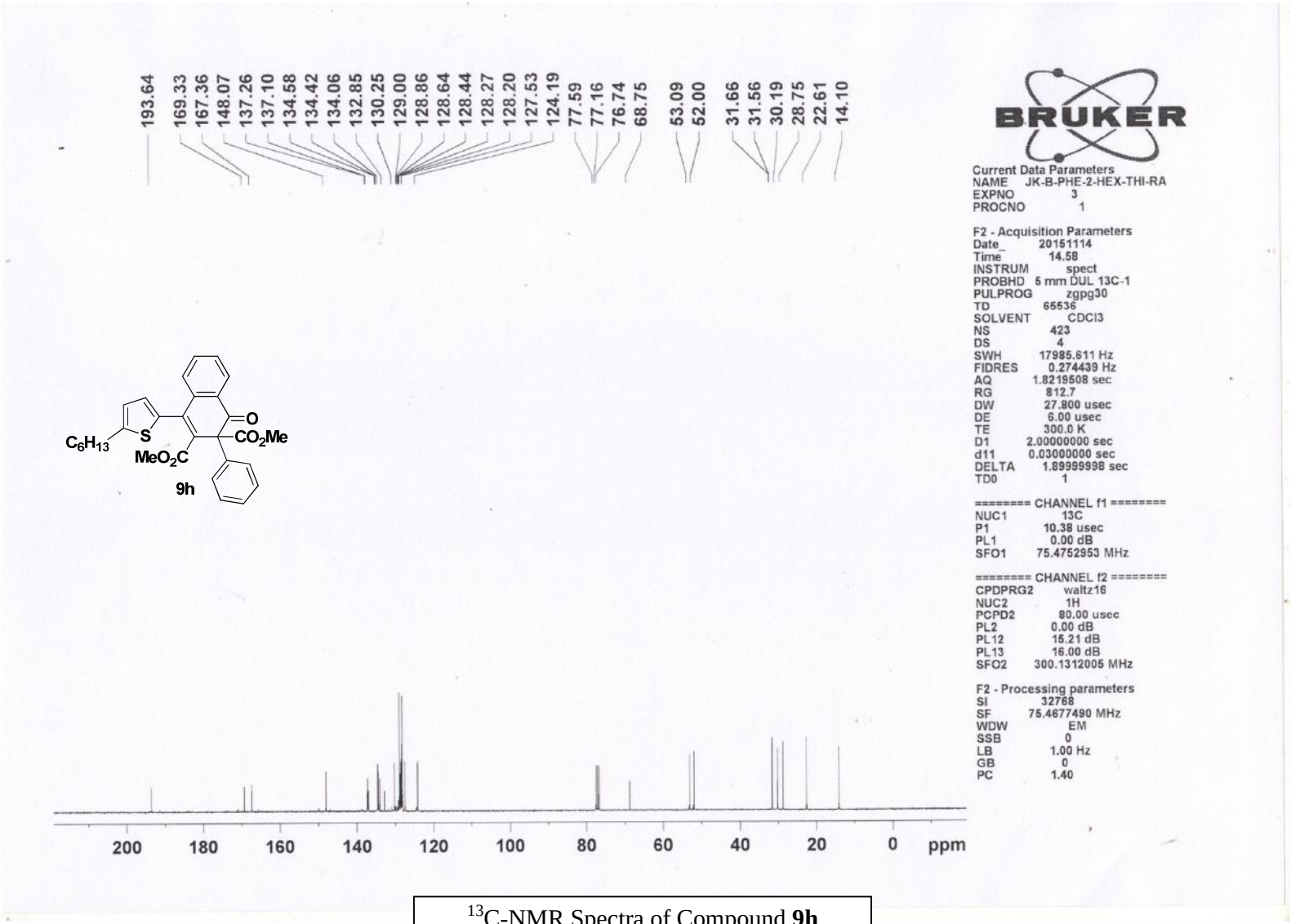

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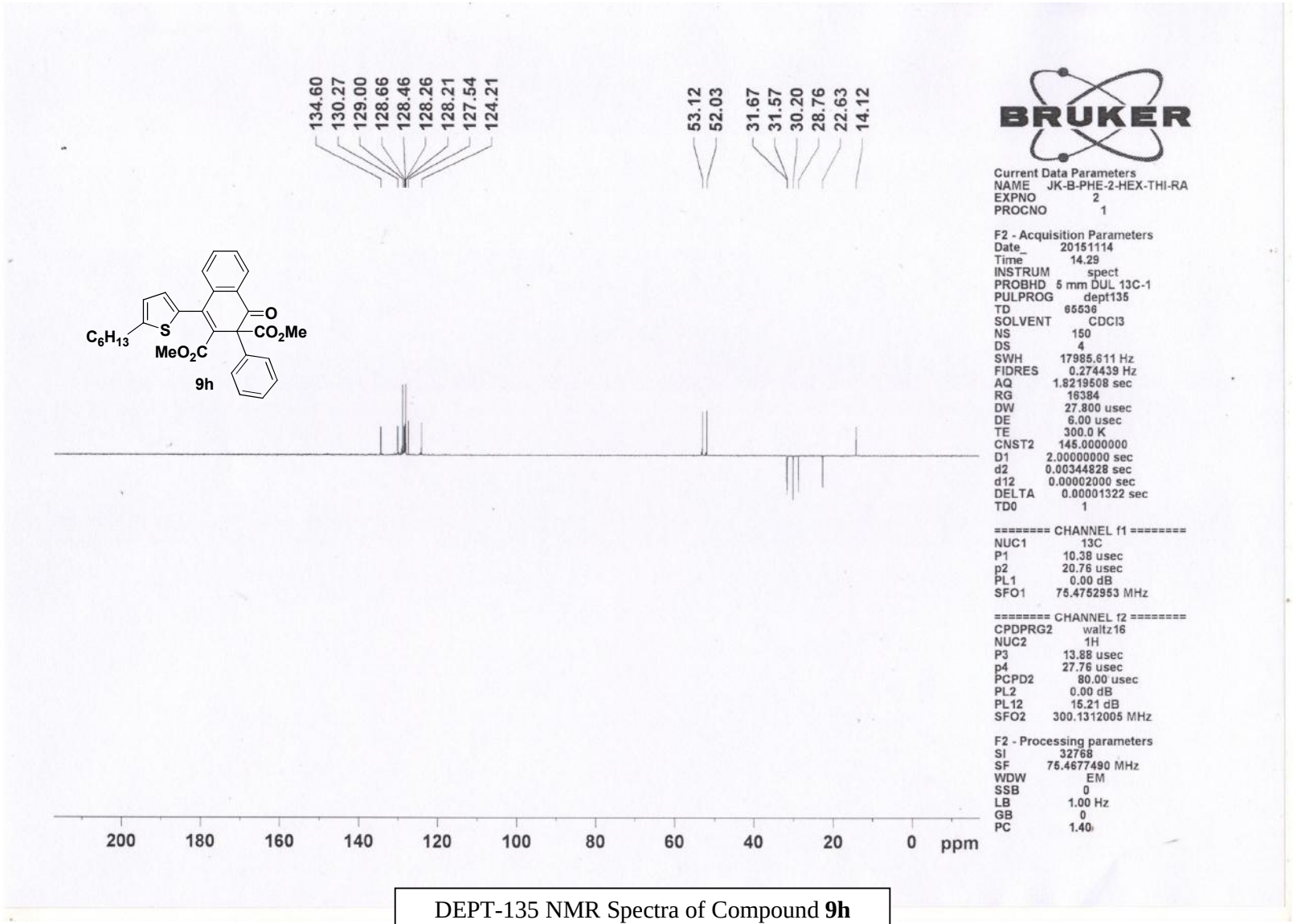

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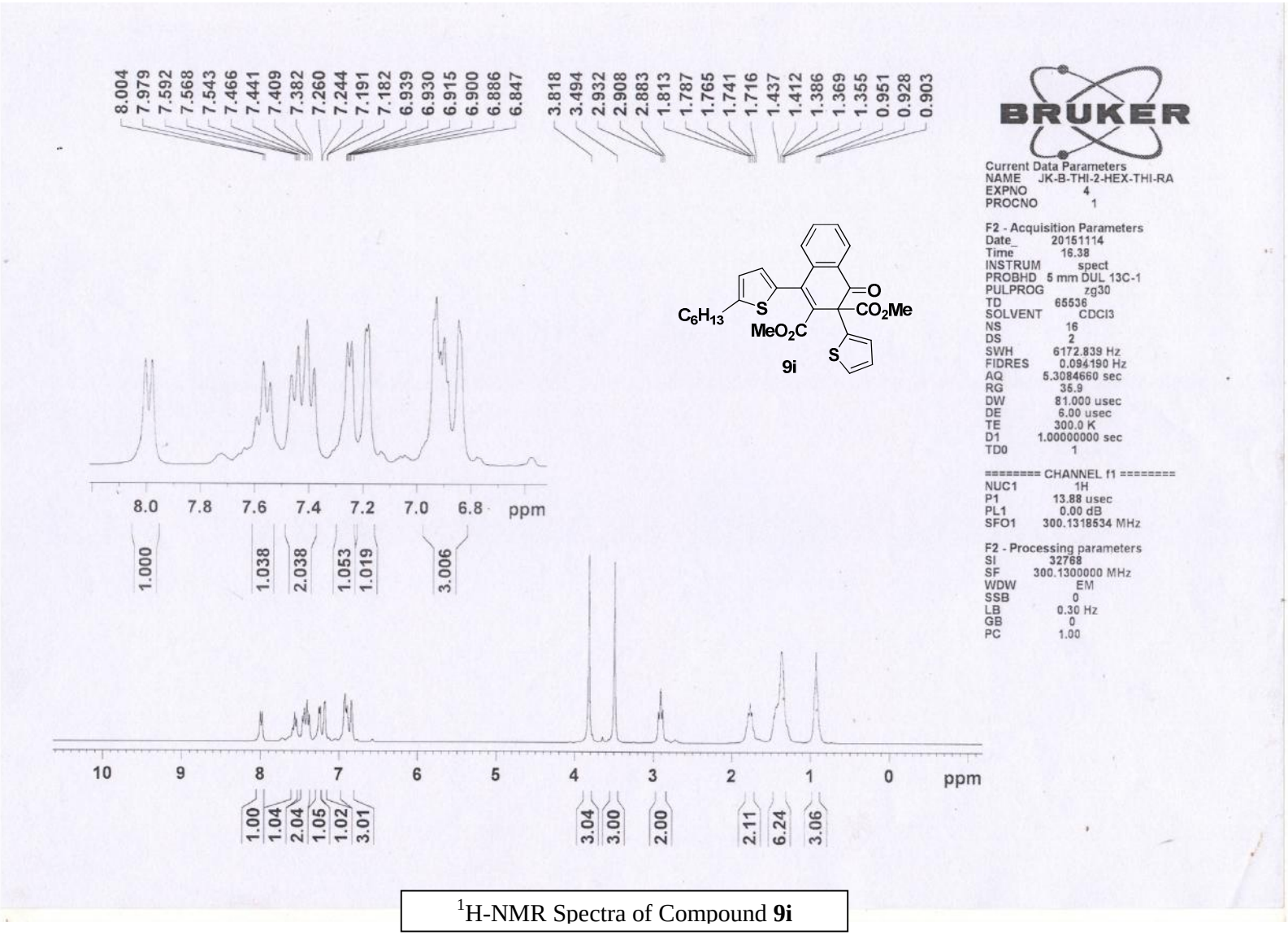

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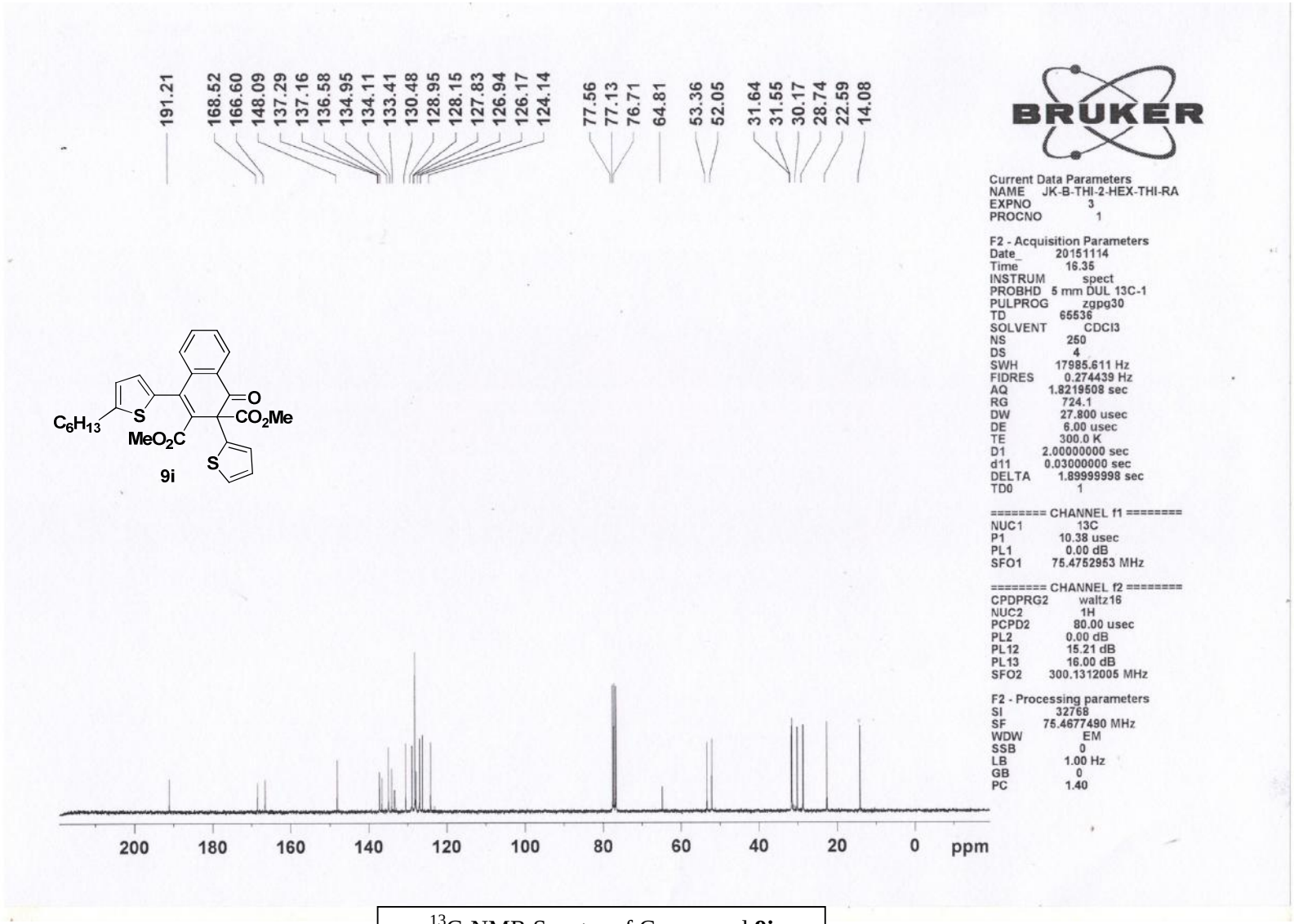

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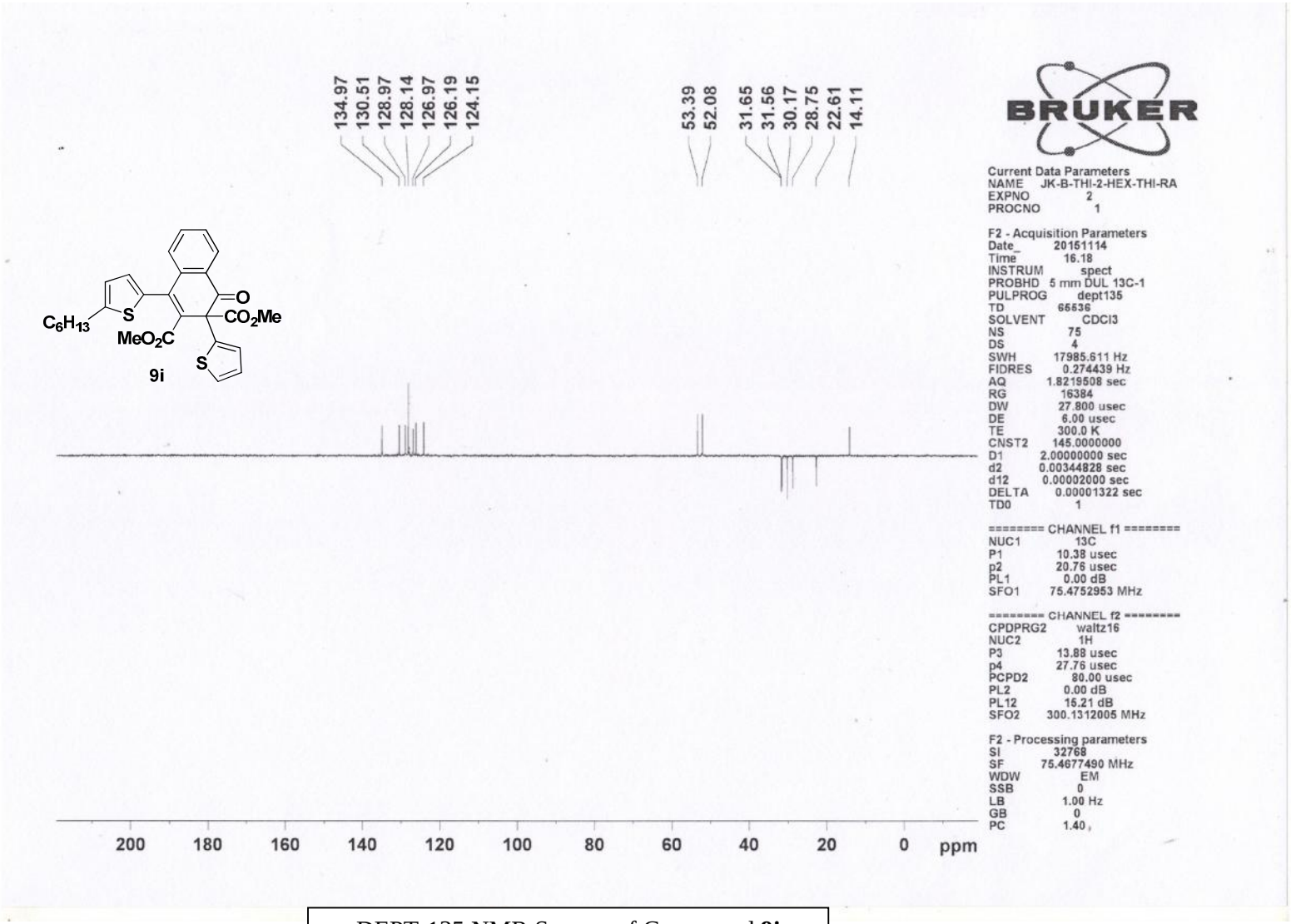
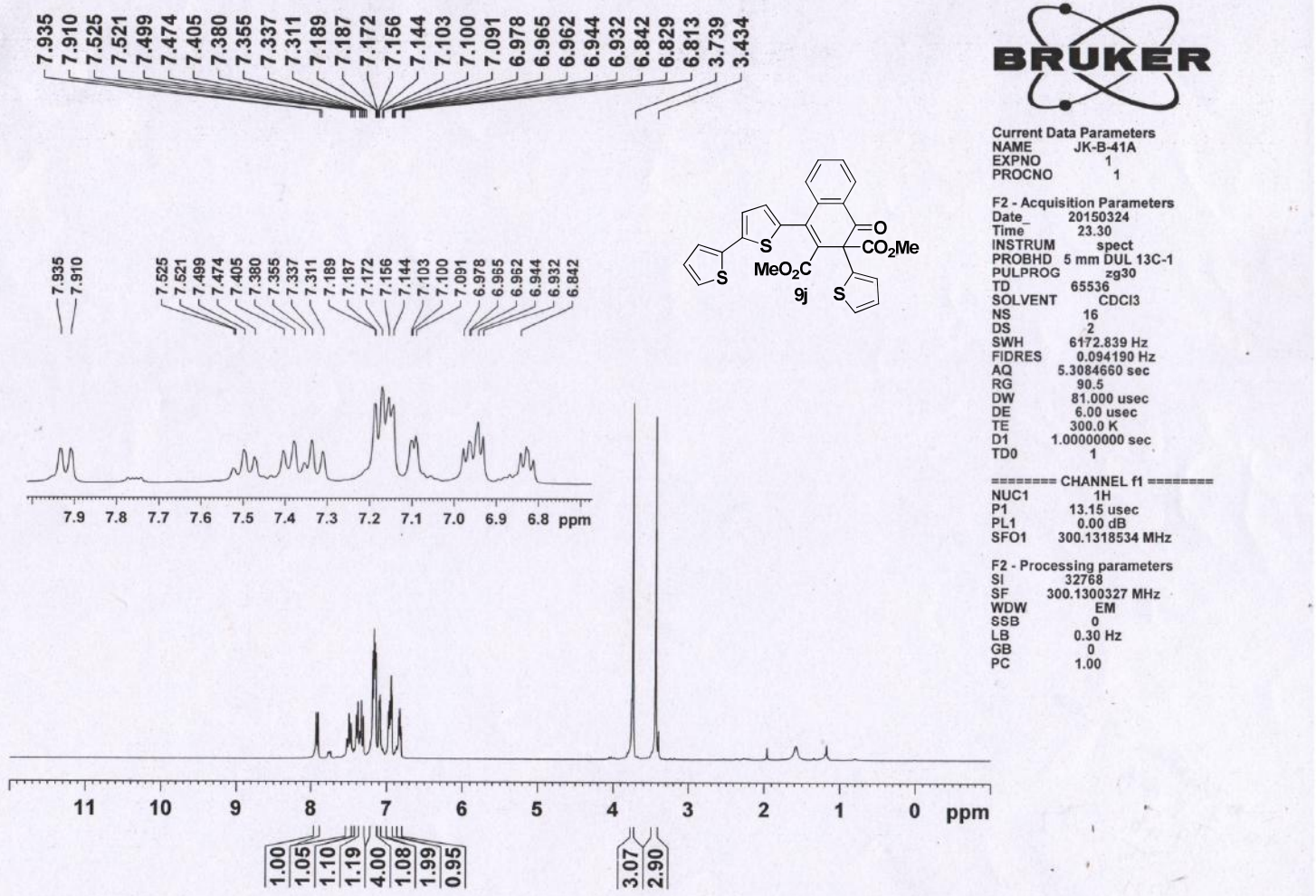
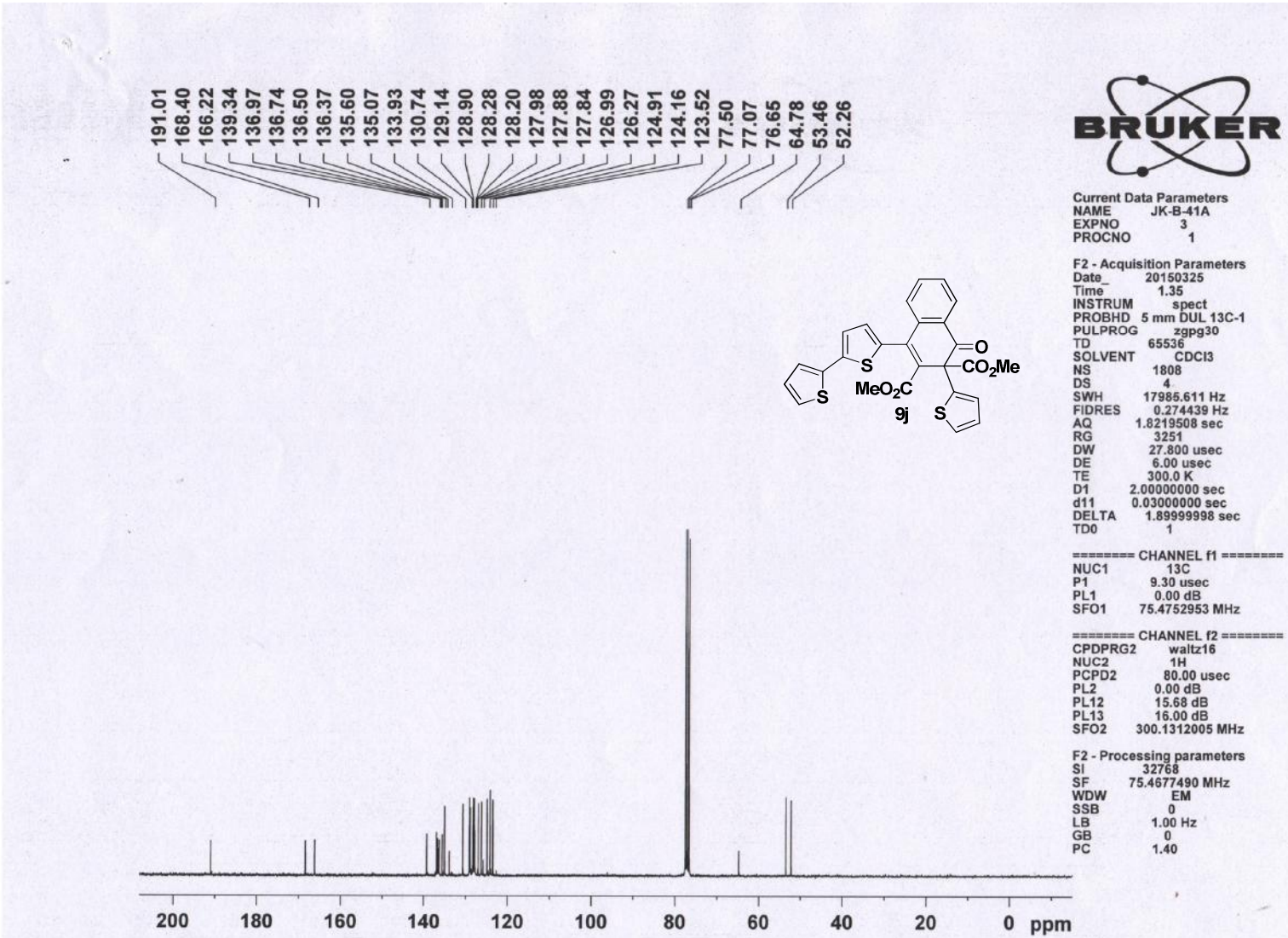


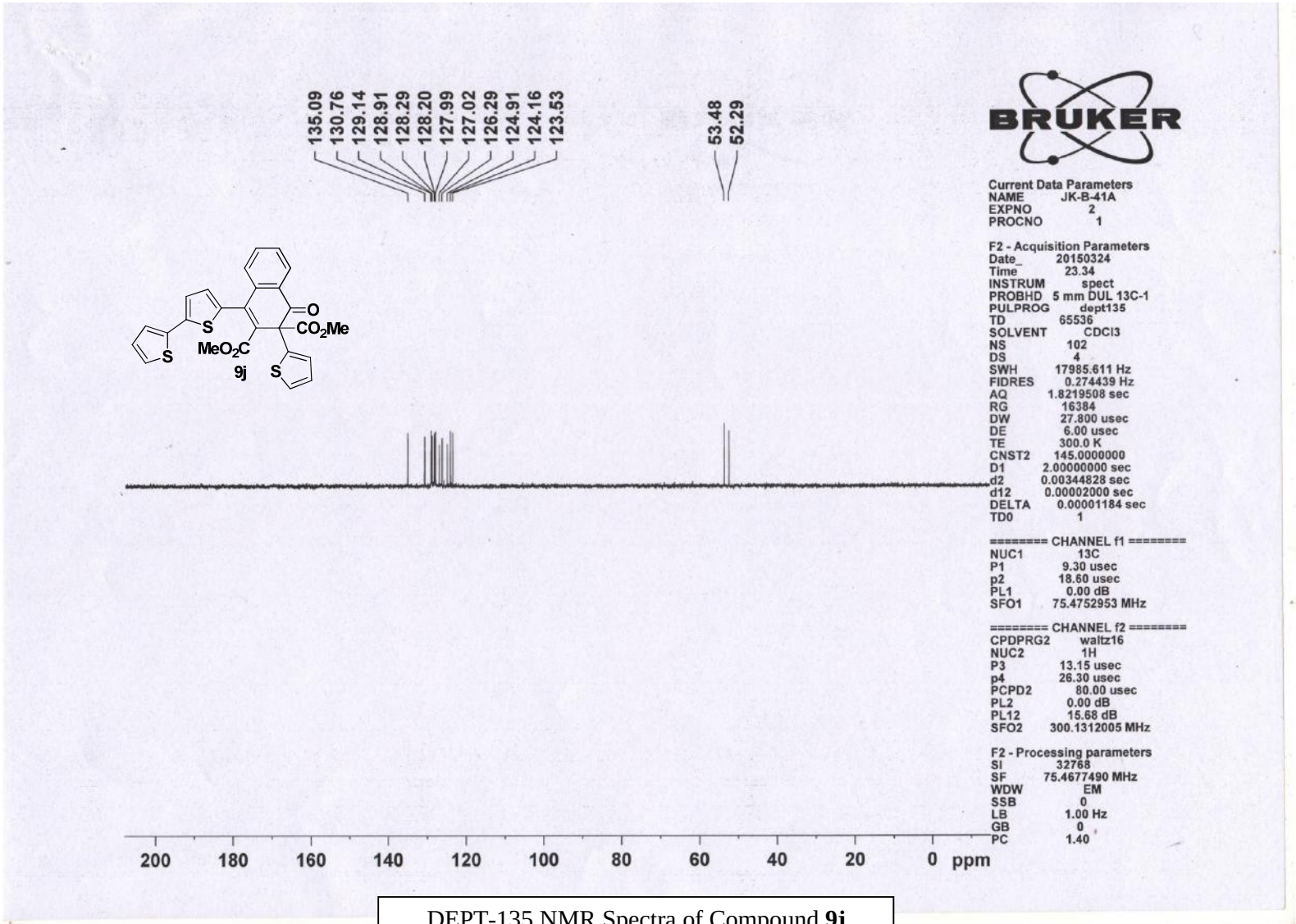
DEPT-135 NMR Spectra of Compound **9i**



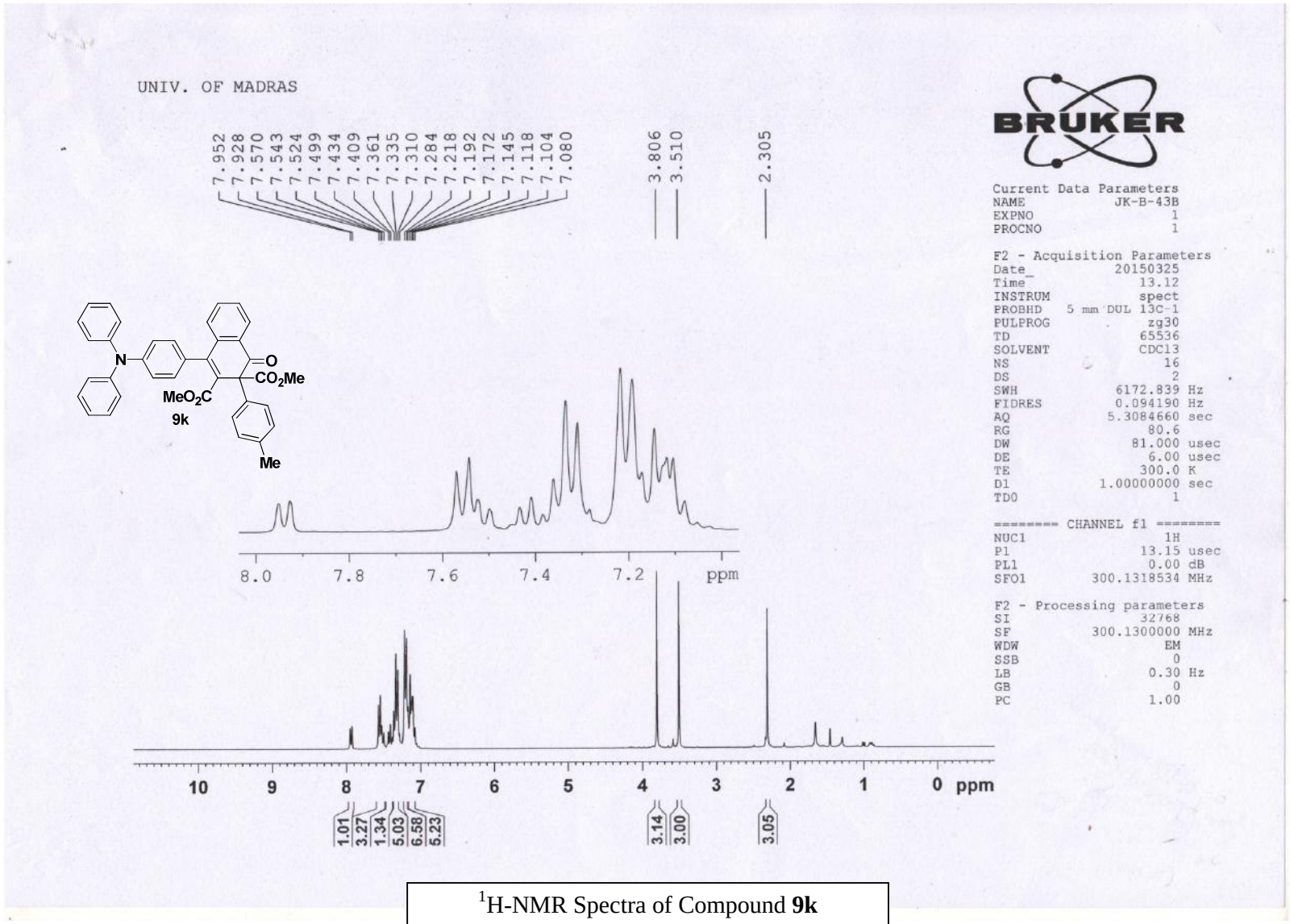
^{1}H-NMR Spectra of Compound **9j**



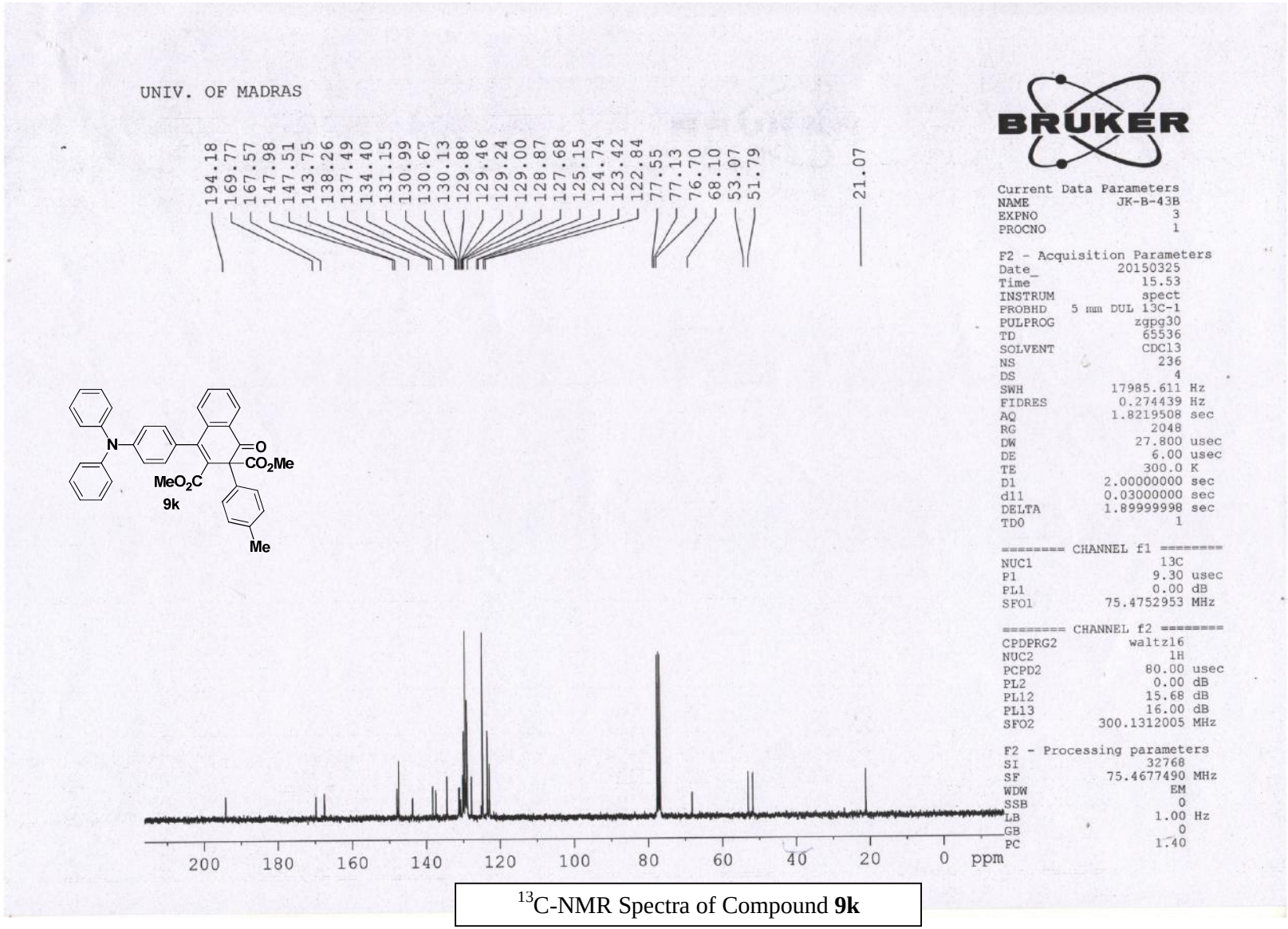
^{13}C-NMR Spectra of Compound **9j**



DEPT-135 NMR Spectra of Compound **9j**



^{1}H-NMR Spectra of Compound **9k**



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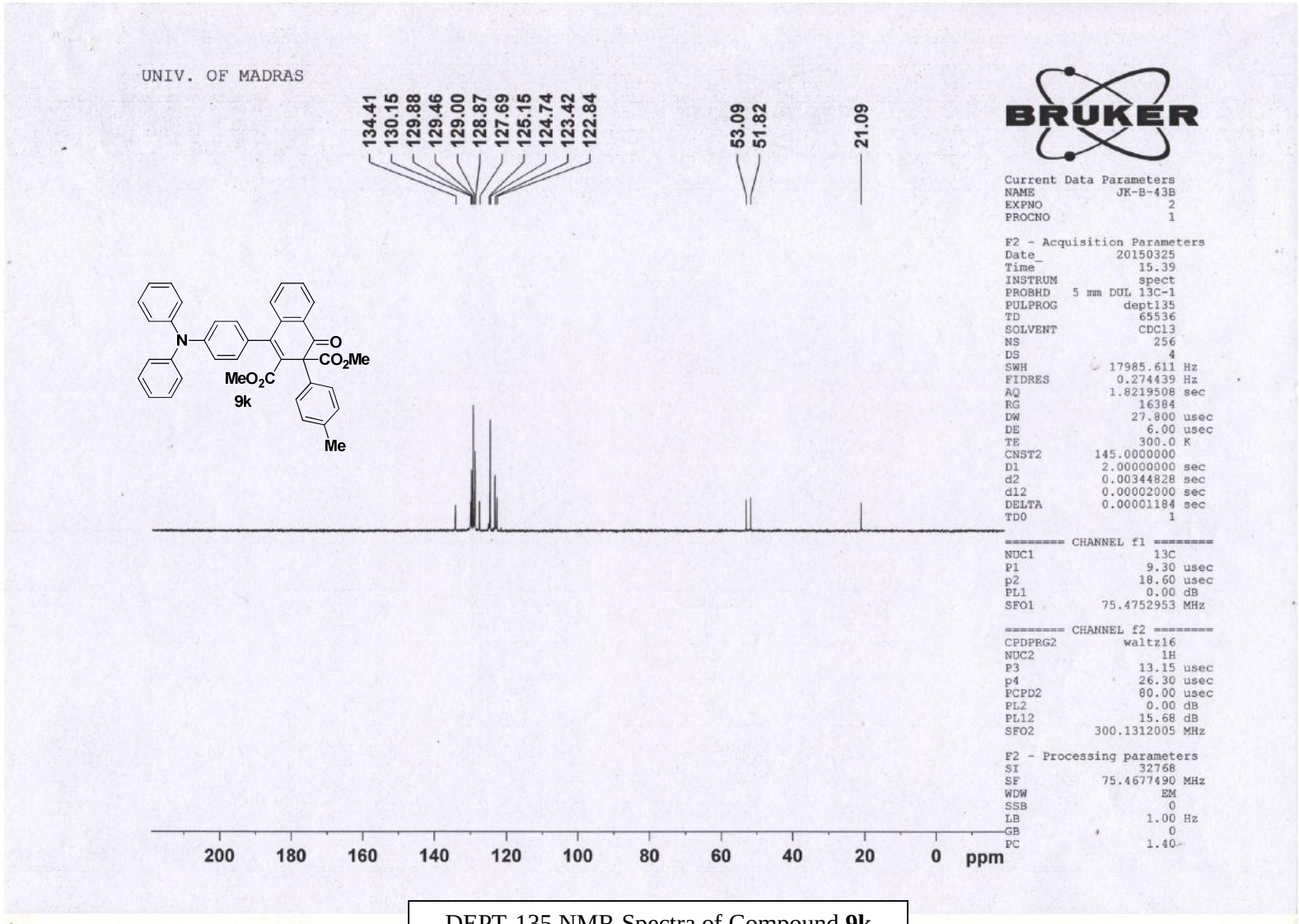
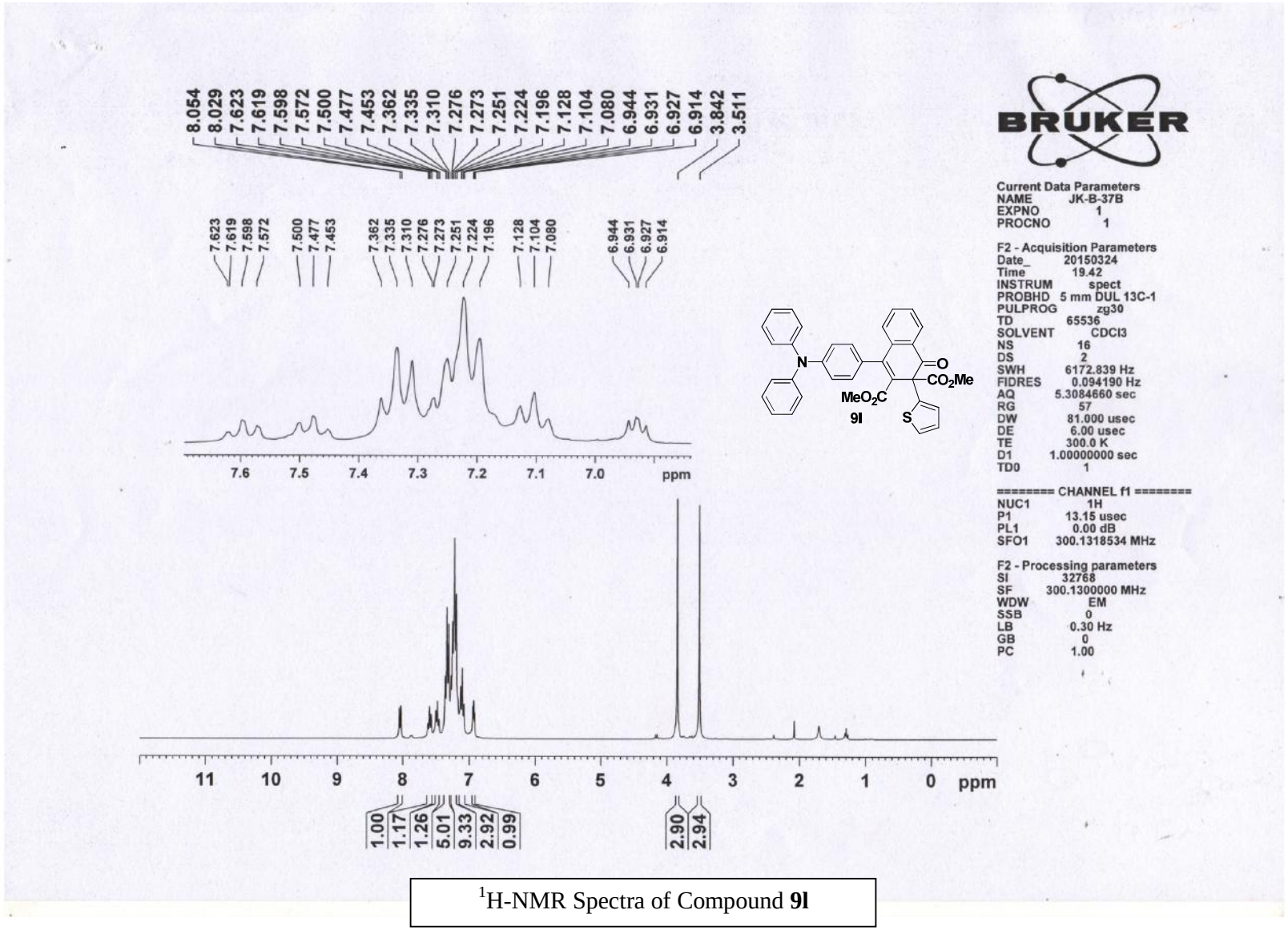
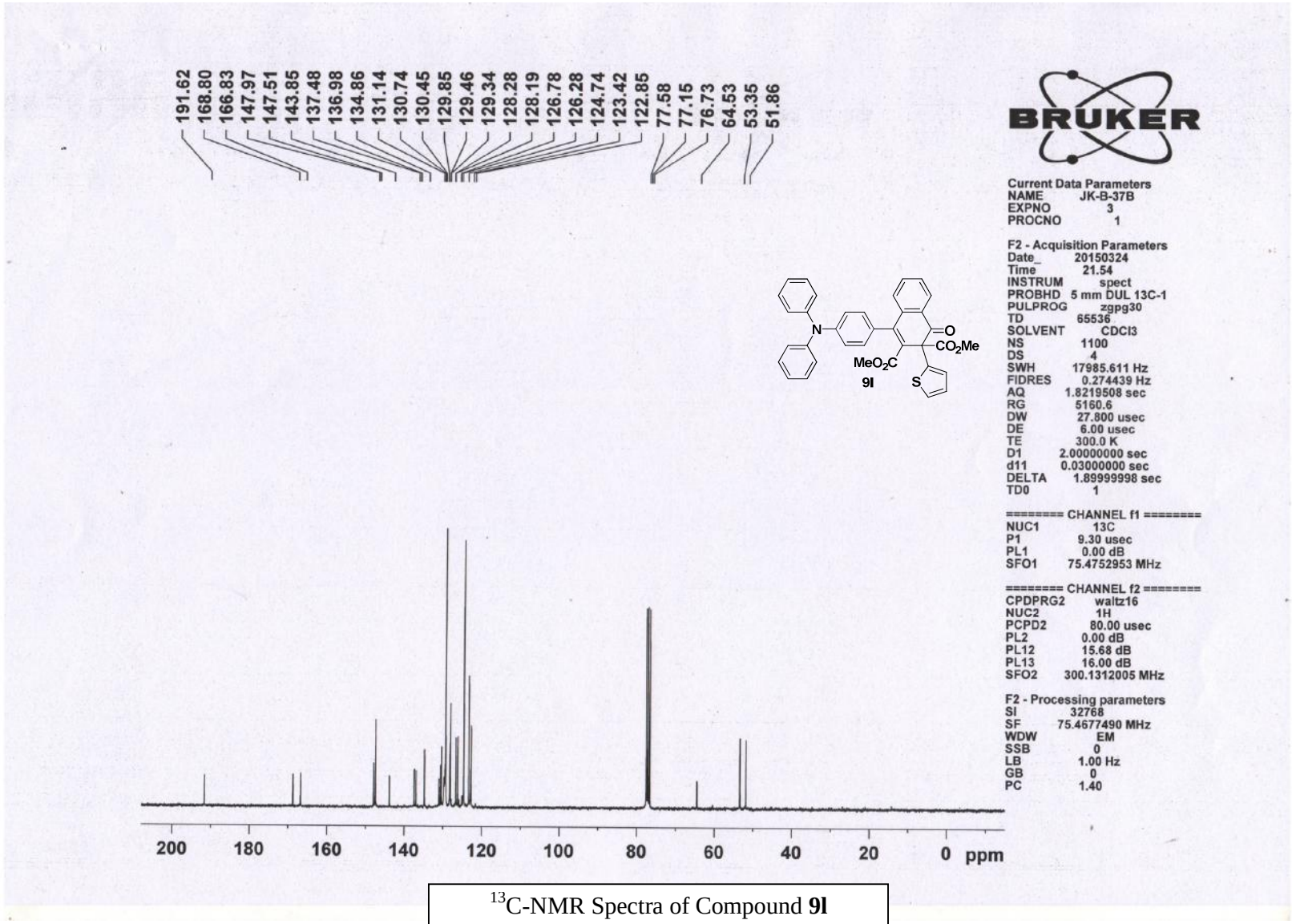


DEPT-135 NMR Spectra of Compound **9k**



^{1}H-NMR Spectra of Compound **9l**



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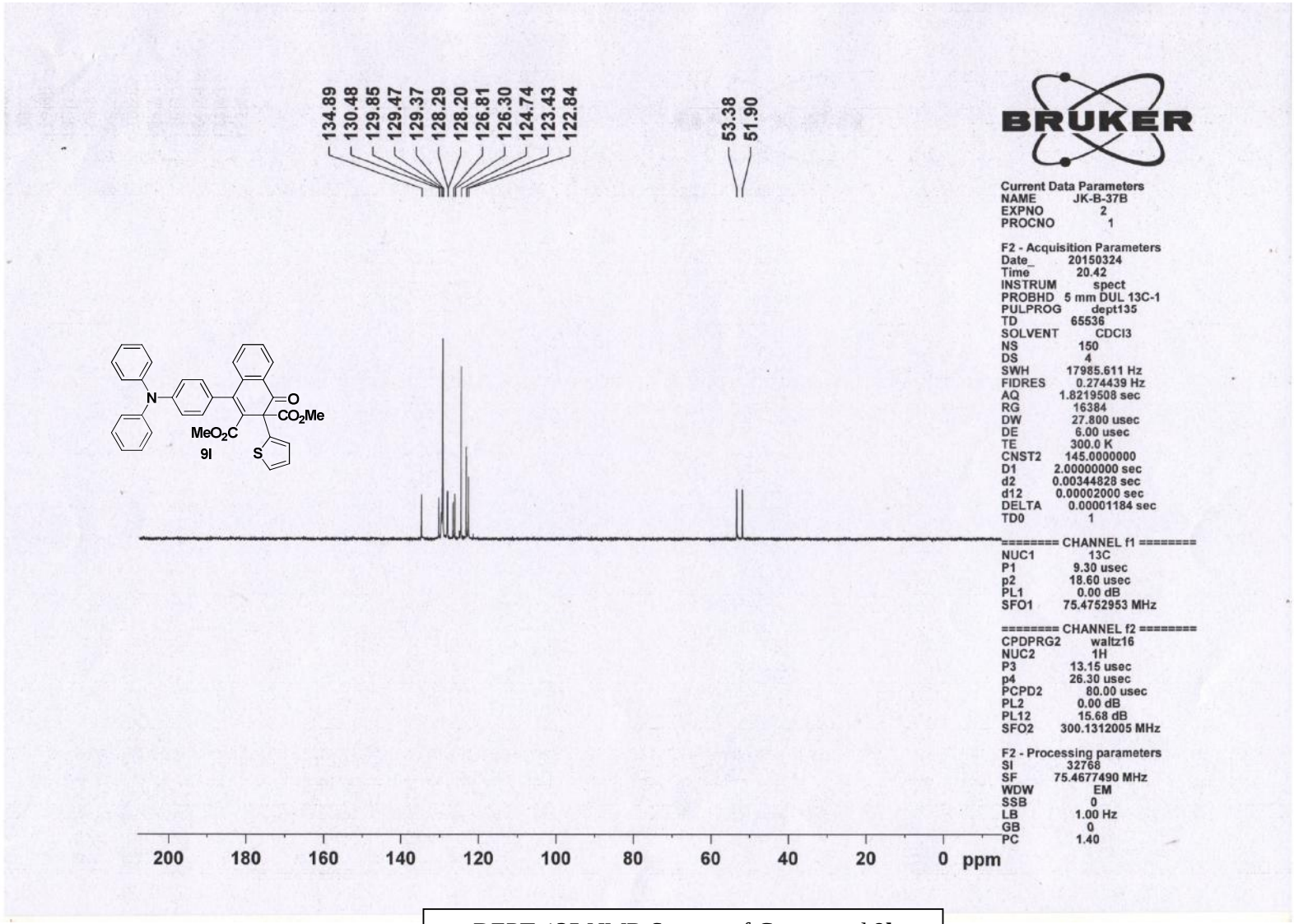
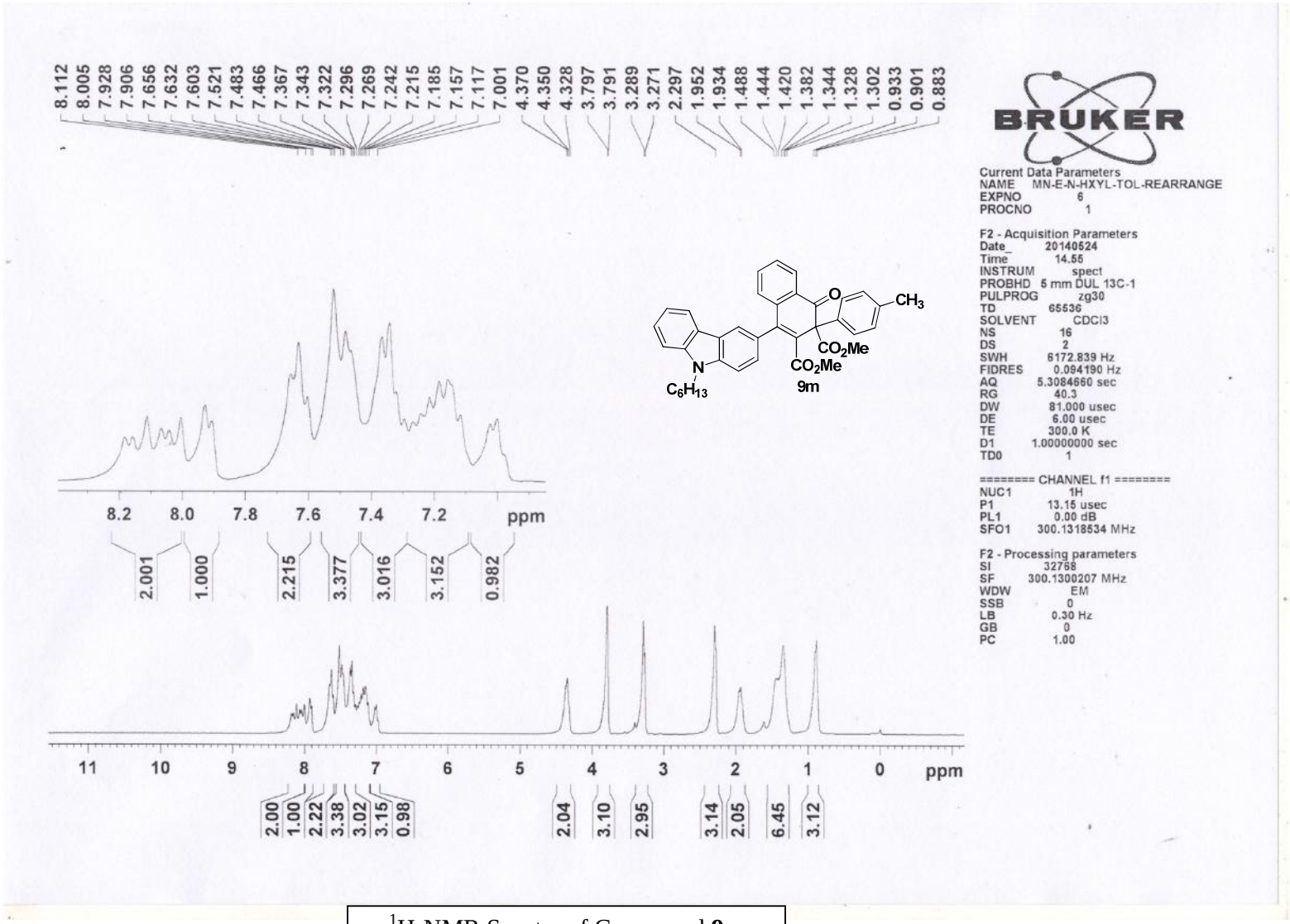


DEPT-135 NMR Spectra of Compound **9l**



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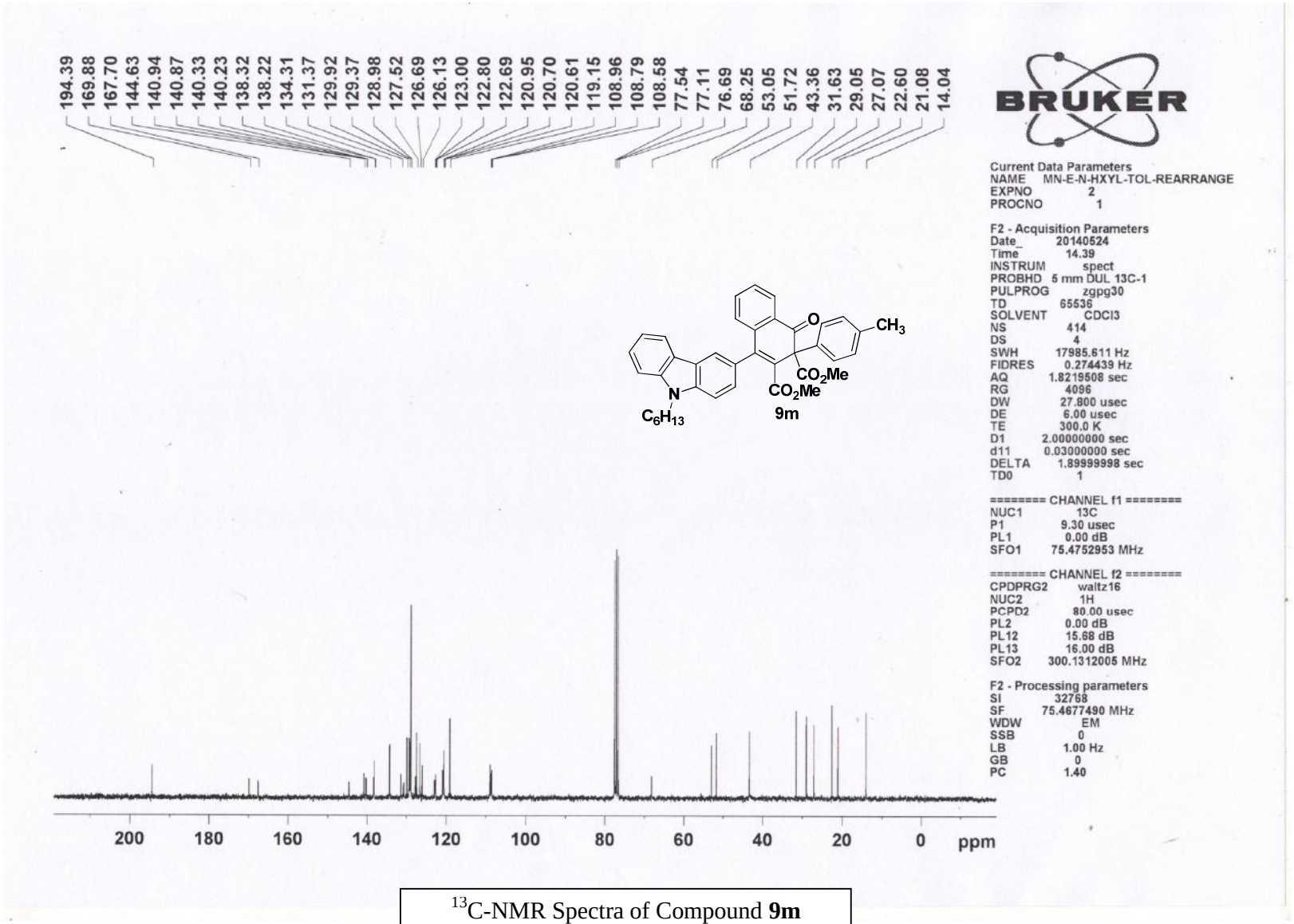
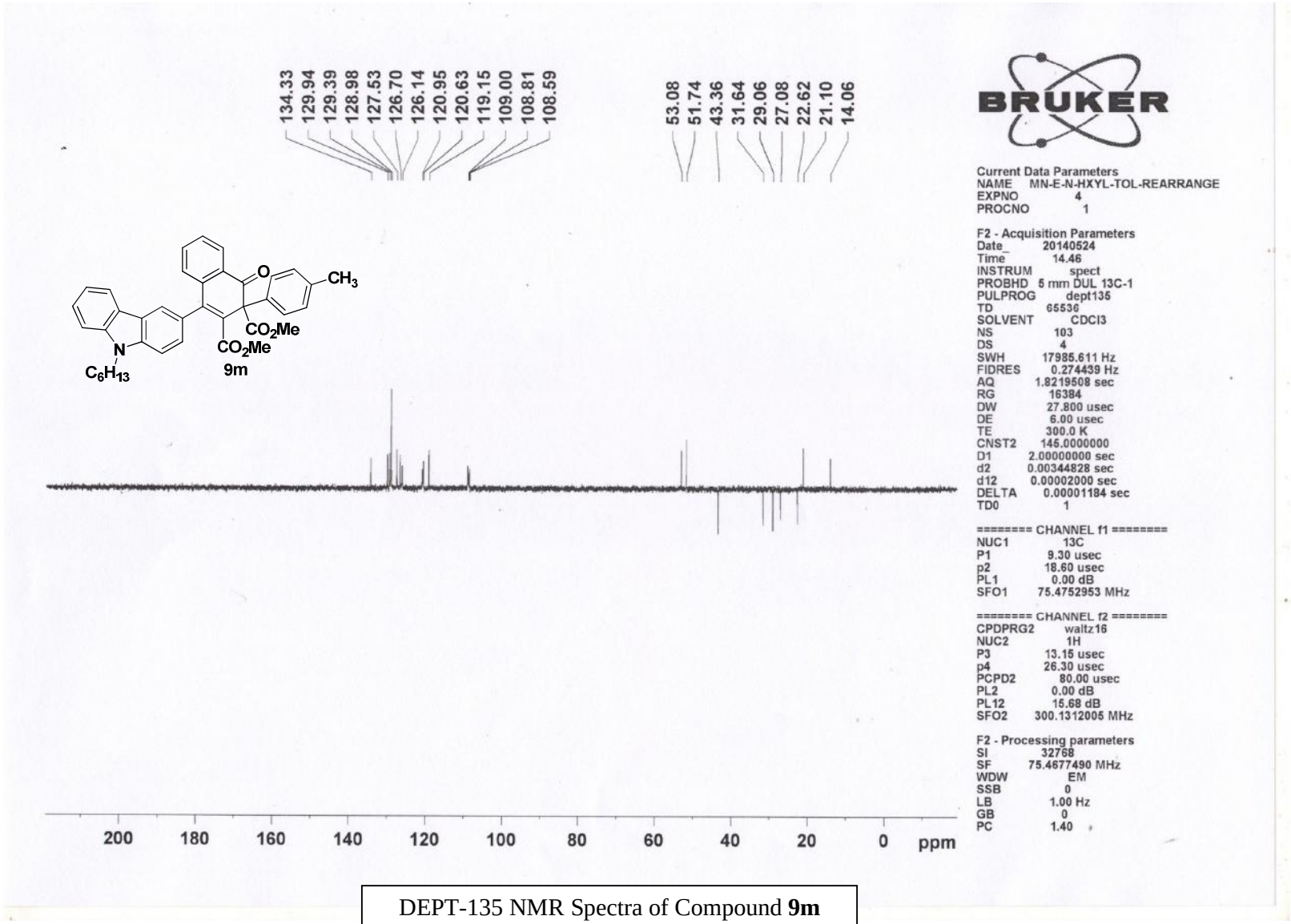


^{13}C-NMR Spectra of Compound **9m**



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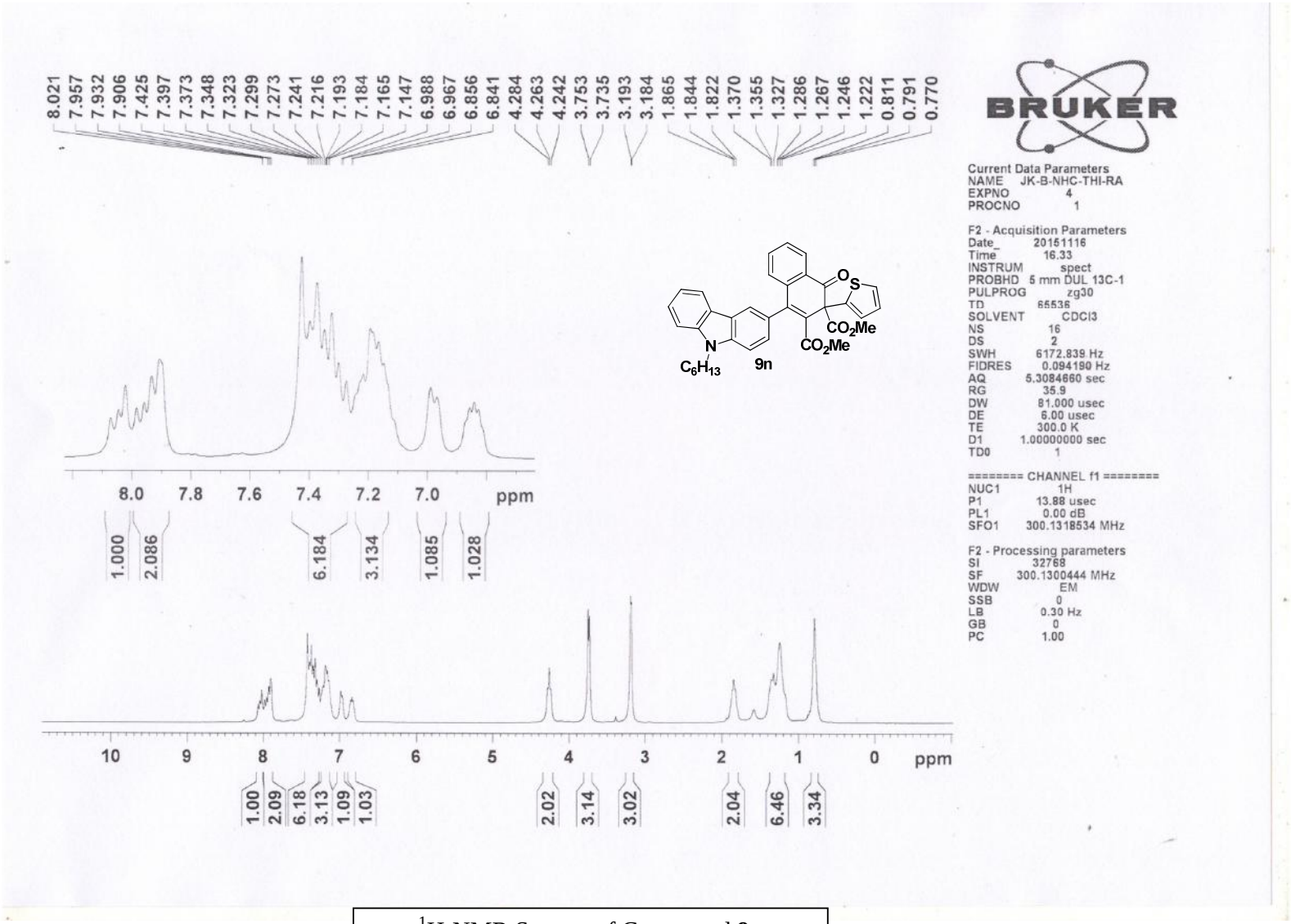
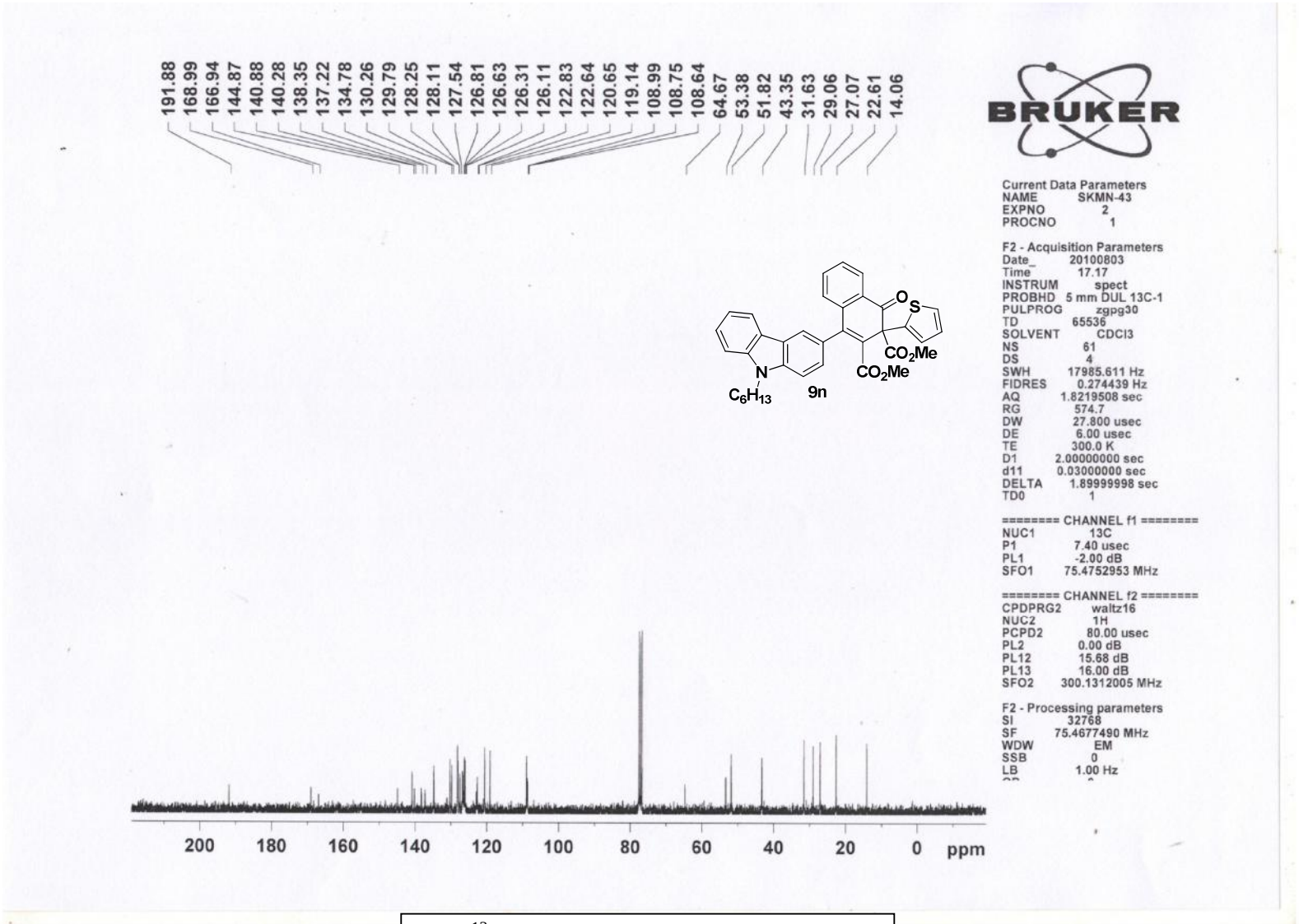
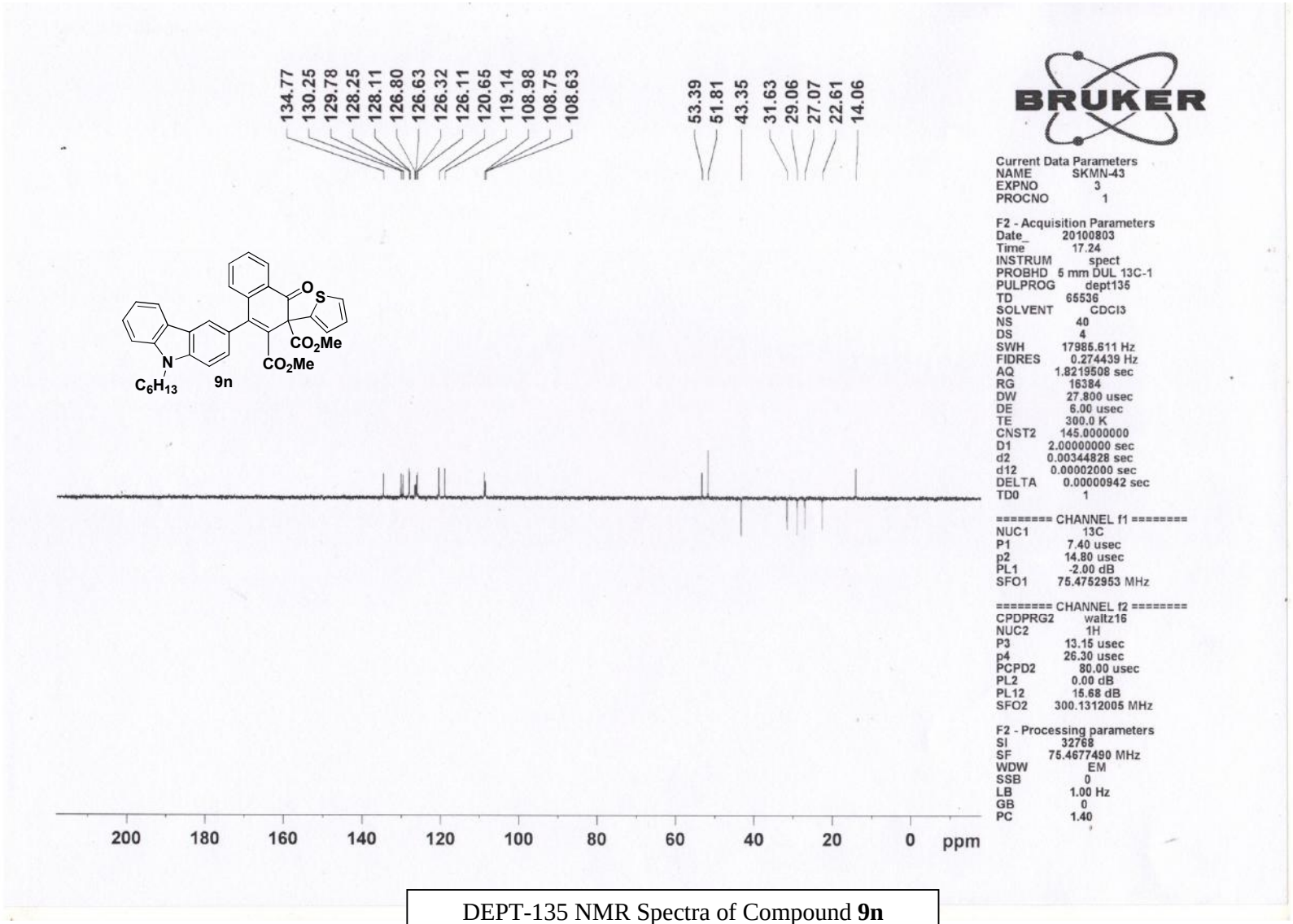


^{1}H-NMR Spectra of Compound **9n**



^{13}C-NMR Spectra of Compound **9n**



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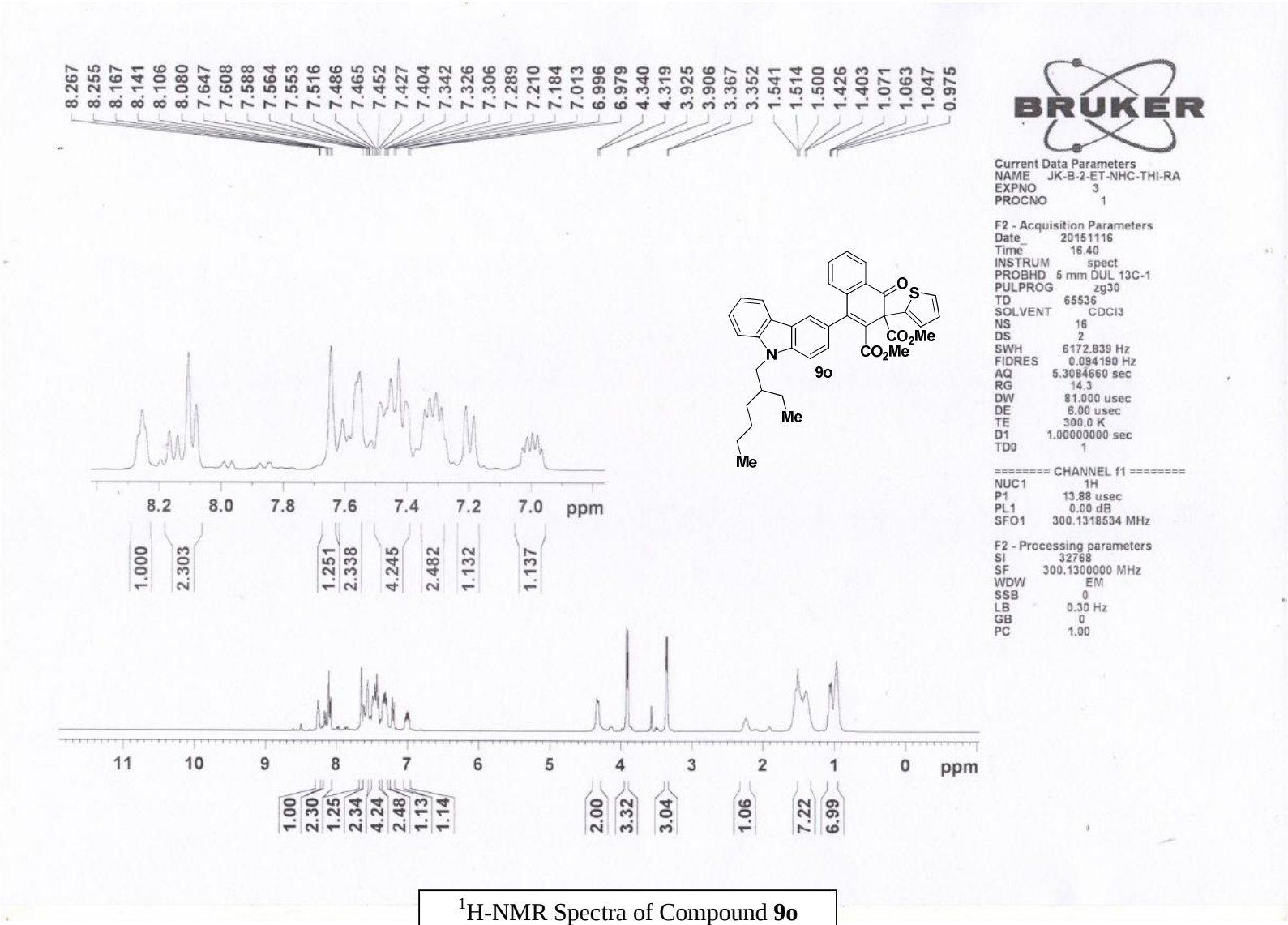
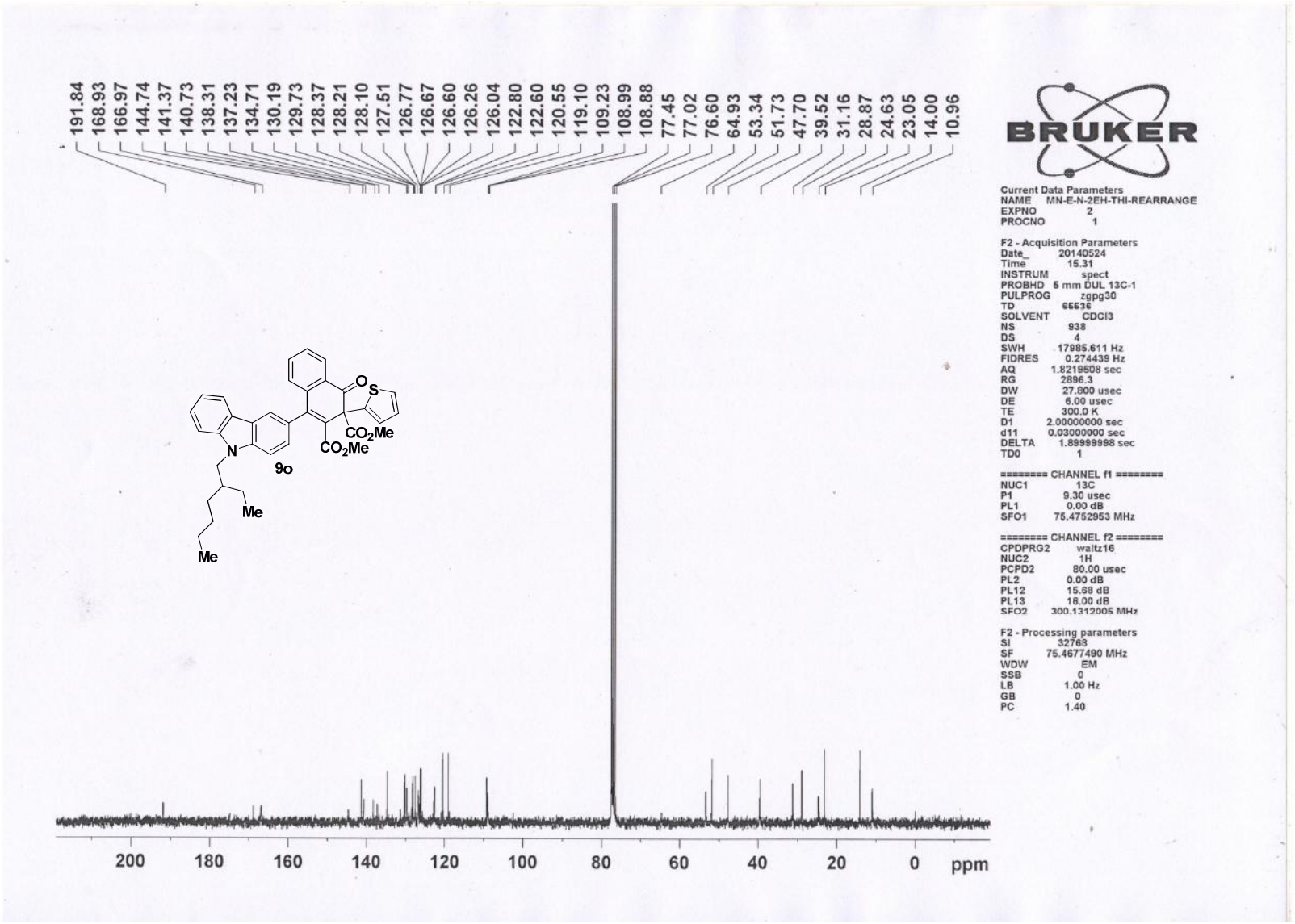
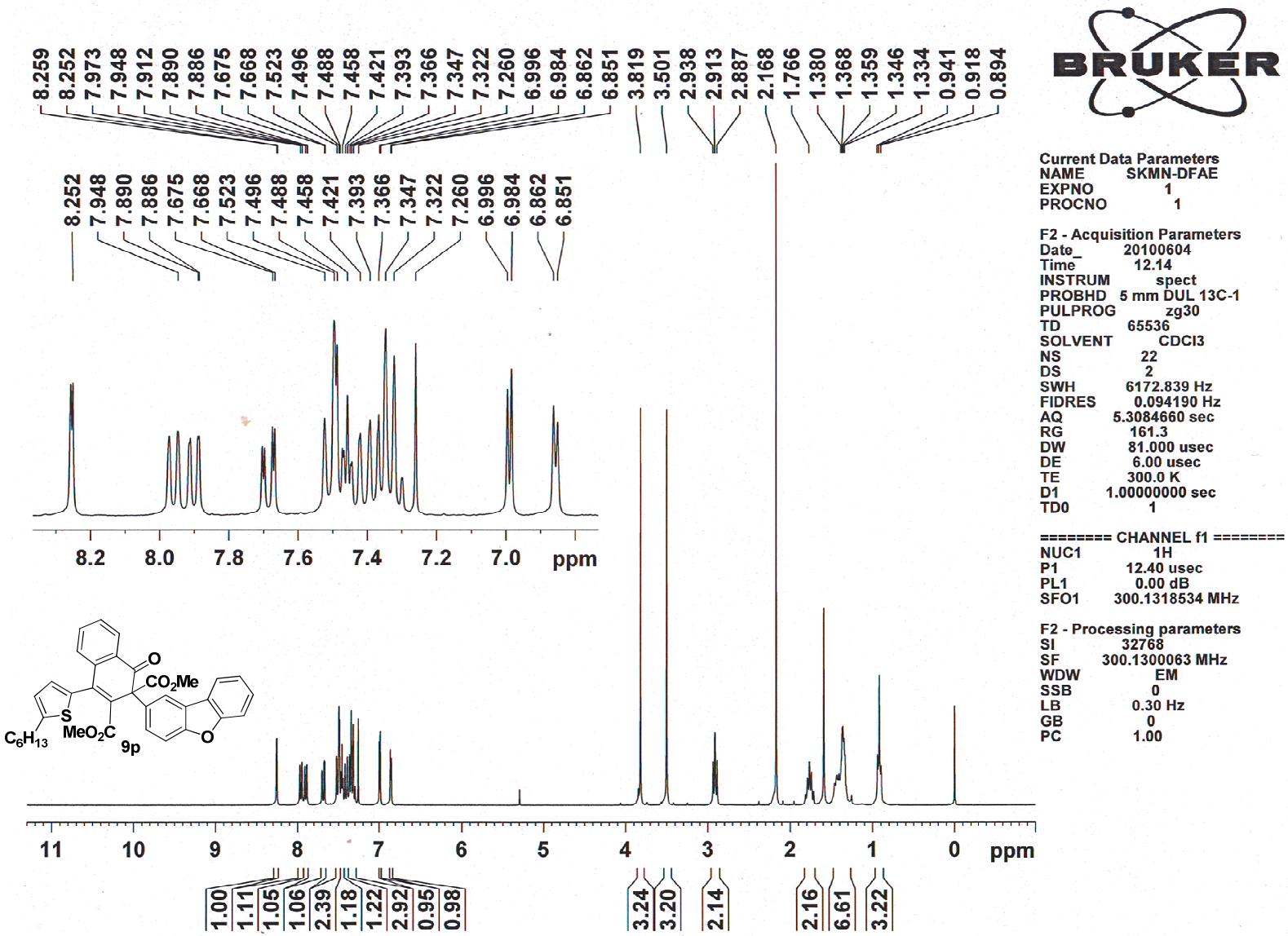


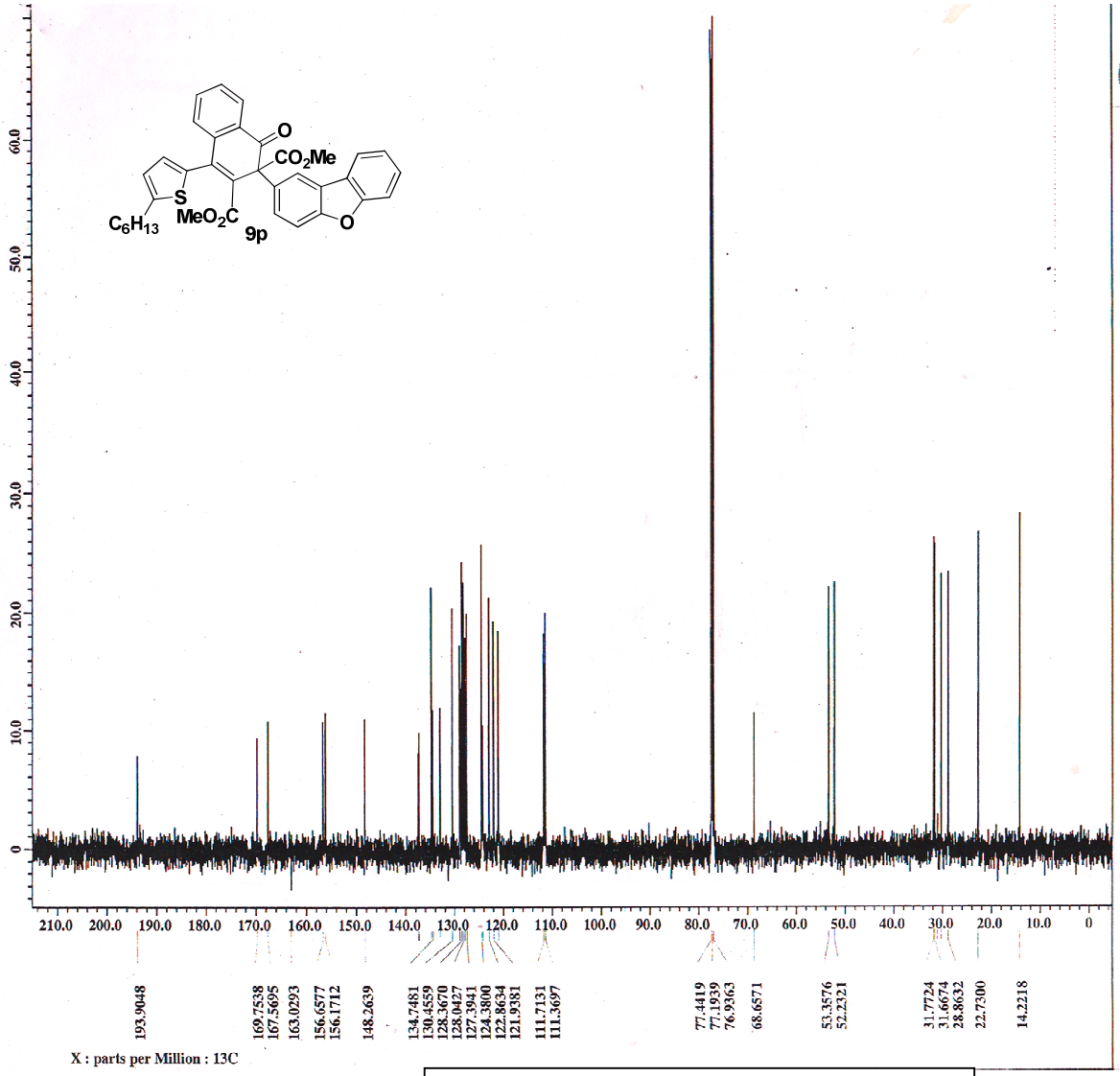
^{1}H-NMR Spectra of Compound **9o**



^{13}C-NMR Spectra of Compound **9o**



^{1}H-NMR Spectra of Compound **9p**



JEOL

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Experiment          = single_pulse_dec
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Solvent             = CHLOROFORM-D
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Revision_time       = 11-JUN-2010 16:21:53
Current_time        = 11-JUN-2010 16:22:00

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Dim_title           = 13C
Dim_units           = [ppm]
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Site                = ECA500
Spectrometer        = DELTA2_NMR

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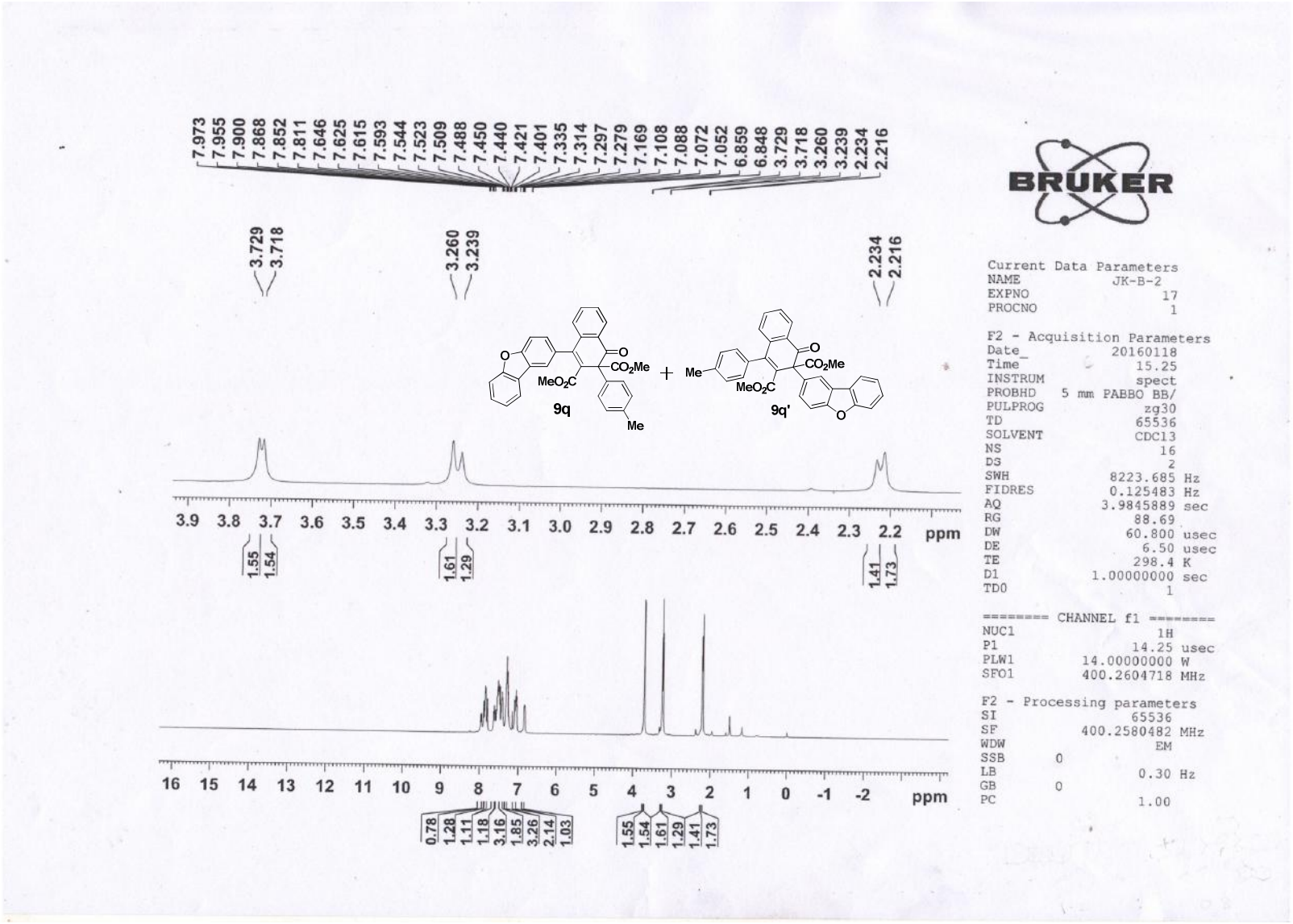
^{13}C-NMR Spectra of Compound **9p**



BRUKER

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PROCNO                     1

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PULPROG                 zg30
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DS                         2
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DE                      6.50 usec
TE                     298.4 K
D1                  1.00000000 sec
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PLW1           14.00000000 W
SFO1           400.2604718 MHz

F2 - Processing parameters
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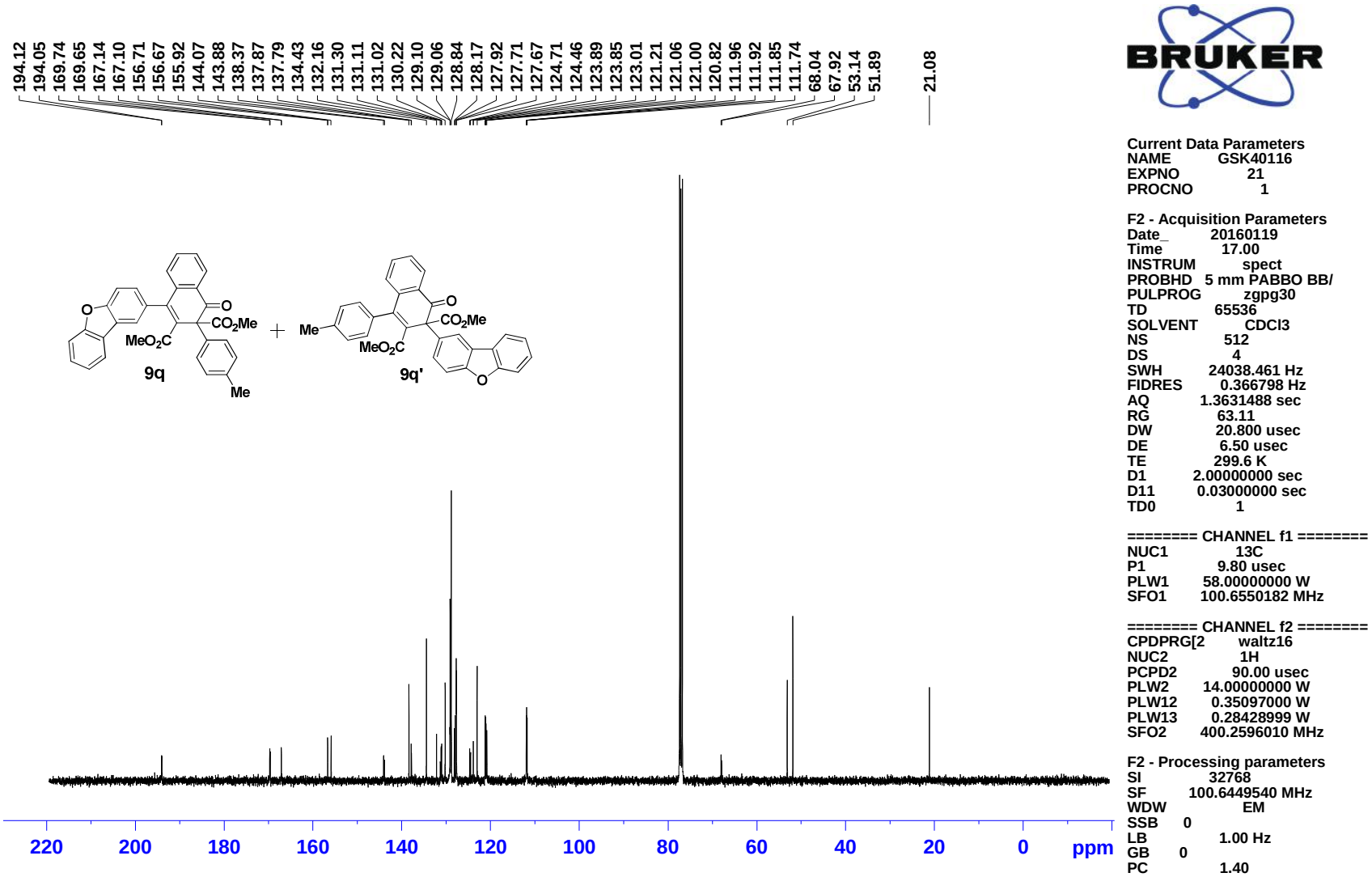
^{1}H-NMR Spectra of Compound **9q & 9q'**



BRUKER

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EXPNO 21
PROCNO 1

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SOLVENT CDCl3
NS 512
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FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 63.11
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DE 6.50 usec
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D11 0.03000000 sec
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F2 - Processing parameters
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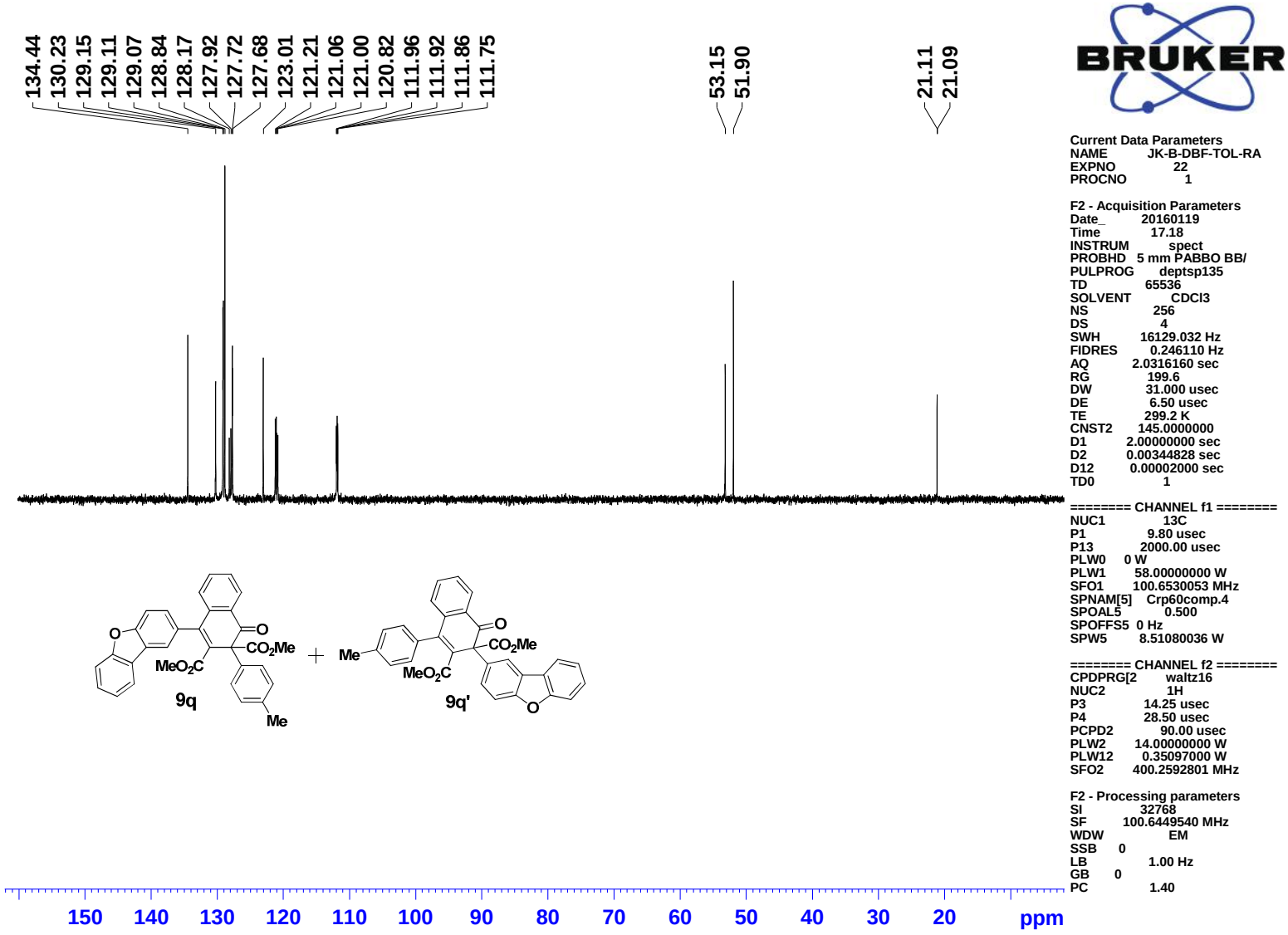
^{13}C-NMR Spectra of Compound **9q & 9q'**



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EXPNO 22
PROCNO 1

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PULPROG deptsp135
TD 65536
SOLVENT CDCl3
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FIDRES 0.246110 Hz
AQ 2.0316160 sec
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DE 6.50 usec
TE 299.2 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
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P13 2000.00 usec
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PLW1 58.00000000 W
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SPOFFS5 0 Hz
SPW5 8.51080036 W

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PLW2 14.00000000 W
PLW12 0.35097000 W
SFO2 400.2592801 MHz

F2 - Processing parameters
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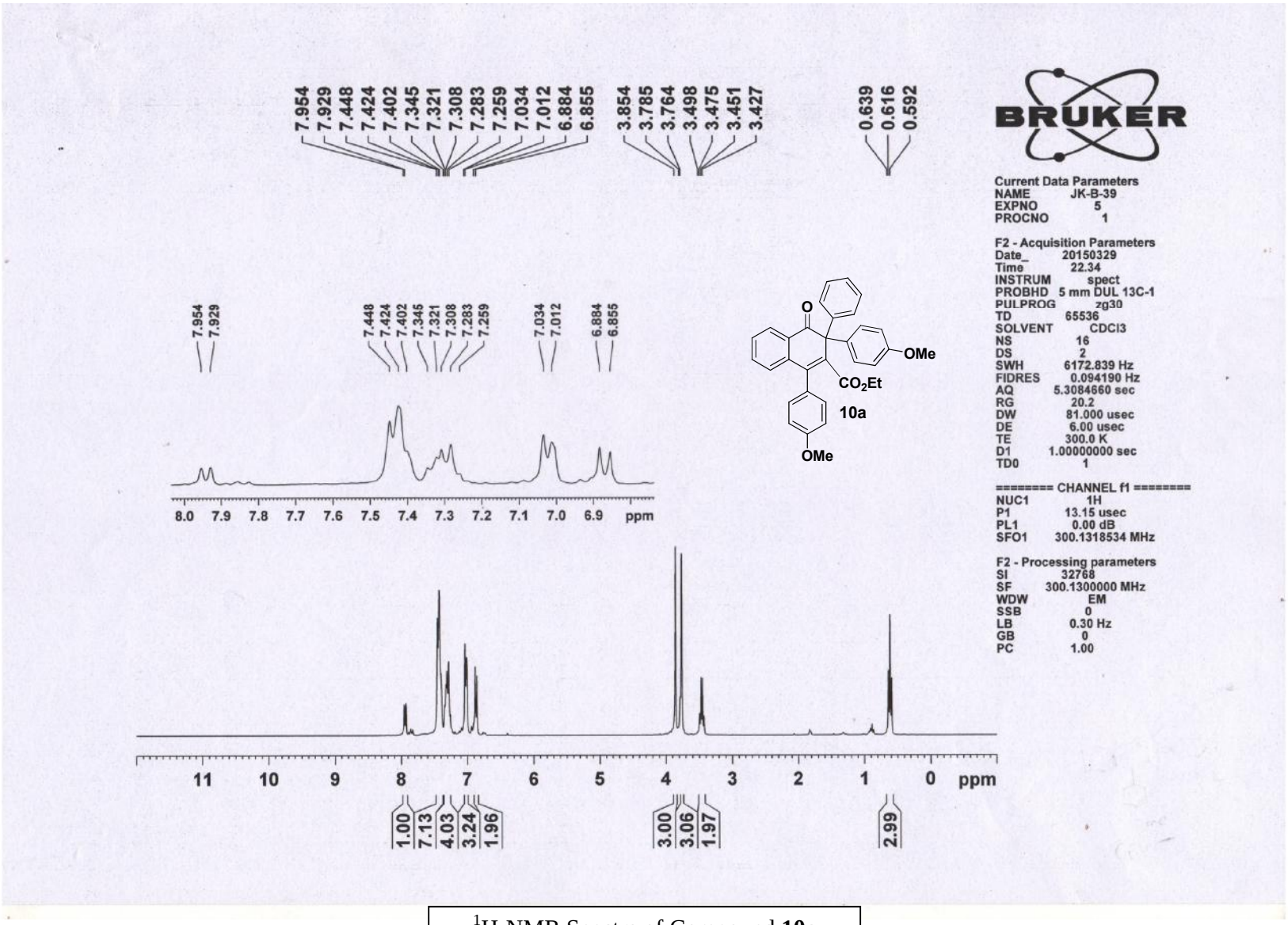
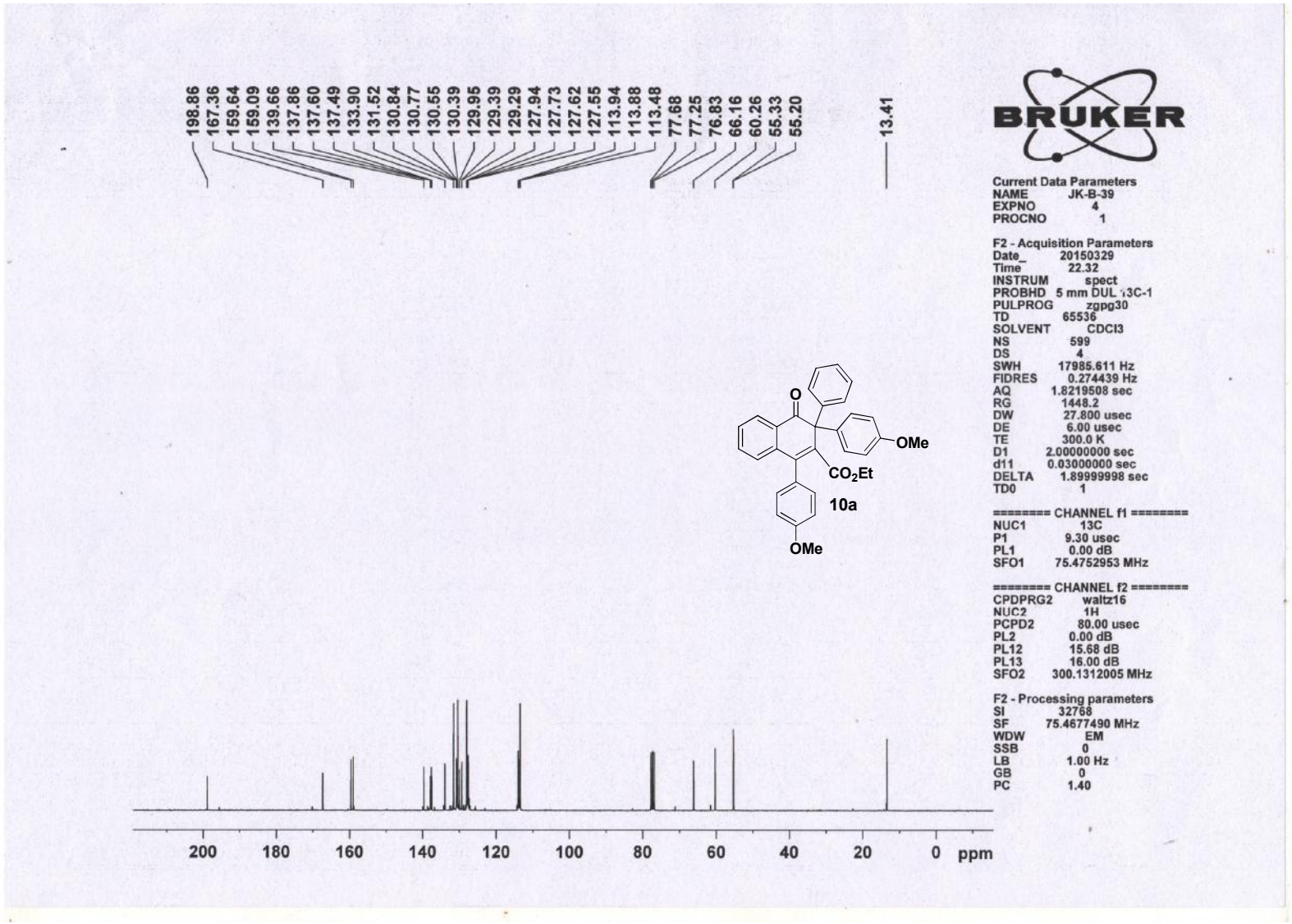
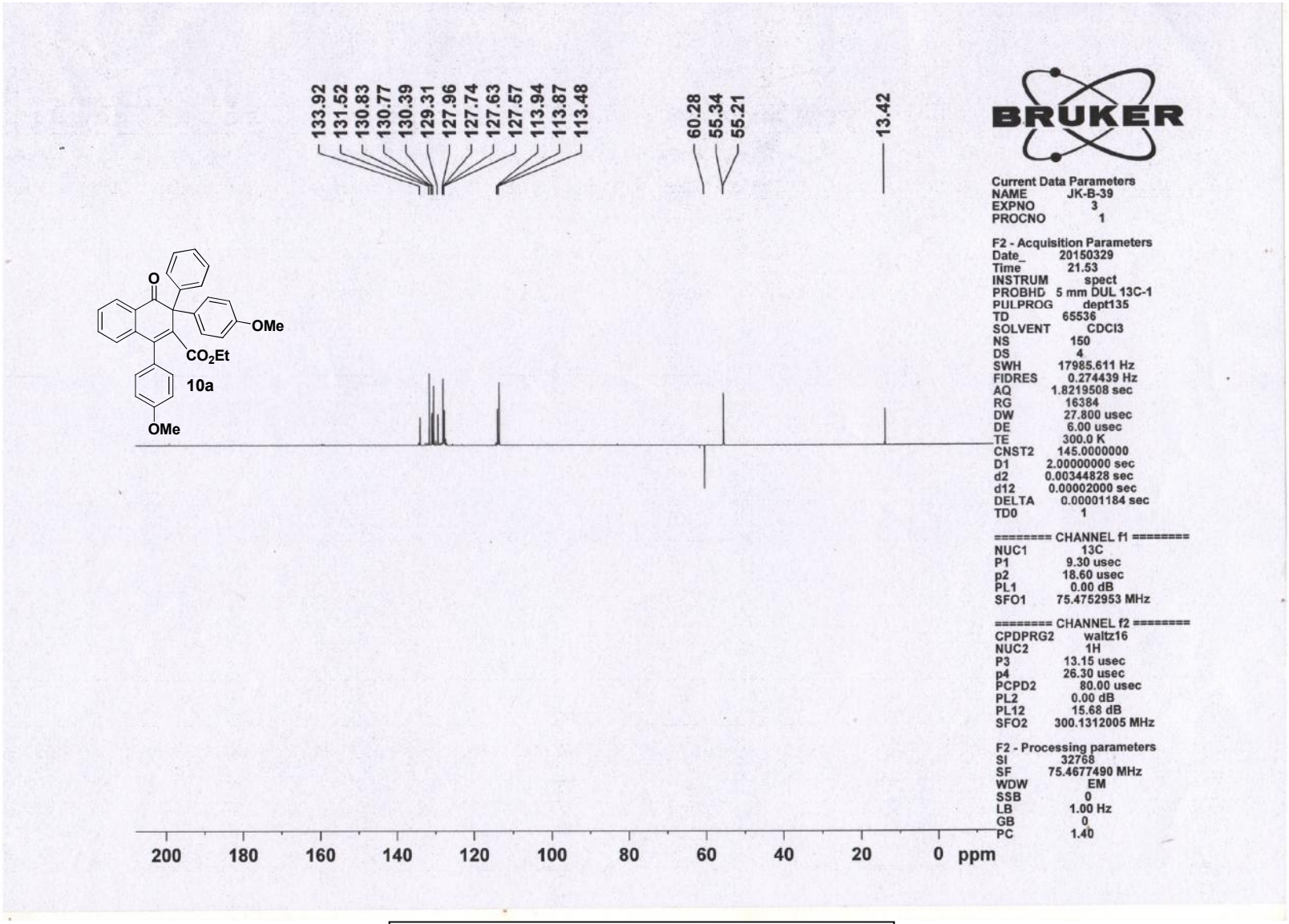
DEPT-135 NMR Spectra of Compound **9q & 9q'**



^{1}H-NMR Spectra of Compound **10a**



^{13}C-NMR Spectra of Compound **10a**



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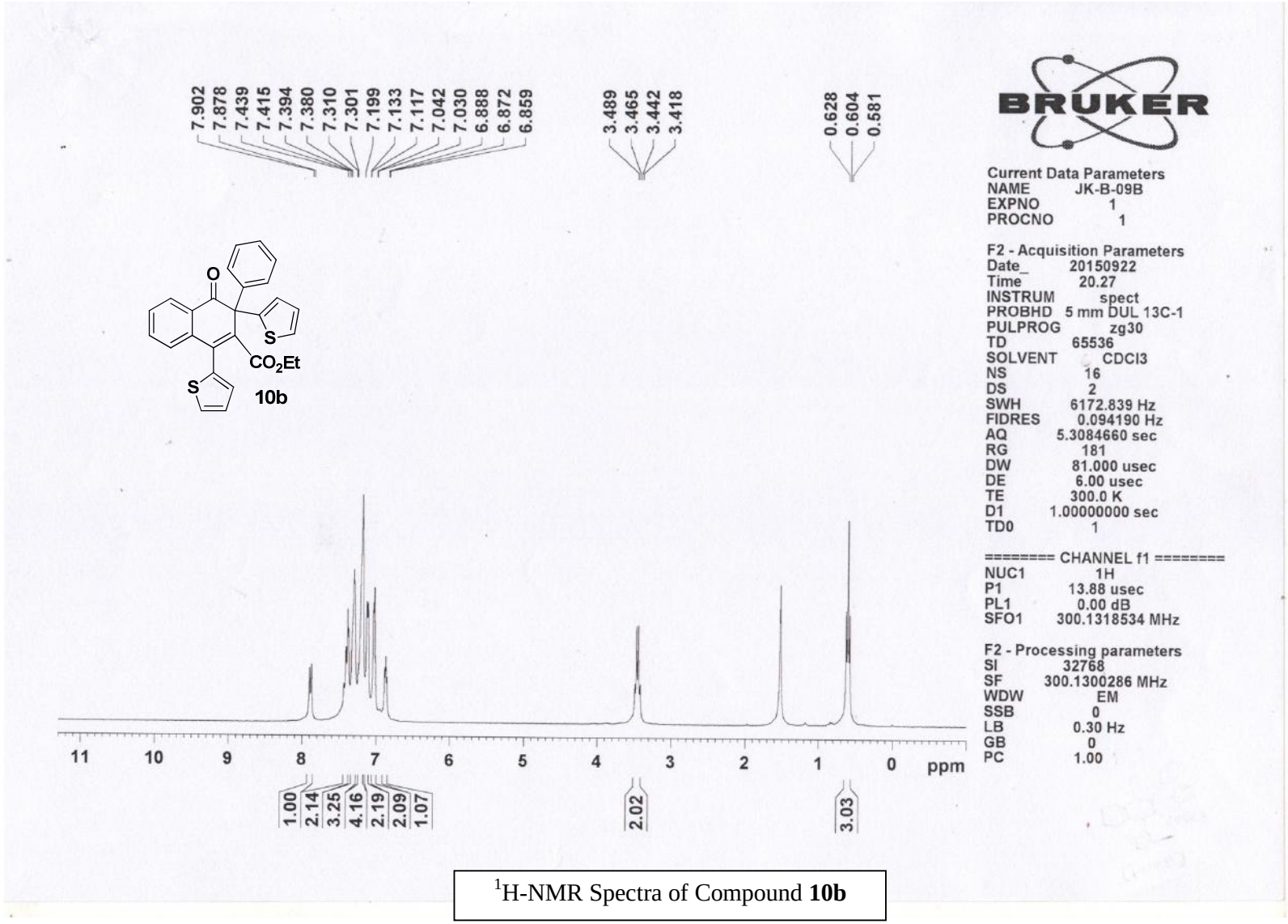

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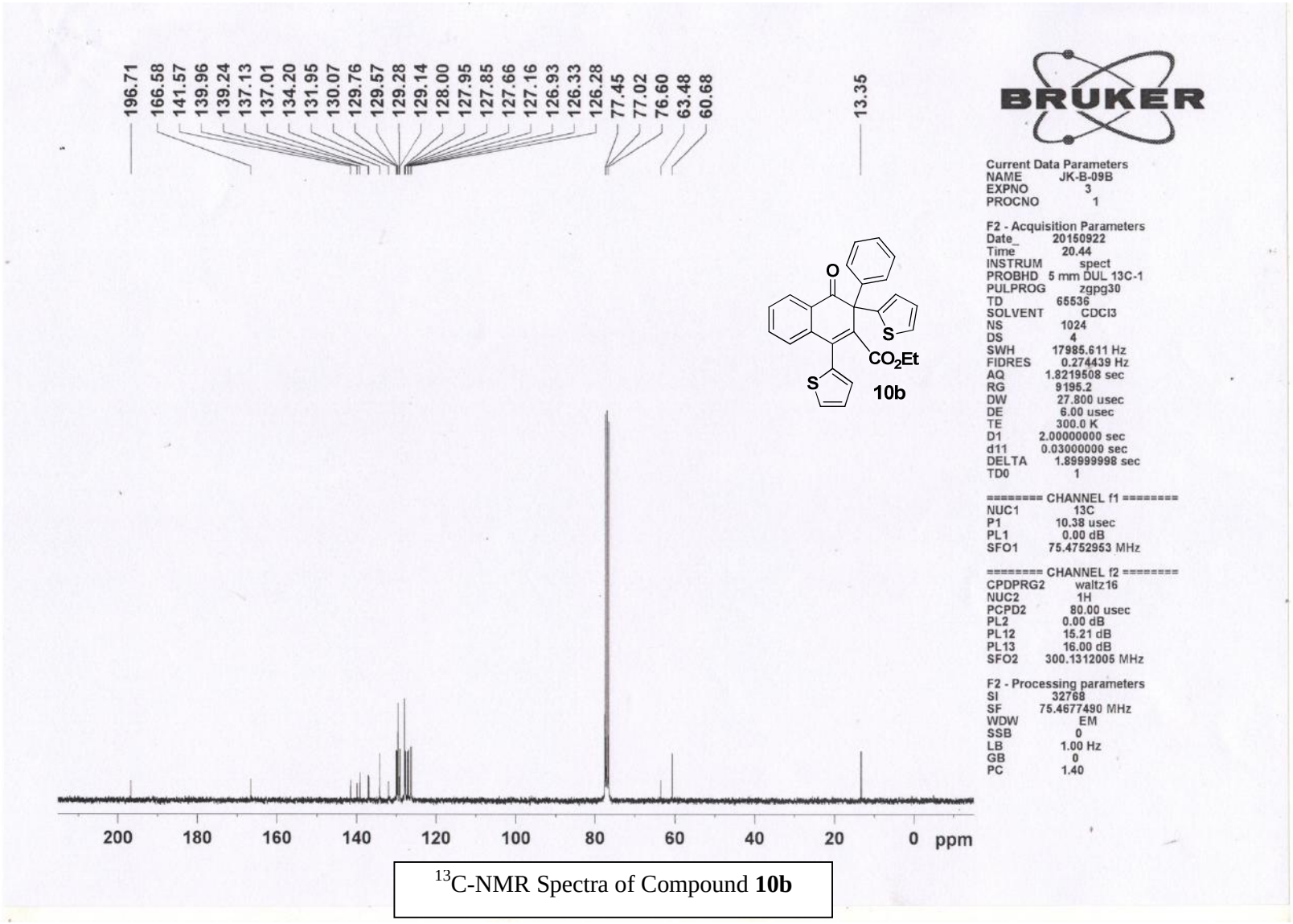
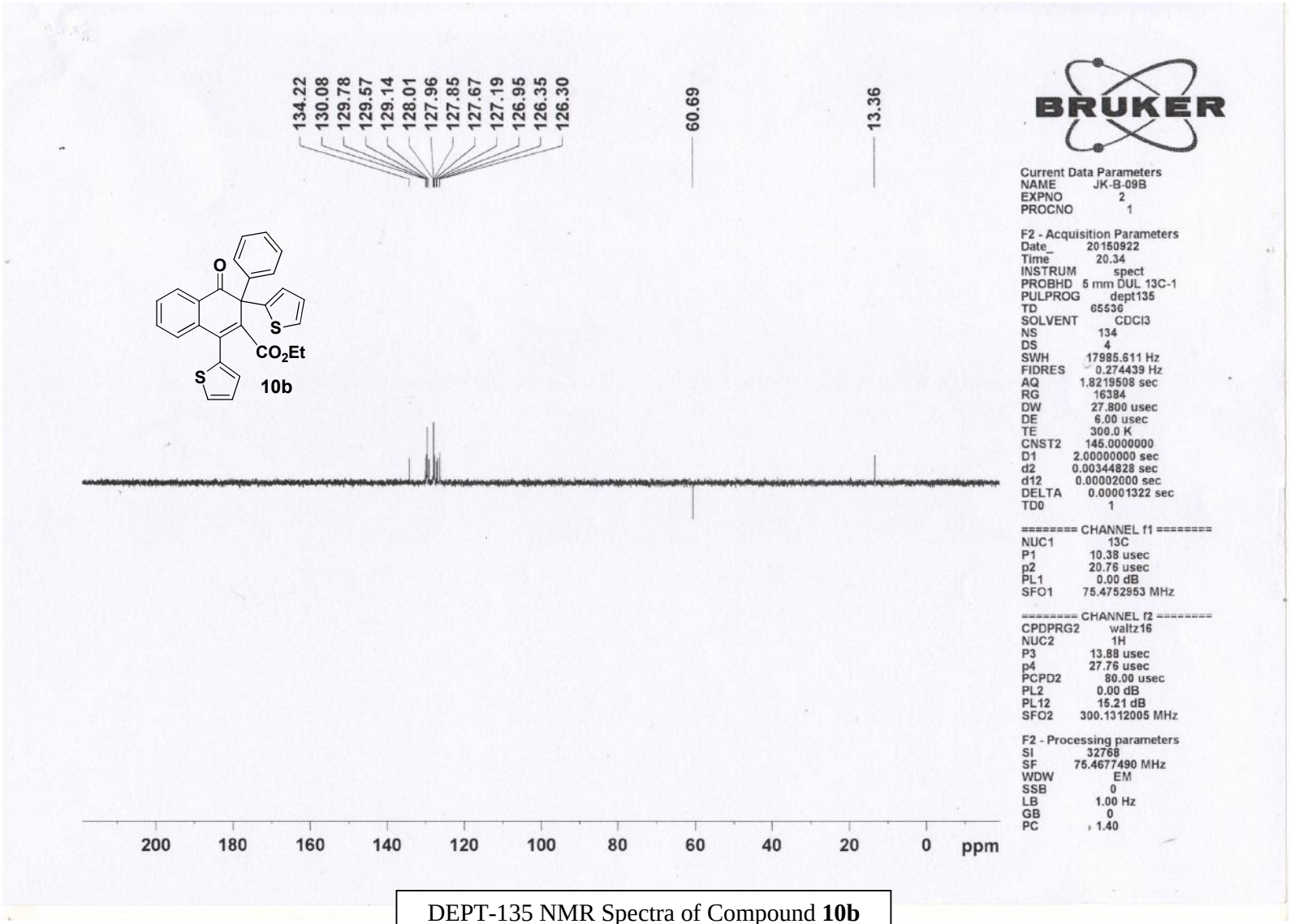
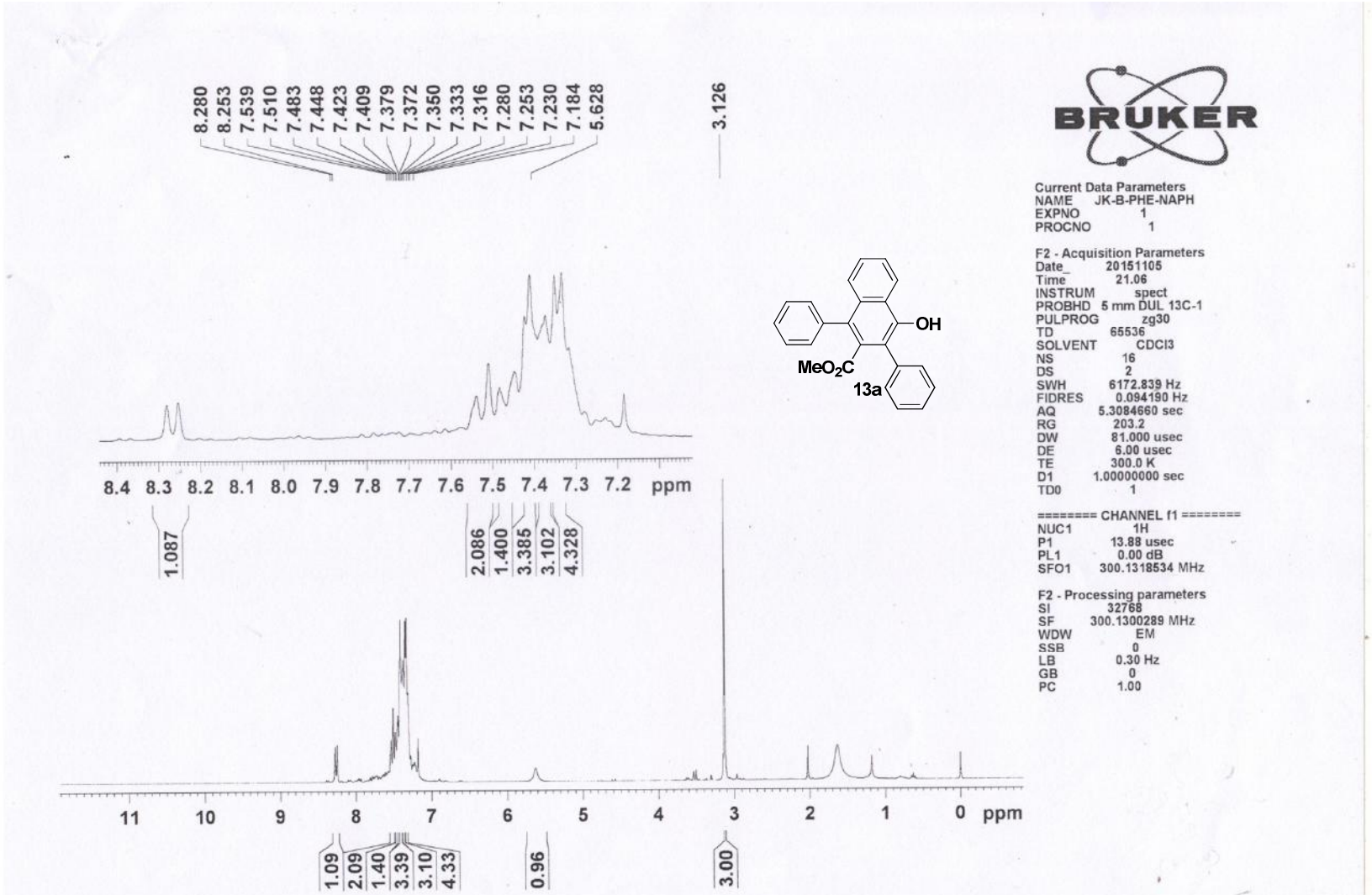


^{13}C-NMR Spectra of Compound **10b**



DEPT-135 NMR Spectra of Compound **10b**



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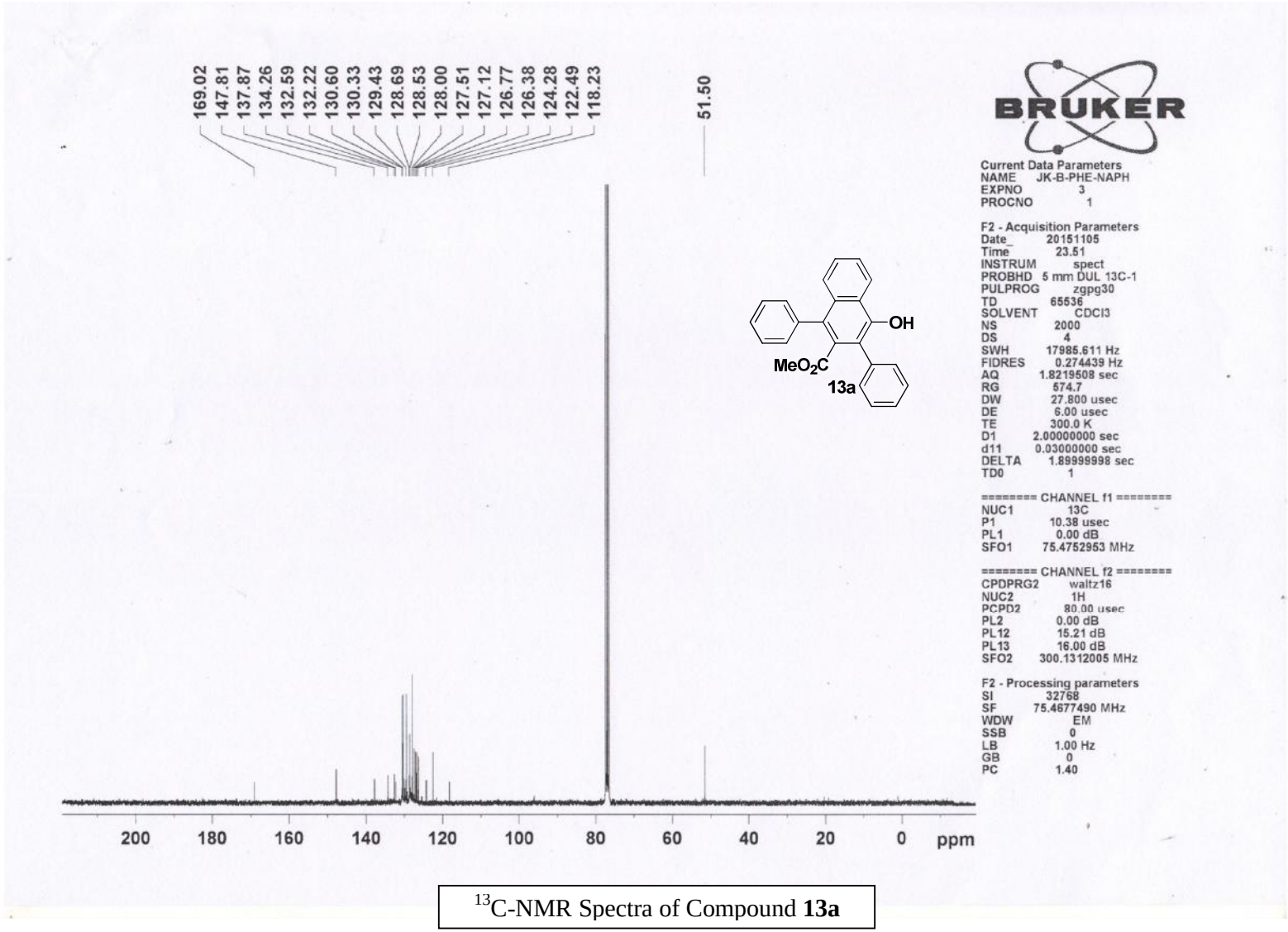

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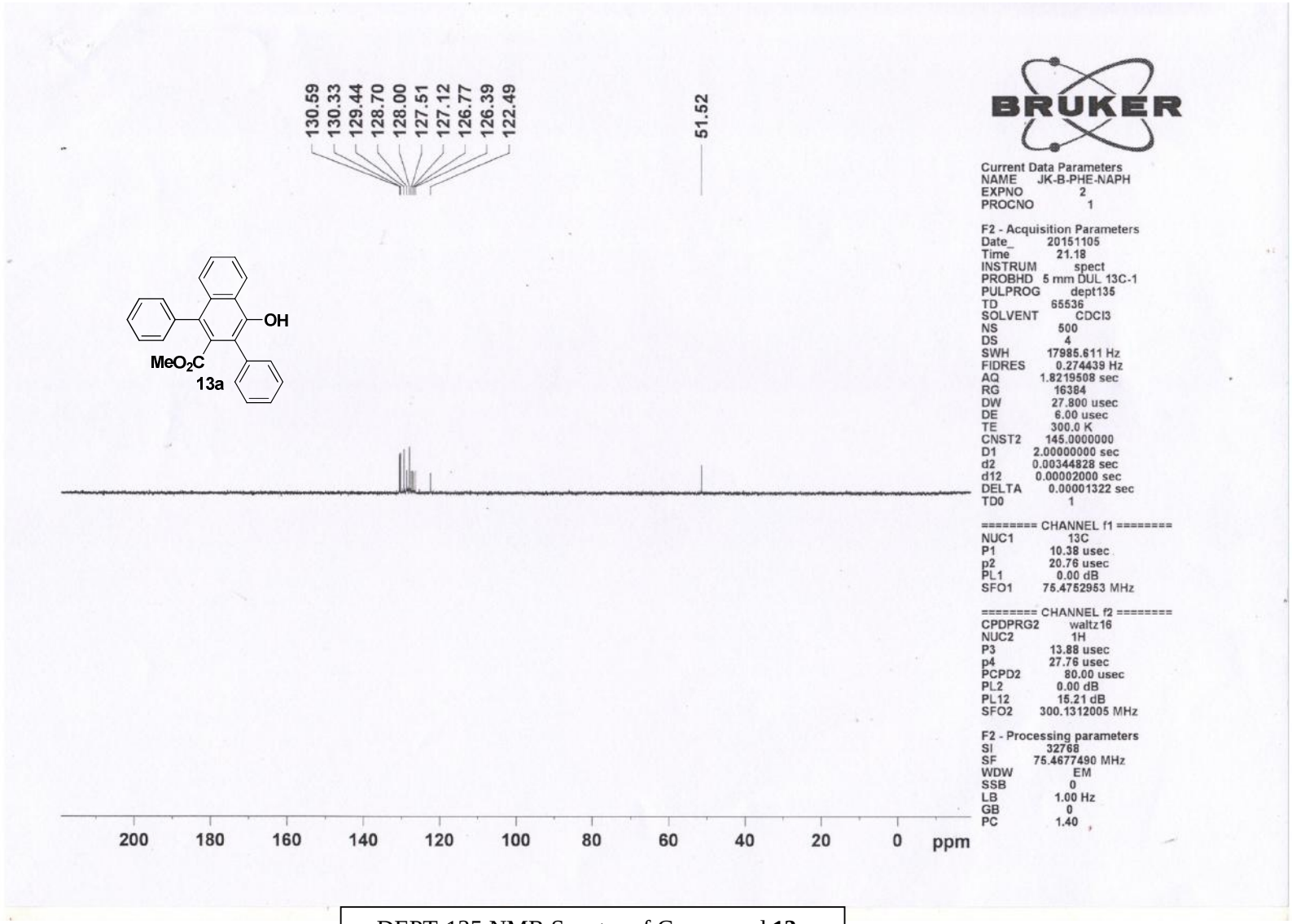

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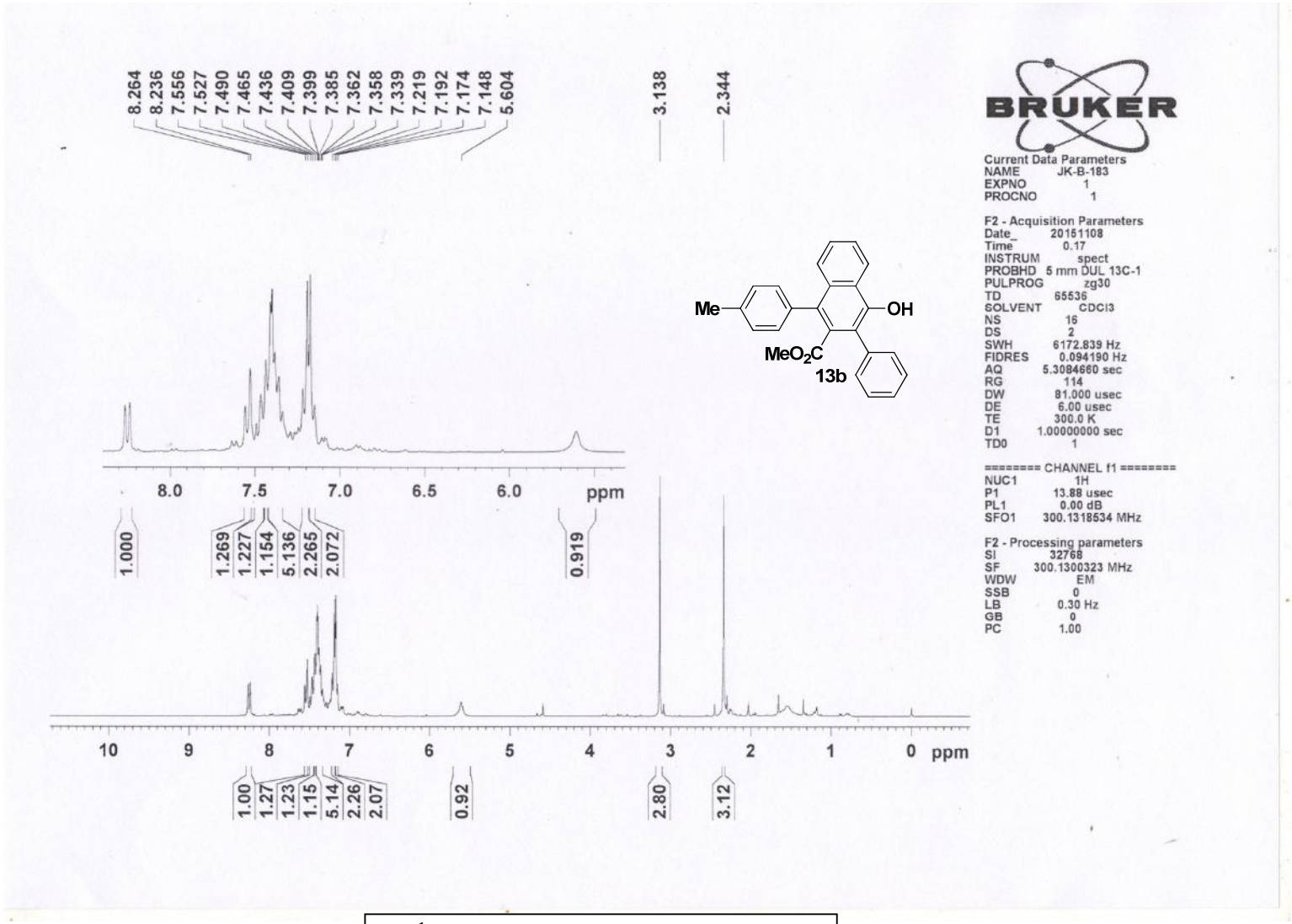

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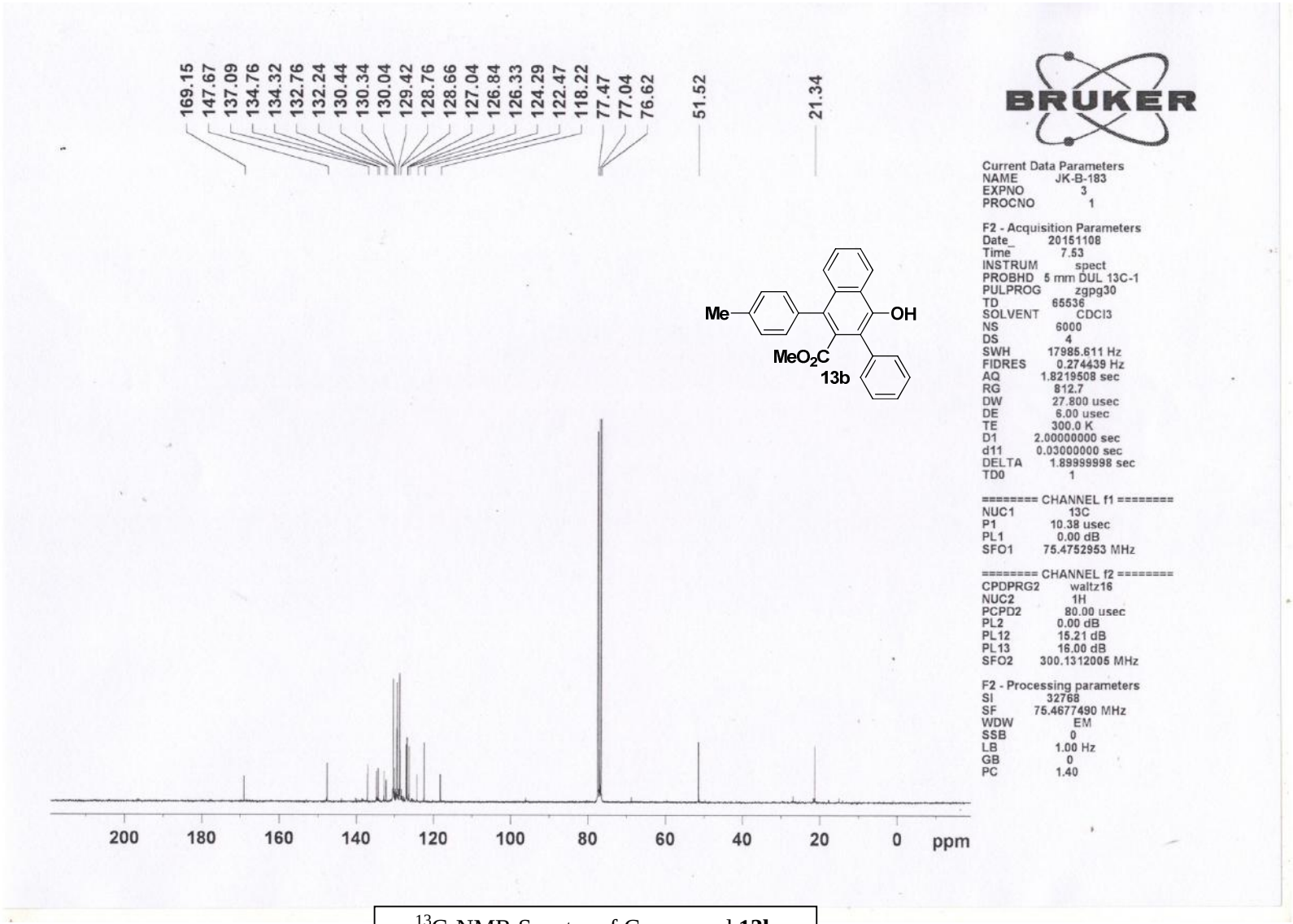

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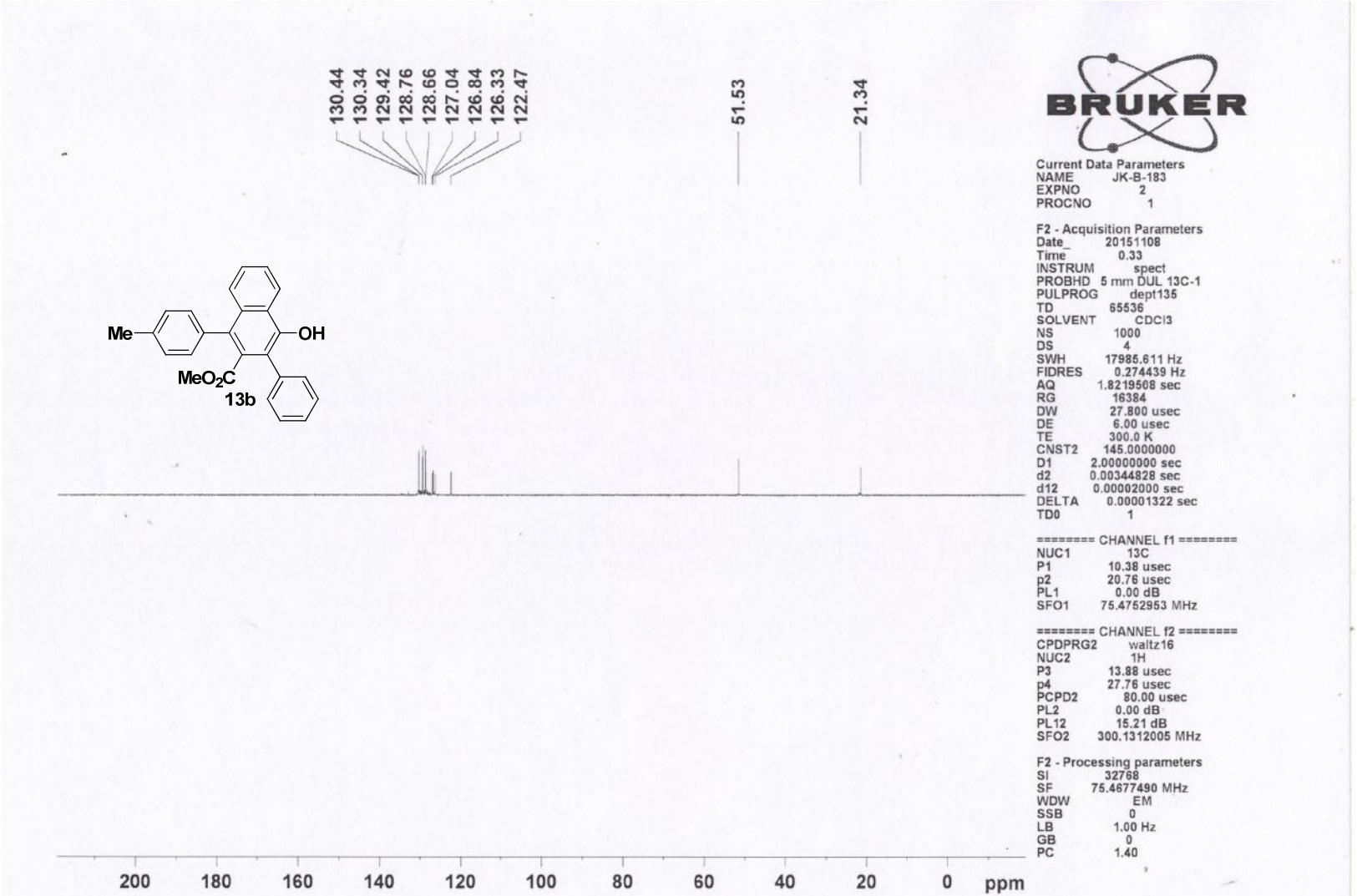

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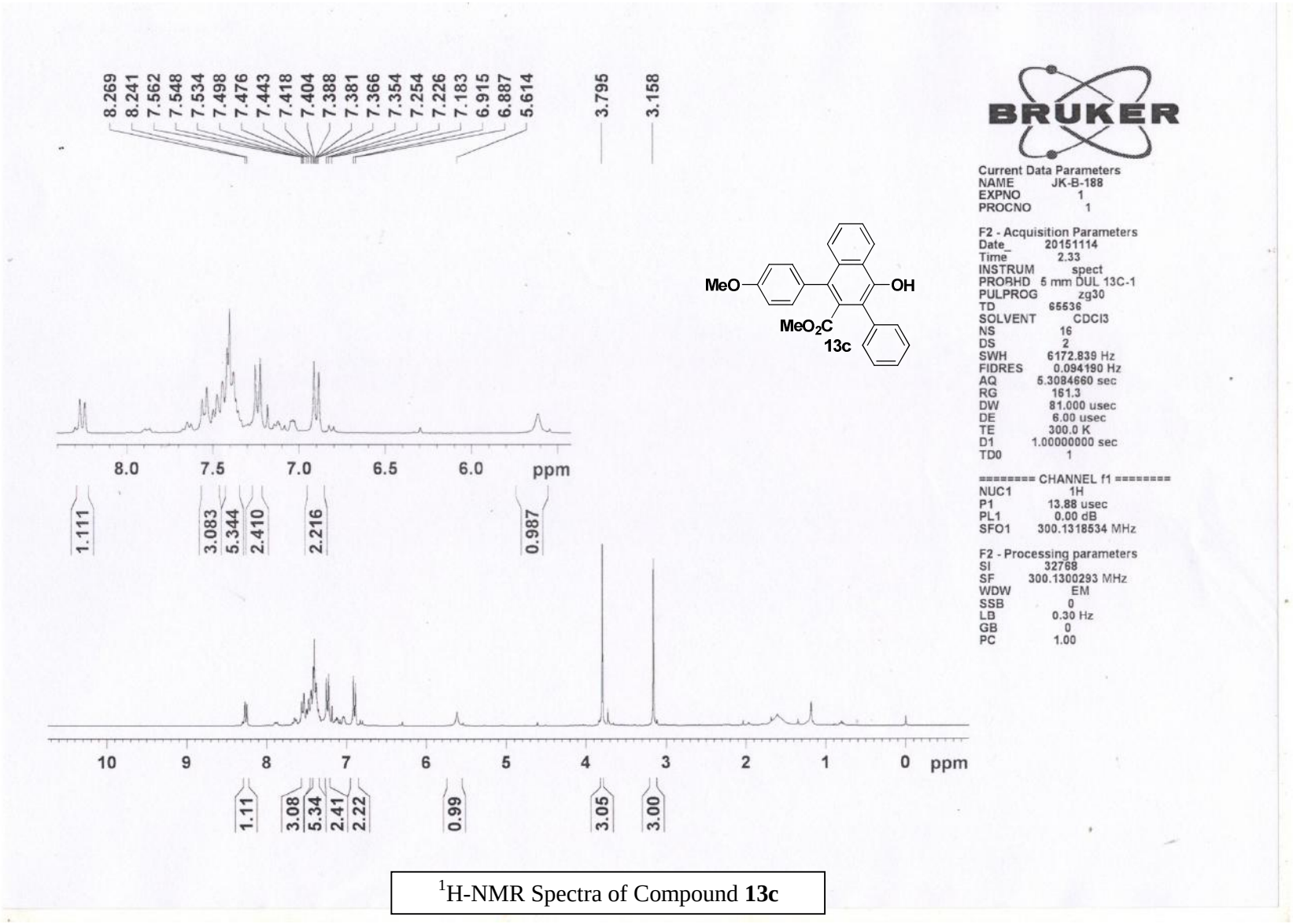

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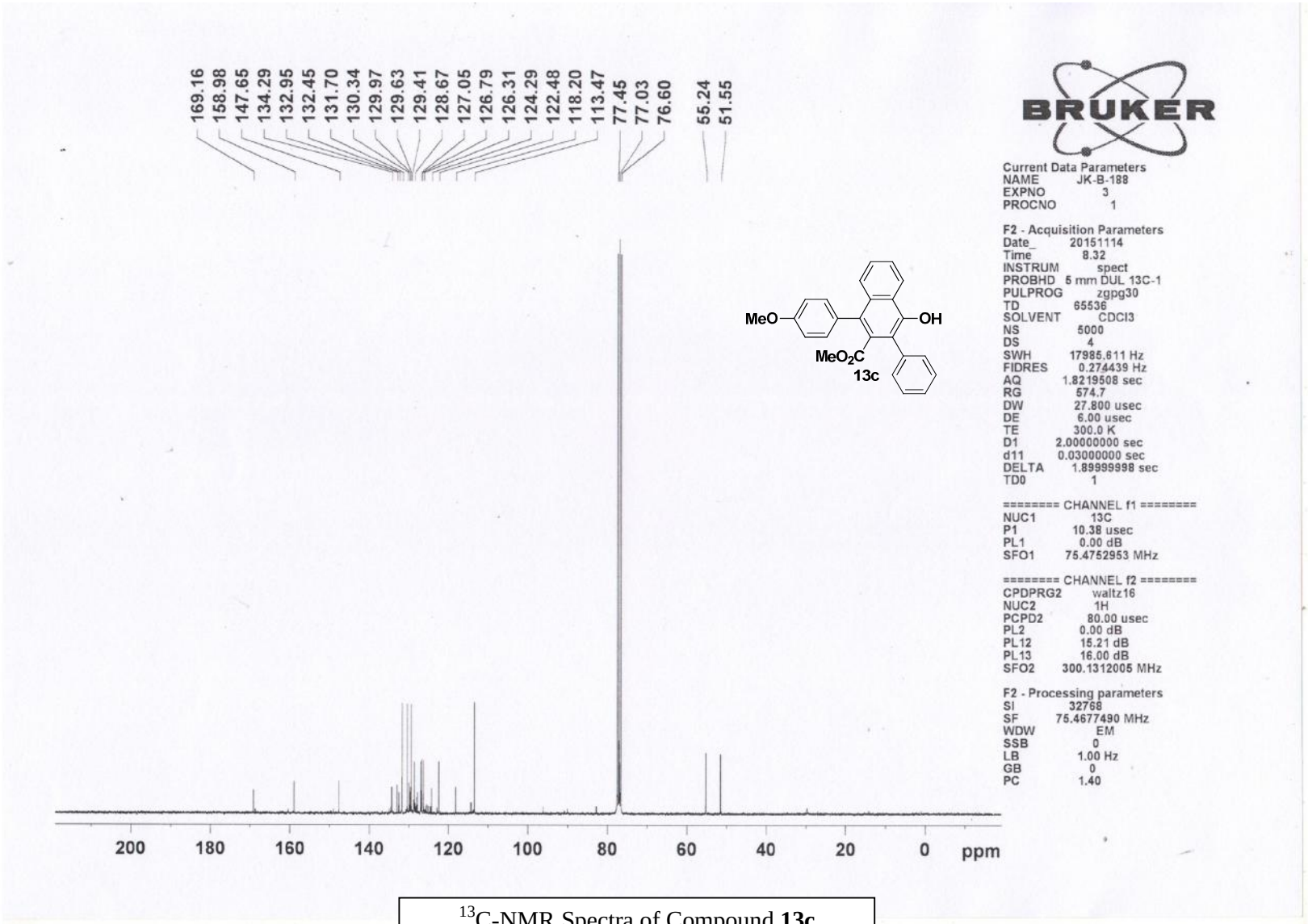
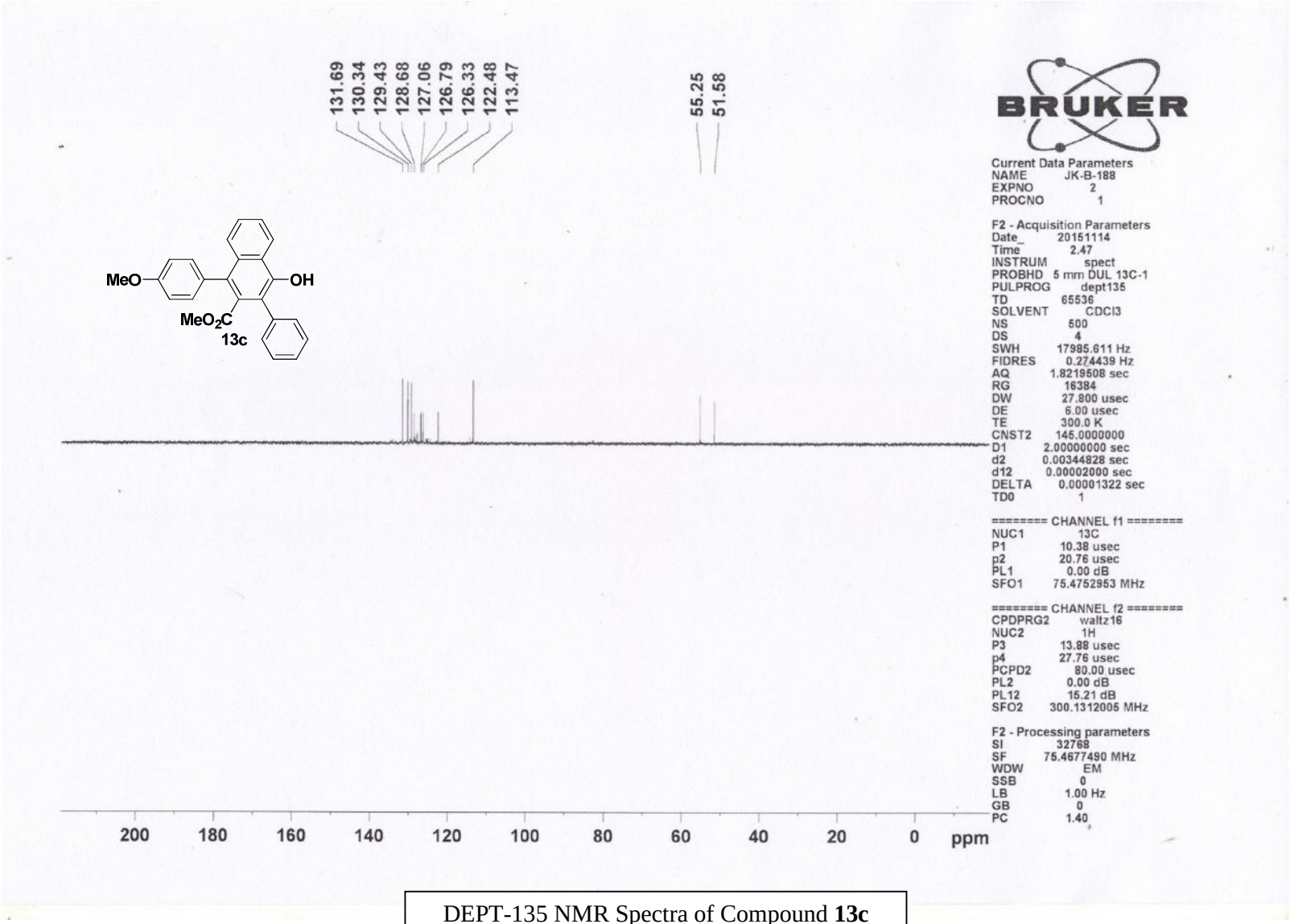


^{13}C-NMR Spectra of Compound **13c**



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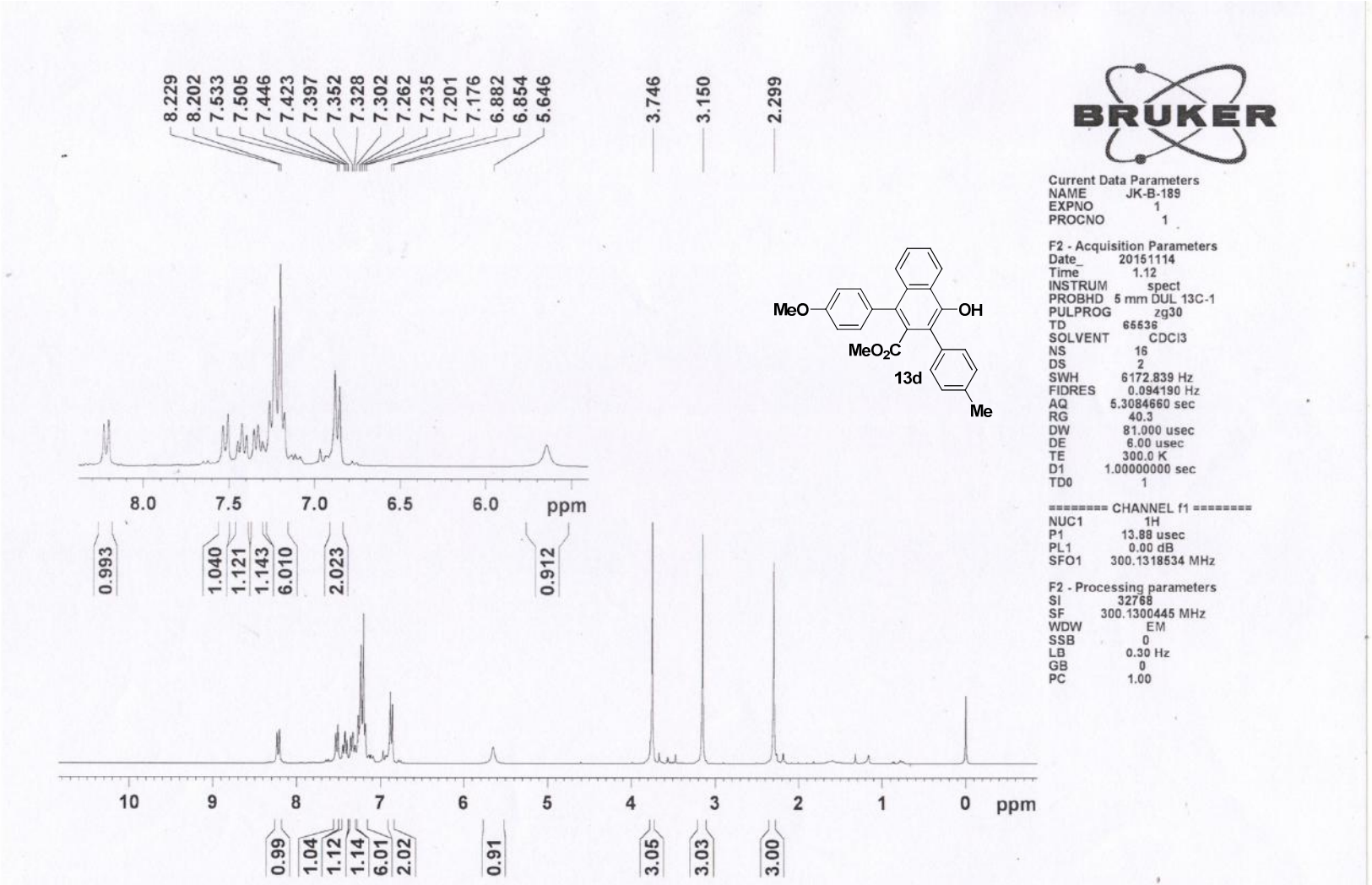

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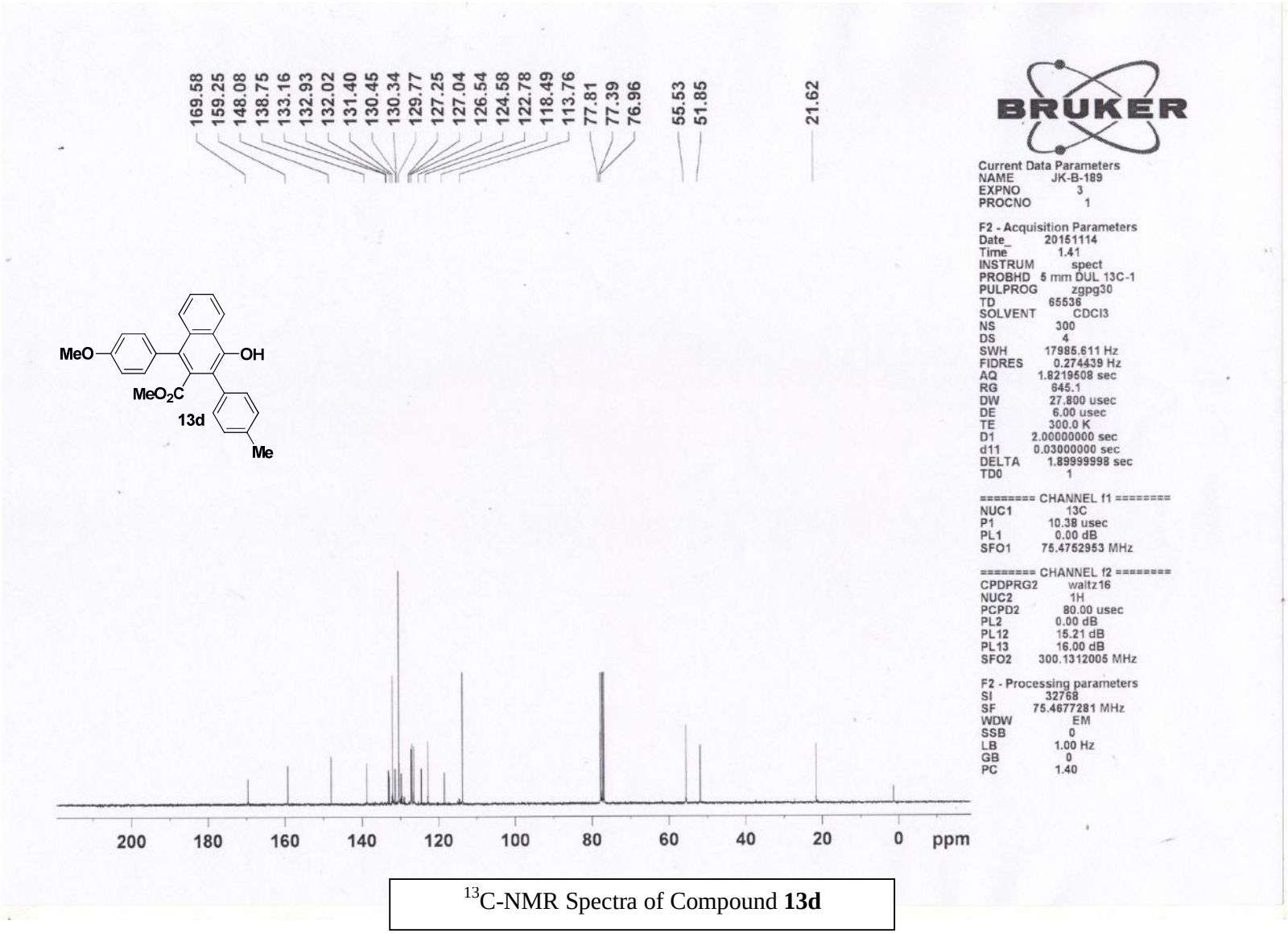

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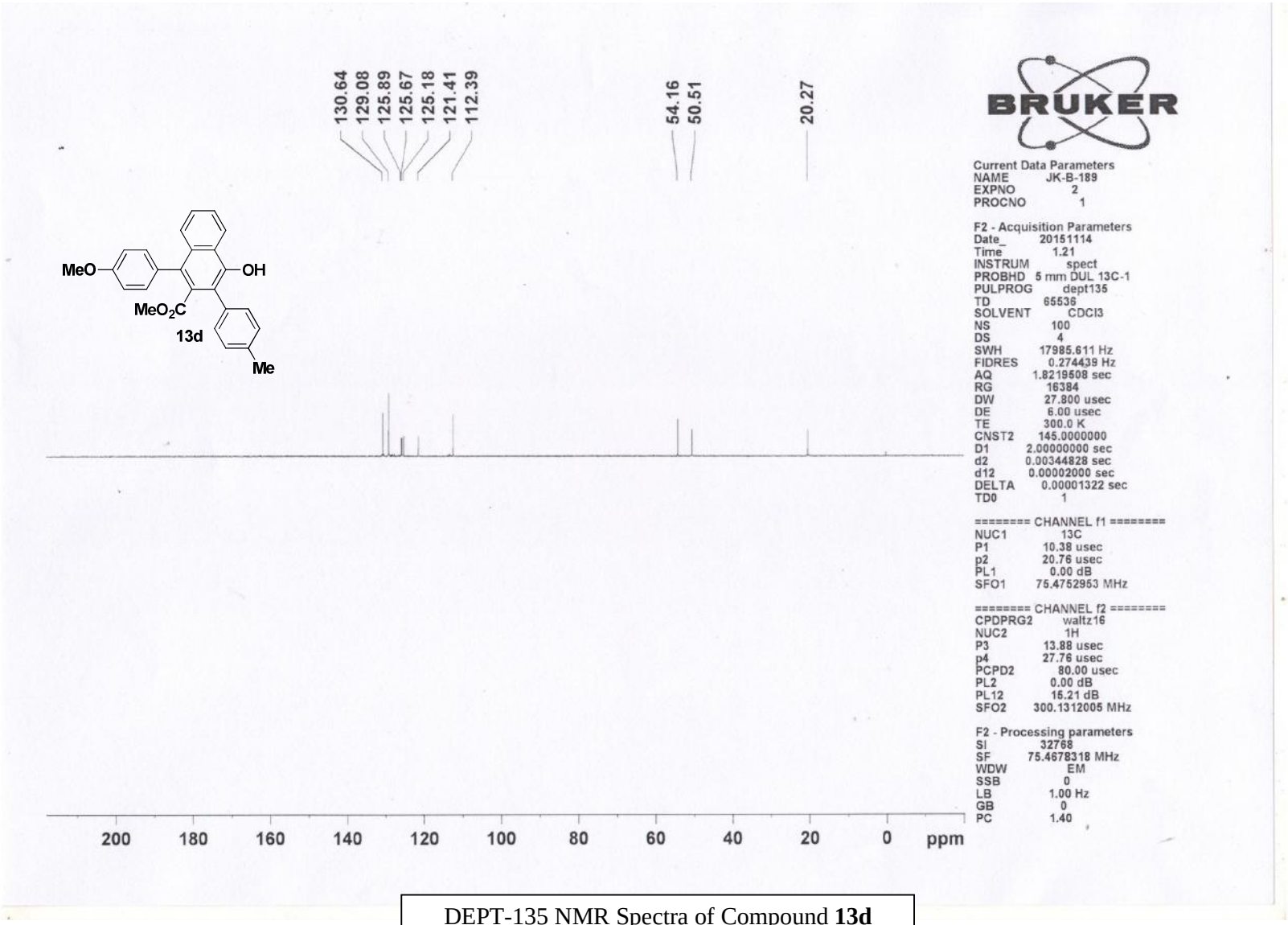

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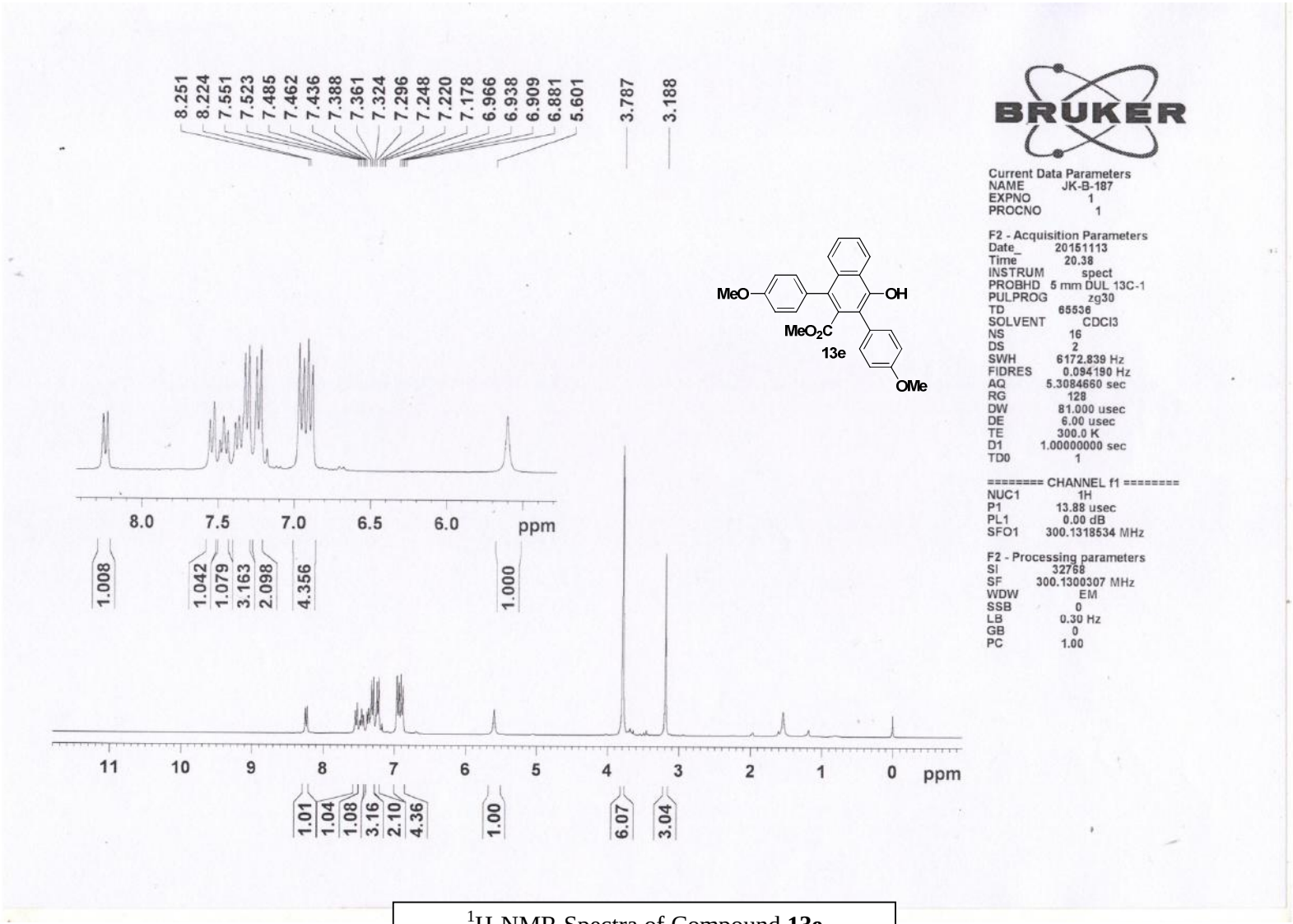

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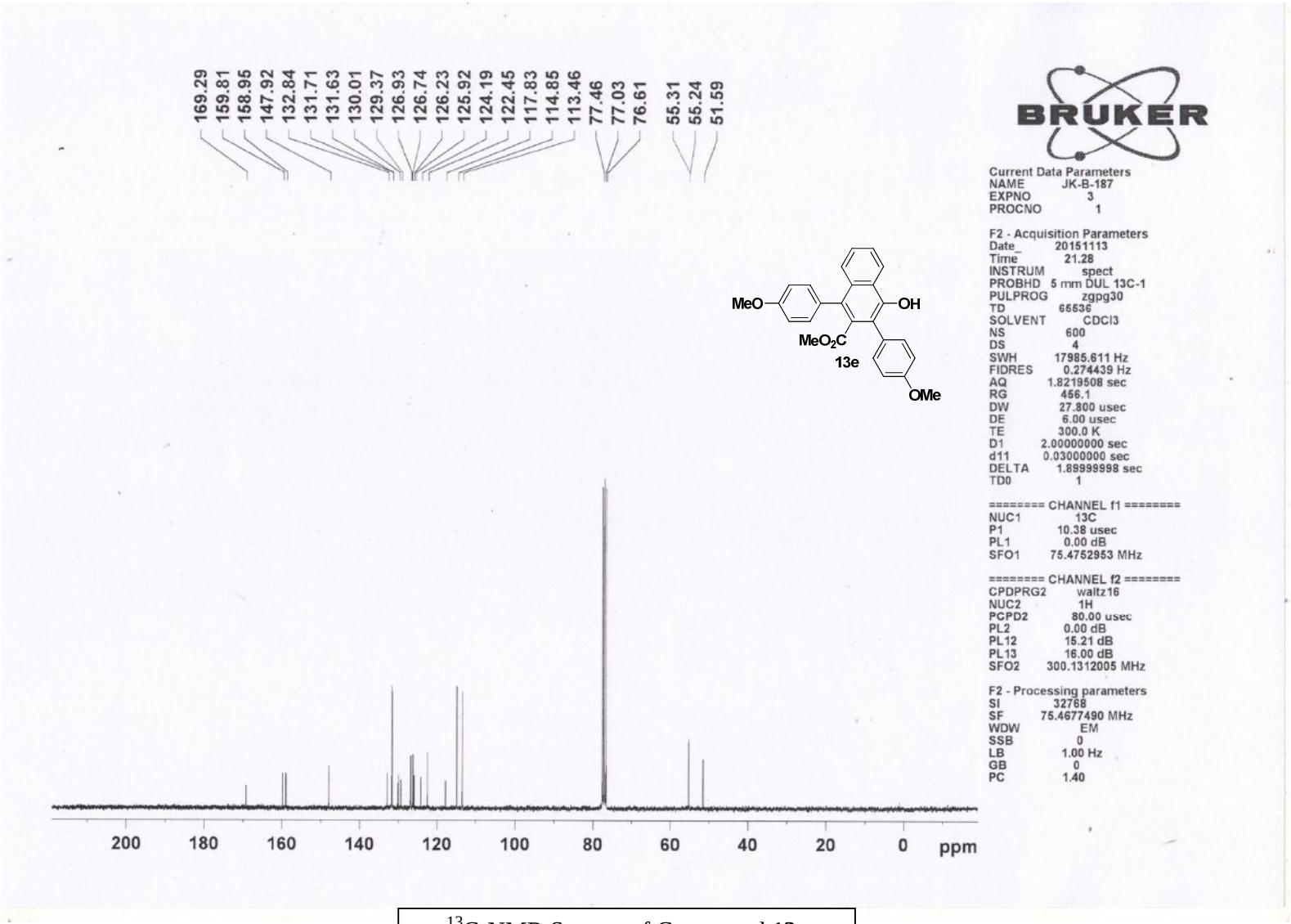

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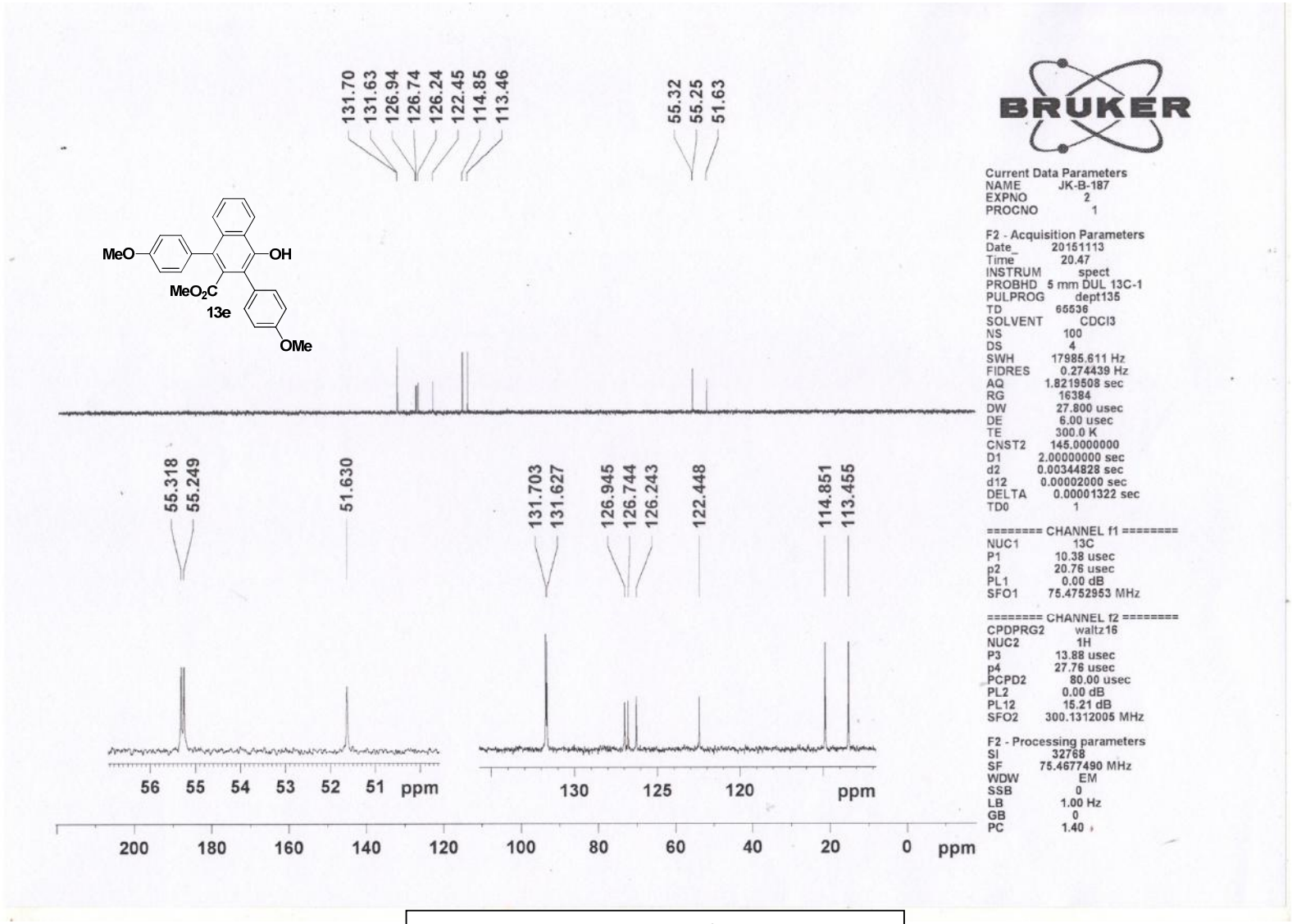

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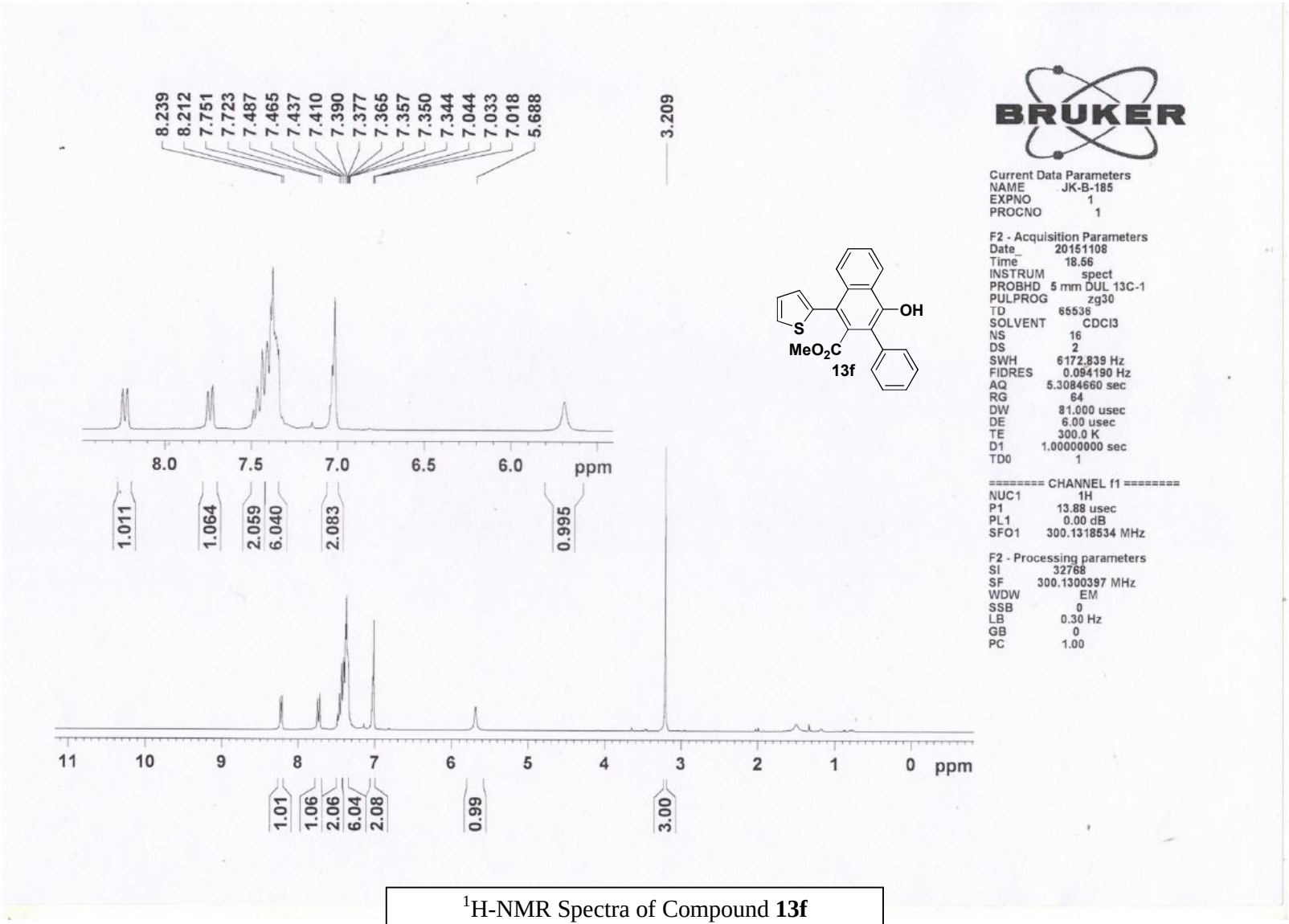
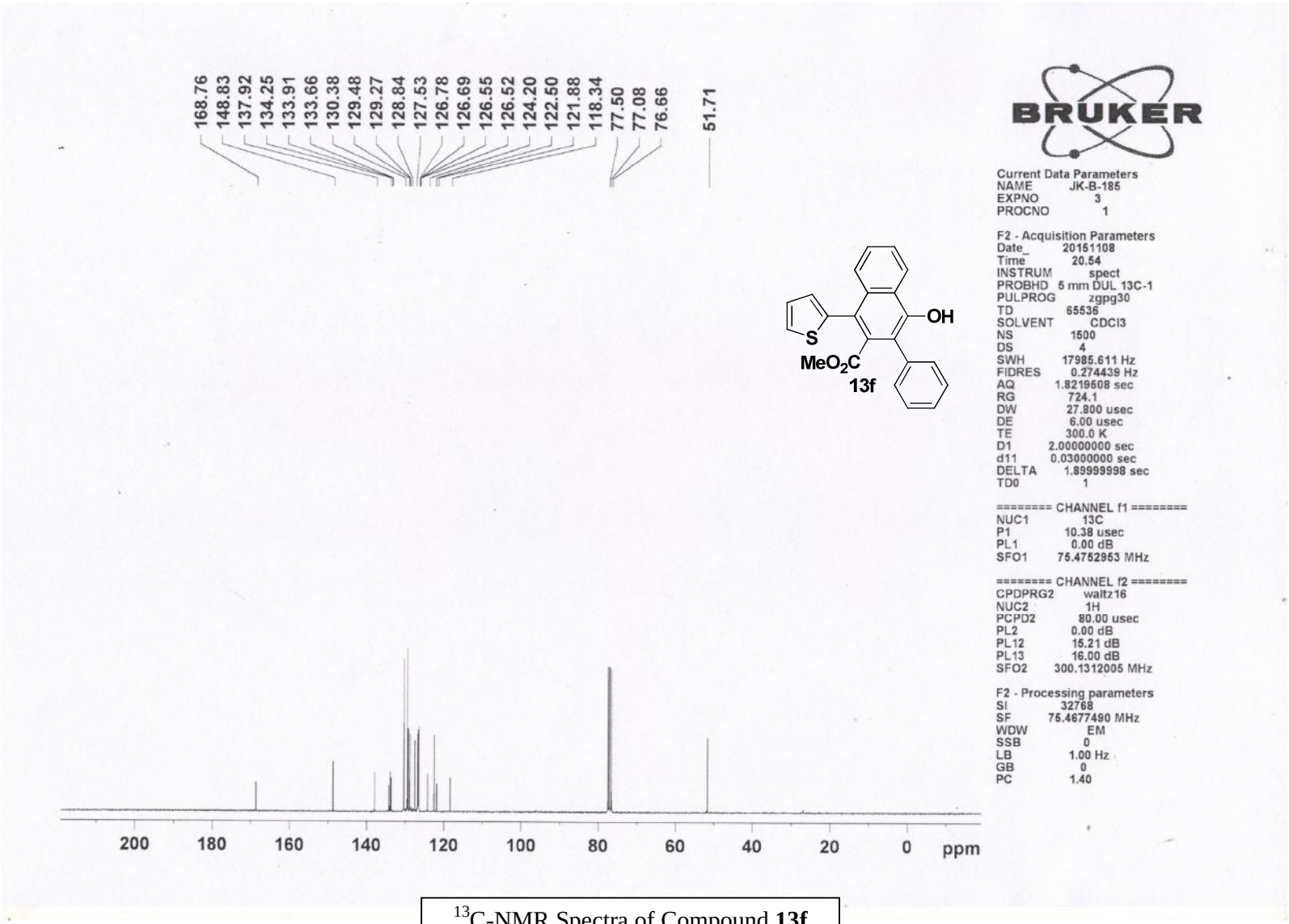


^{1}H-NMR Spectra of Compound **13f**



BRUKER

Current Data Parameters
NAME JK-B-185
EXPNO 3
PROCNO 1

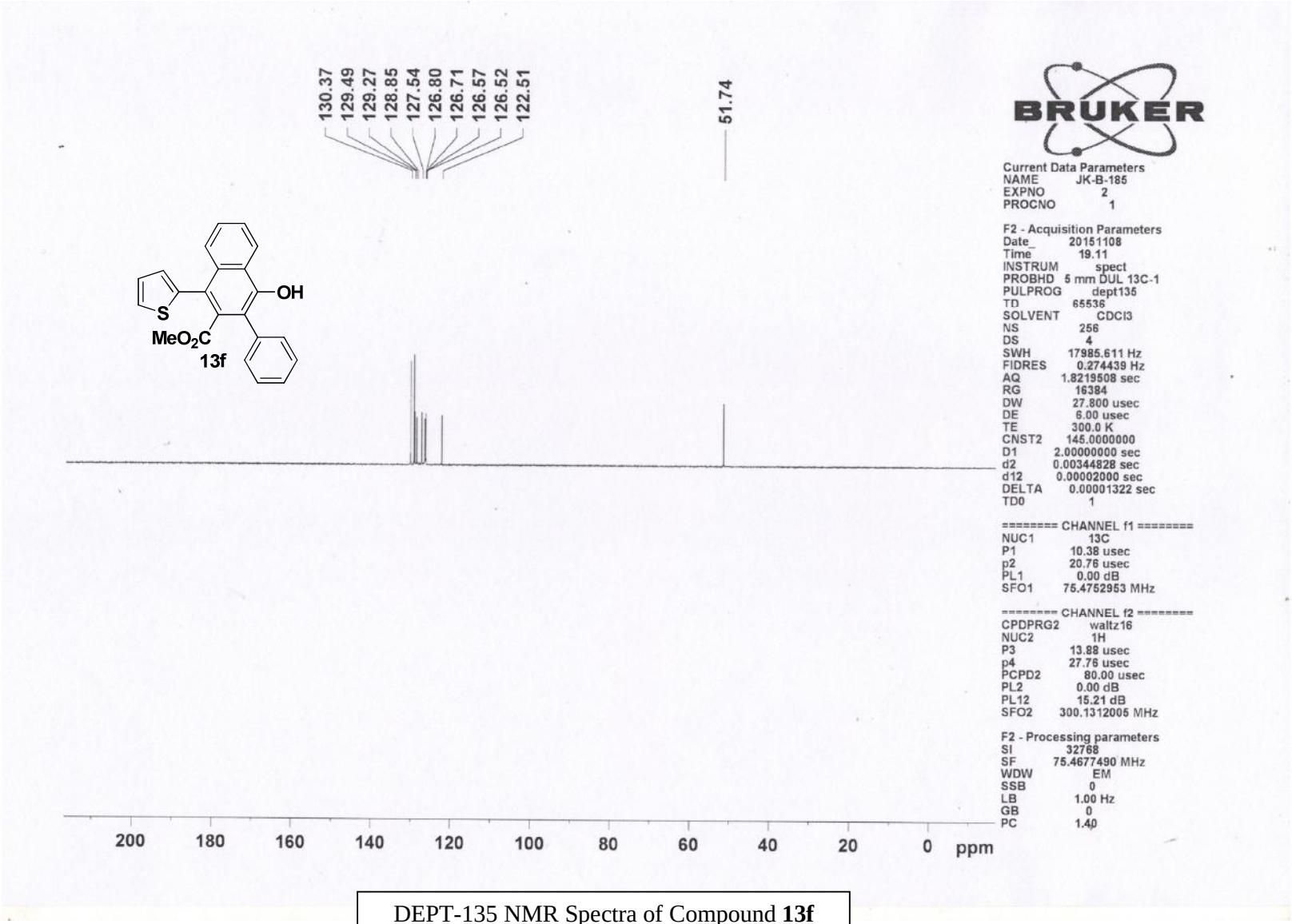
F2 - Acquisition Parameters
Date_ 20151108
Time 20.54
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1500
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 724.1
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.38 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

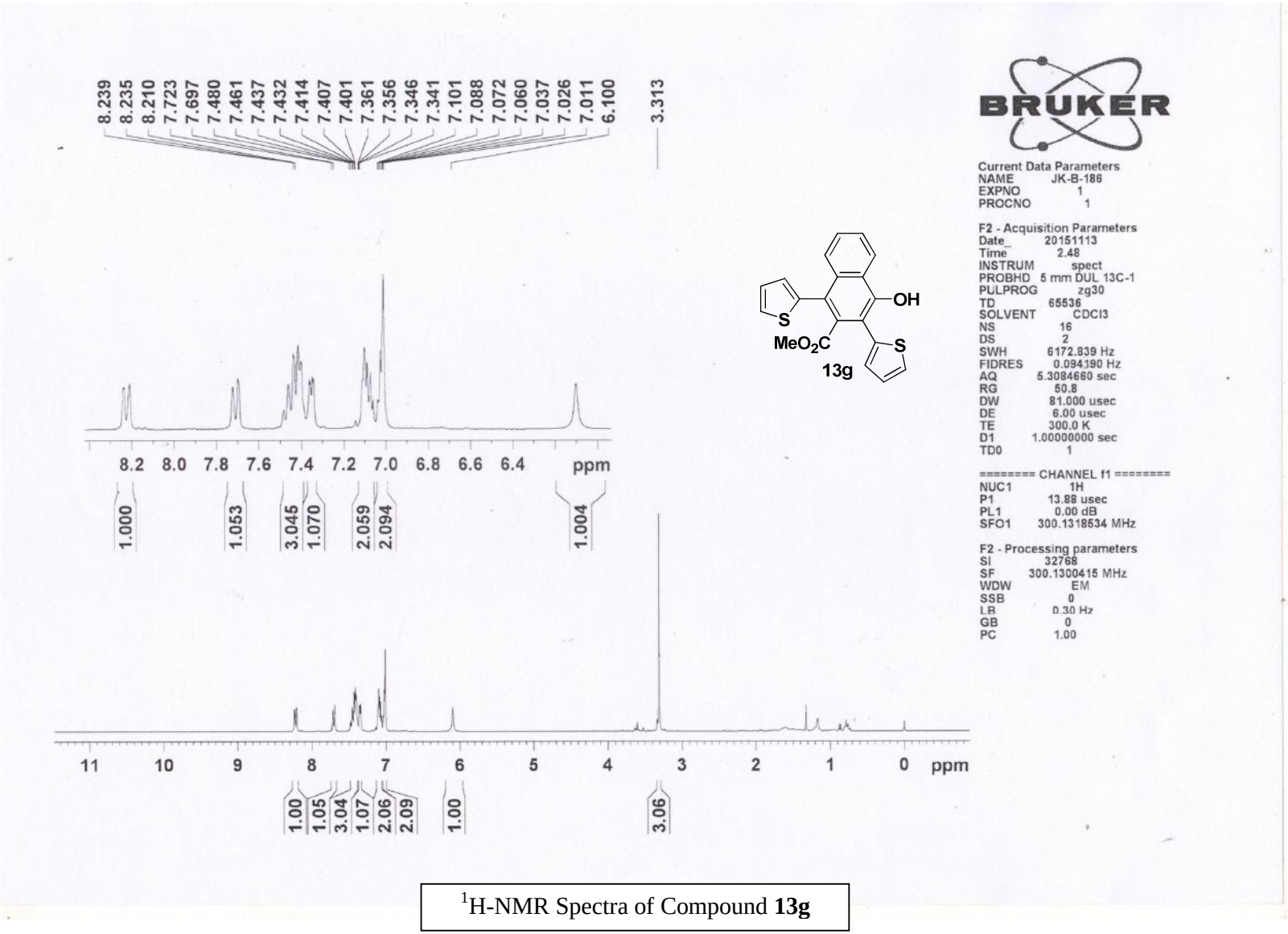
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.21 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

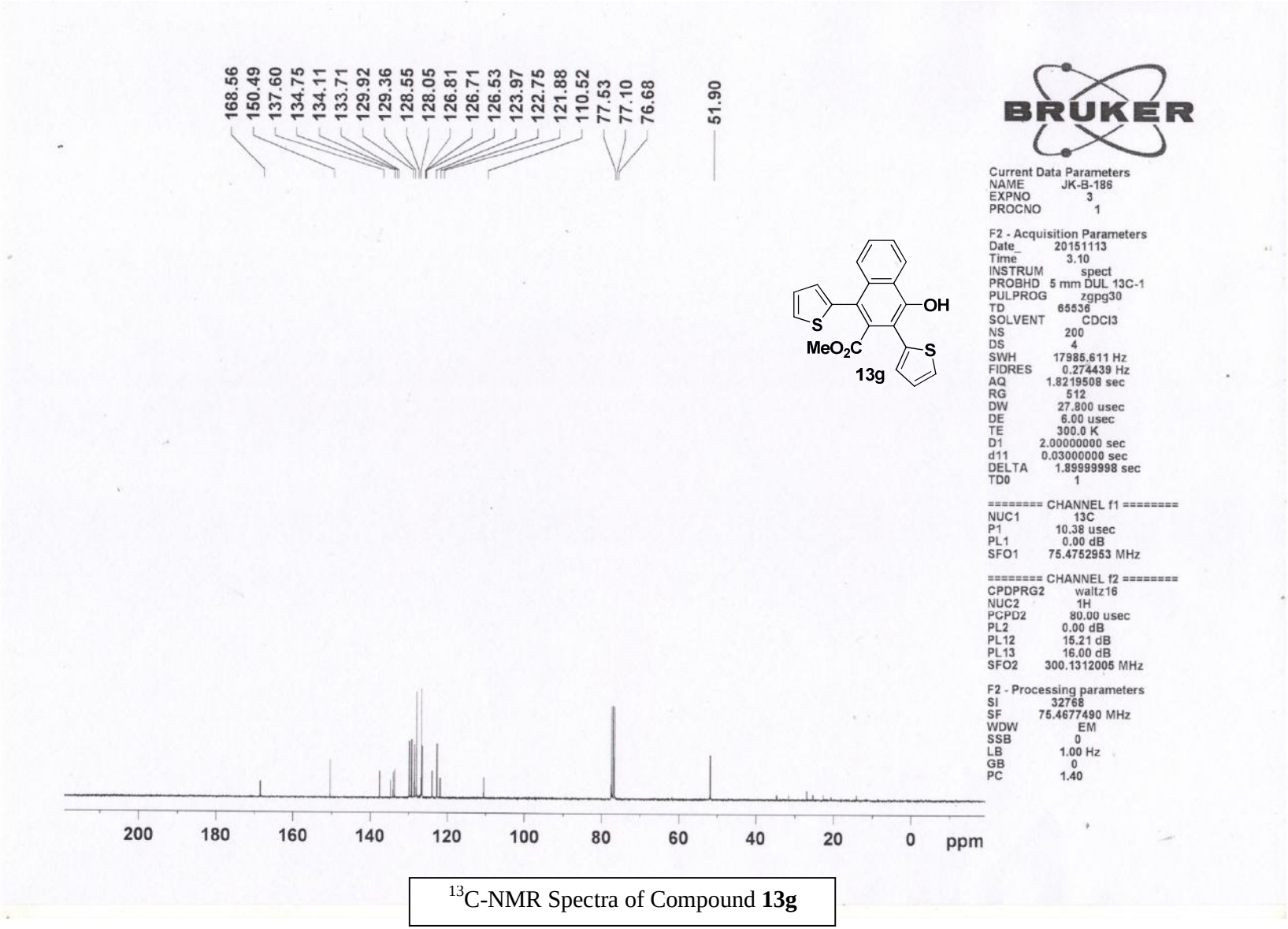
^{13}C-NMR Spectra of Compound **13f**



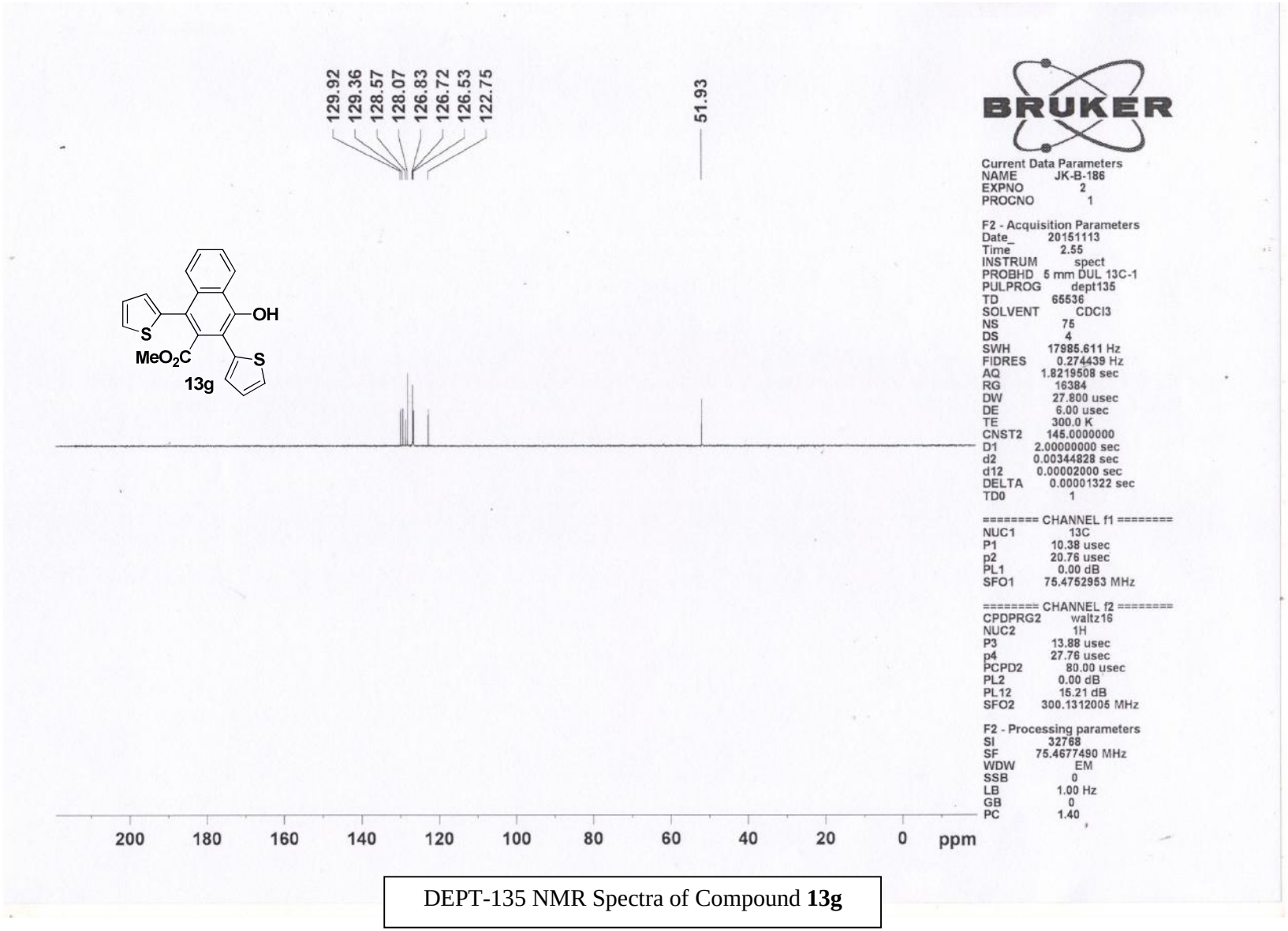
DEPT-135 NMR Spectra of Compound **13f**



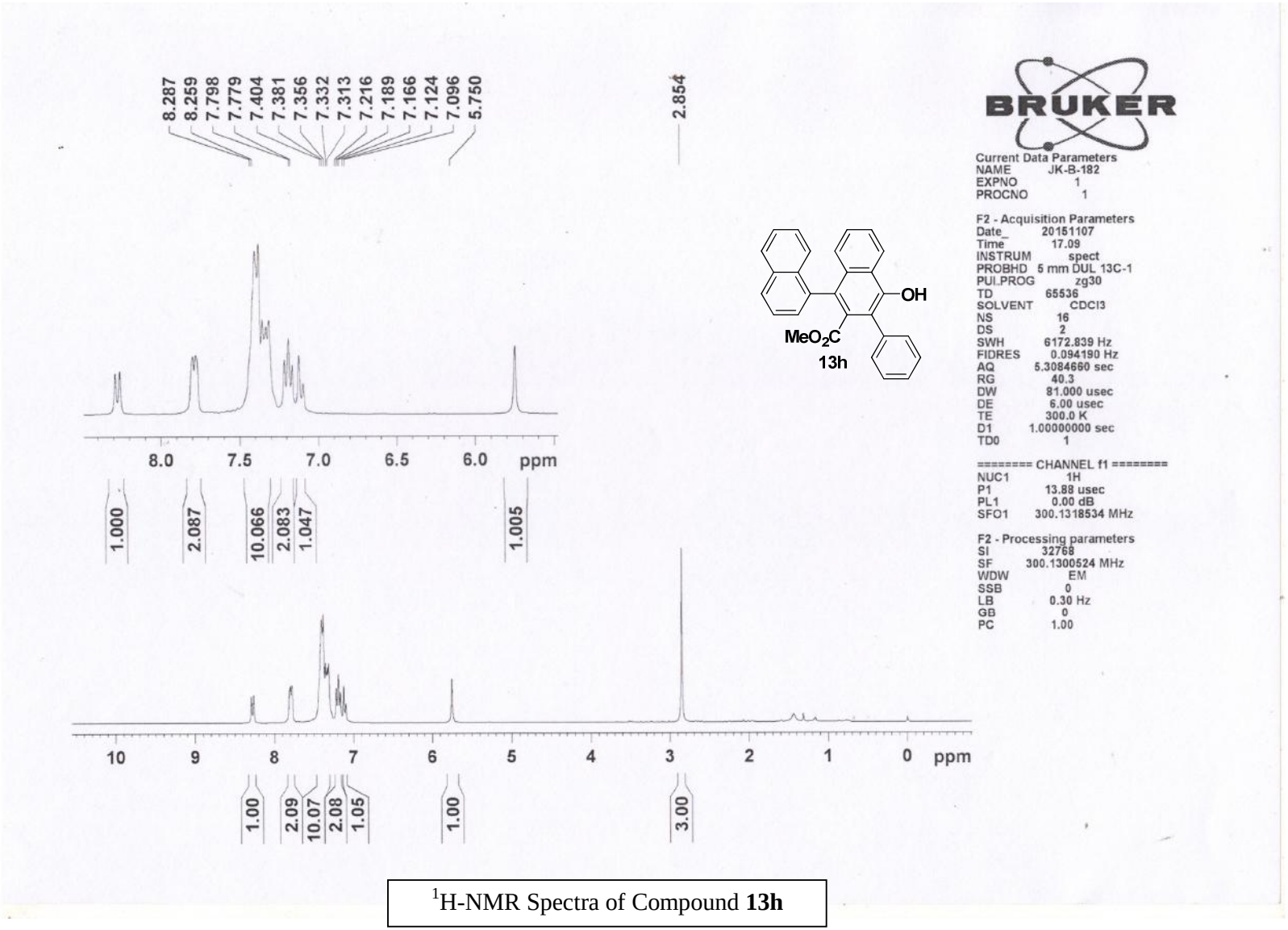
^{1}H-NMR Spectra of Compound **13g**



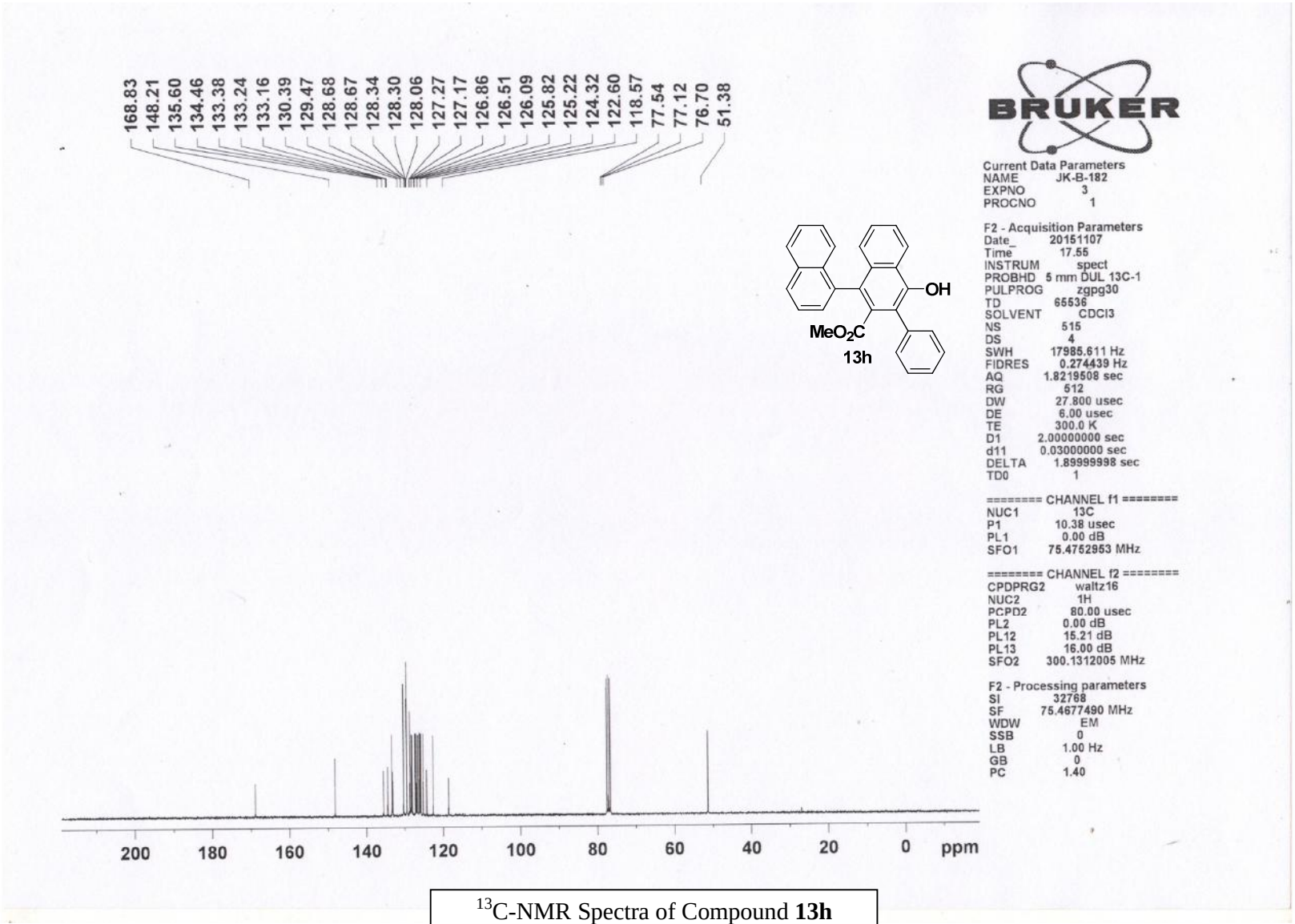
^{13}C-NMR Spectra of Compound **13g**



DEPT-135 NMR Spectra of Compound **13g**



^{1}H-NMR Spectra of Compound **13h**



BRUKER

Current Data Parameters
NAME JK-B-182
EXPNO 3
PROCNO 1

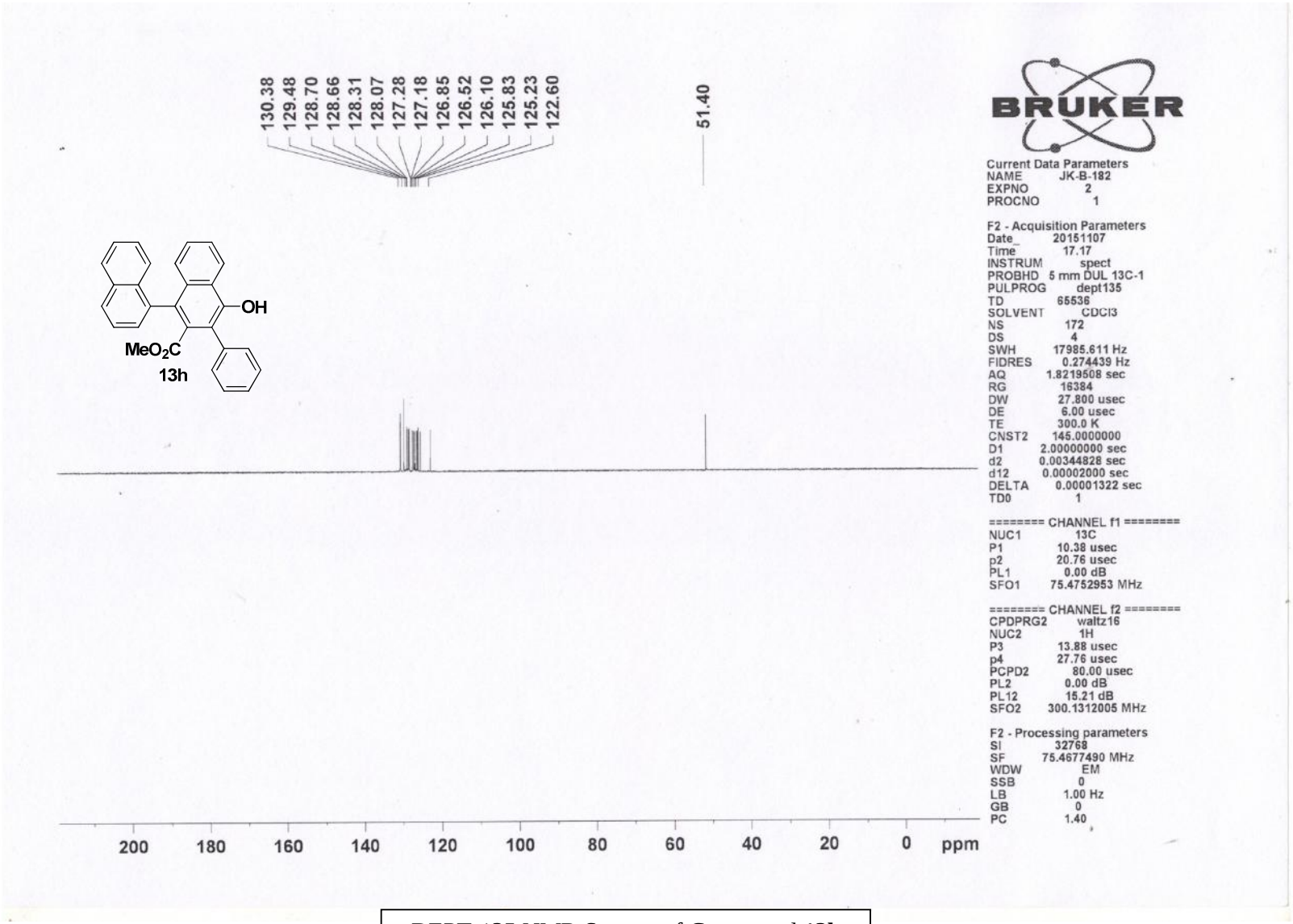
F2 - Acquisition Parameters
Date_ 20151107
Time 17.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 515
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 512
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.38 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

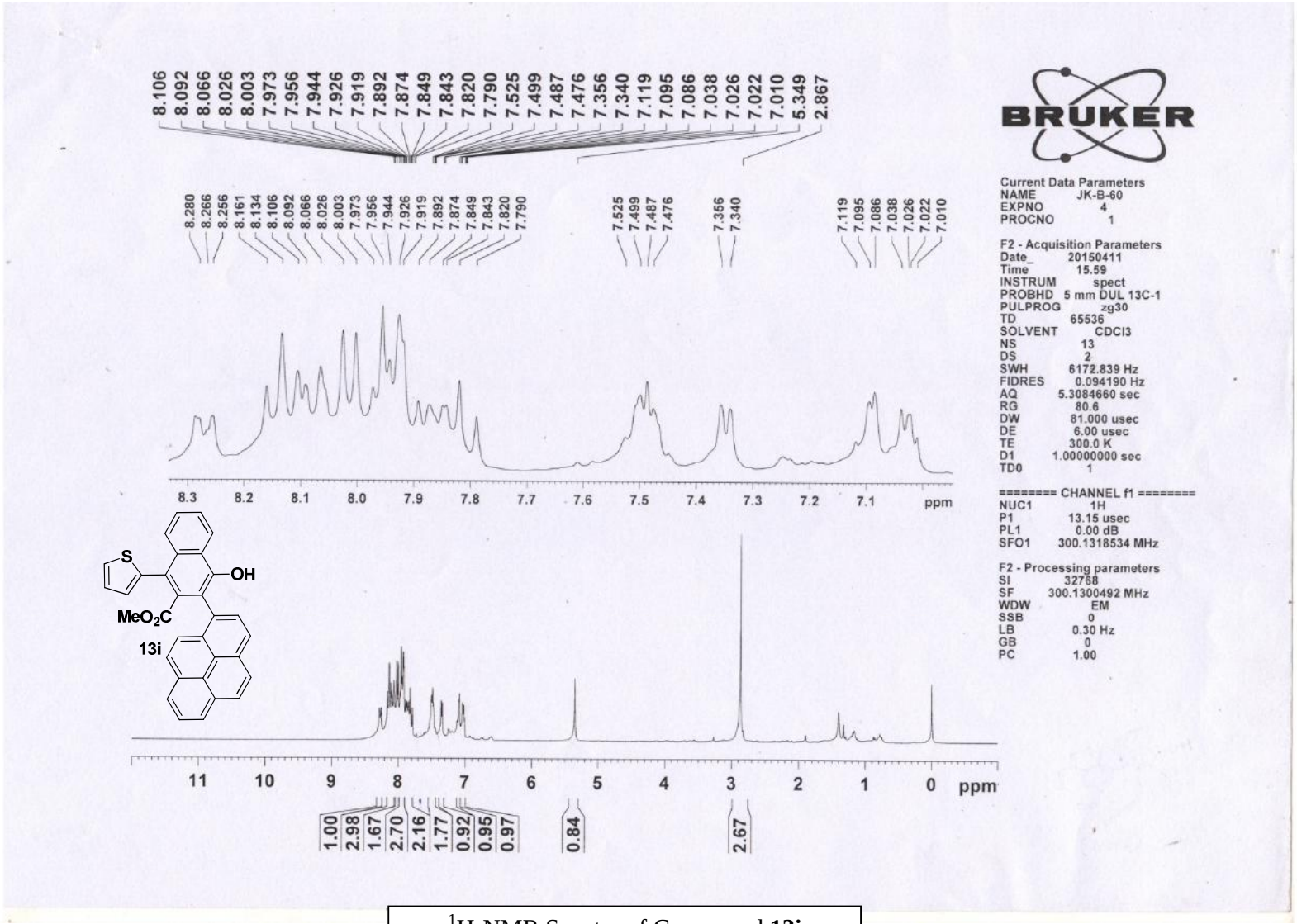
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.21 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

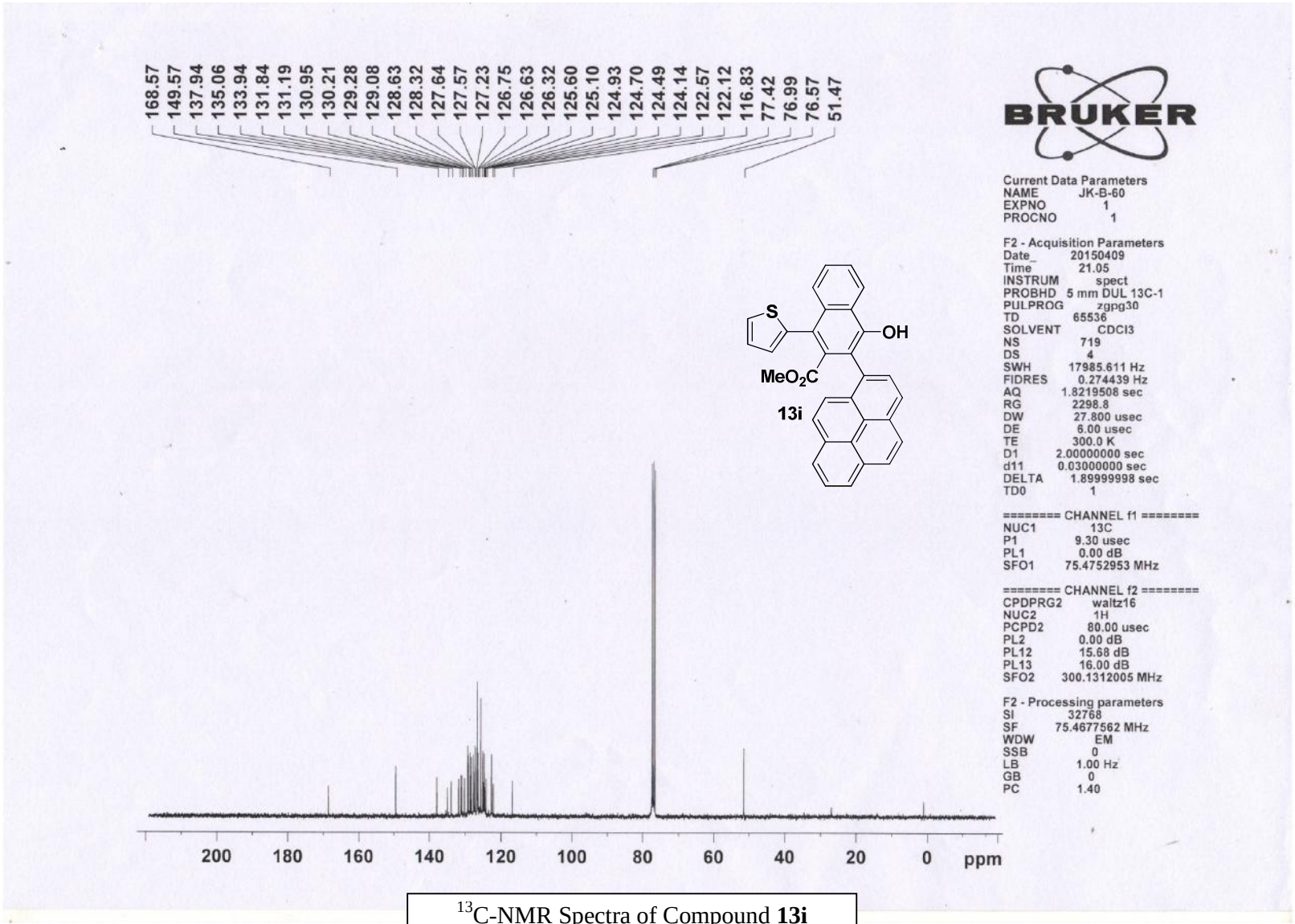
^{13}C-NMR Spectra of Compound **13h**



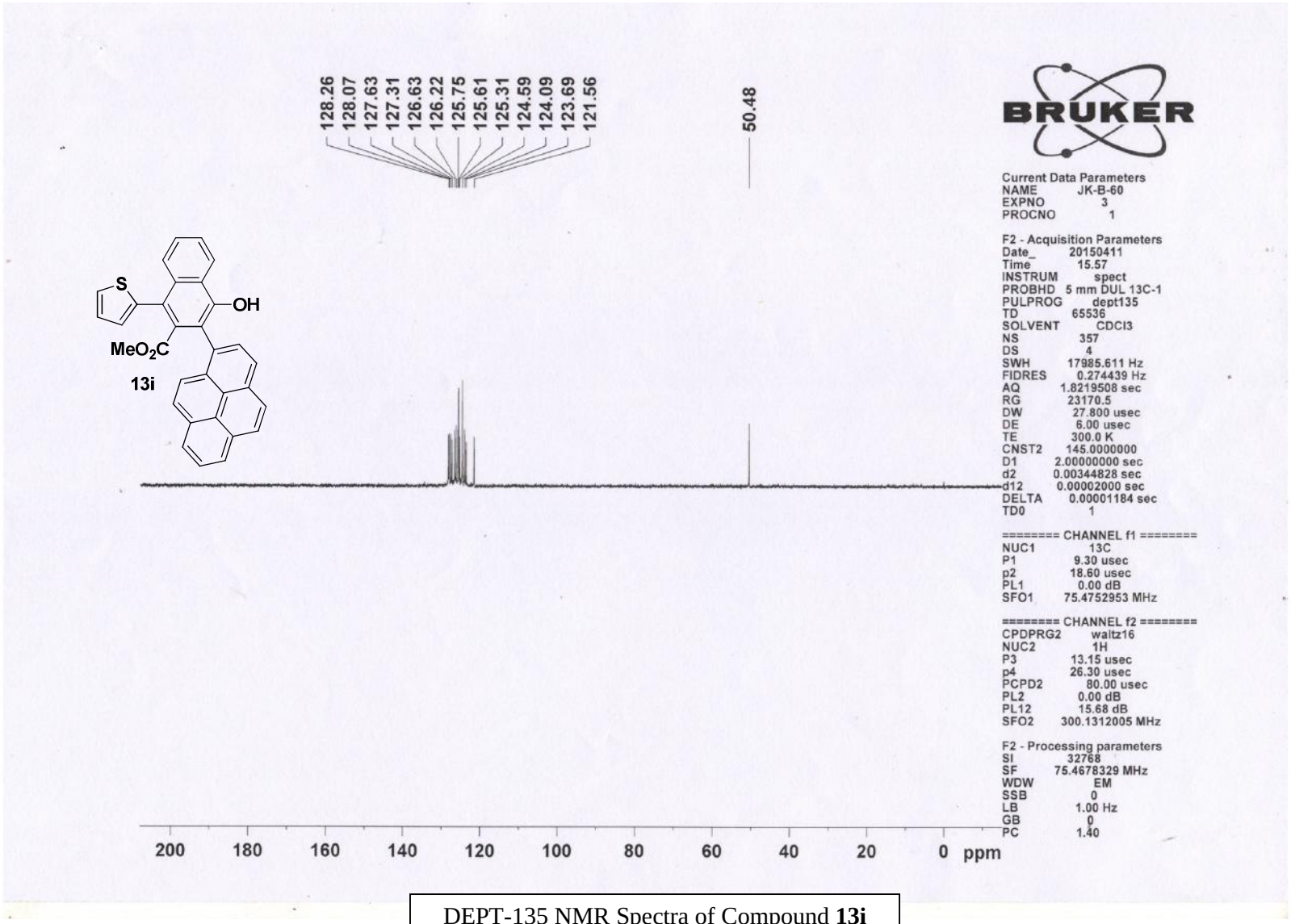
DEPT-135 NMR Spectra of Compound **13h**



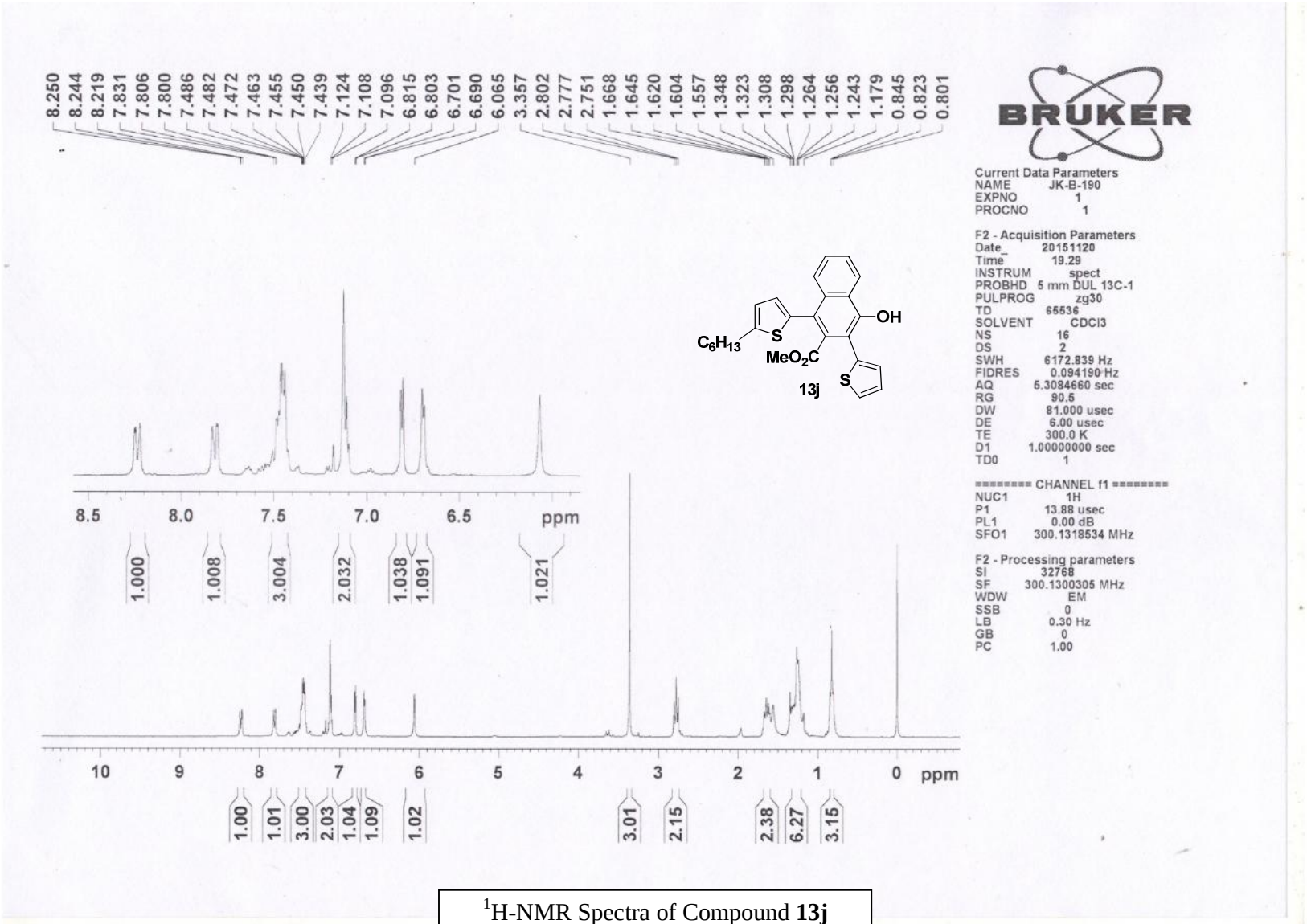
^{1}H-NMR Spectra of Compound **13i**



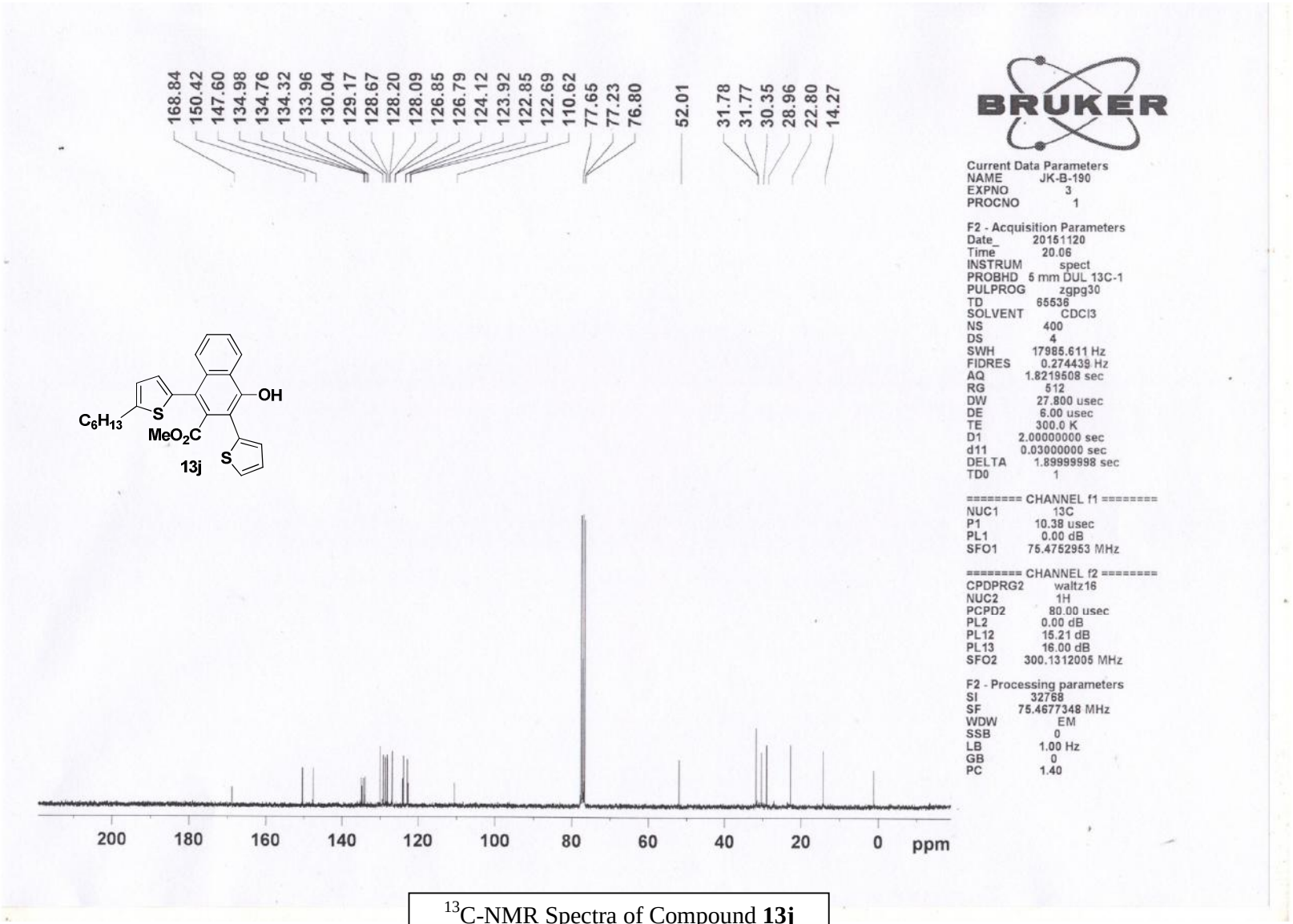
^{13}C-NMR Spectra of Compound **13i**



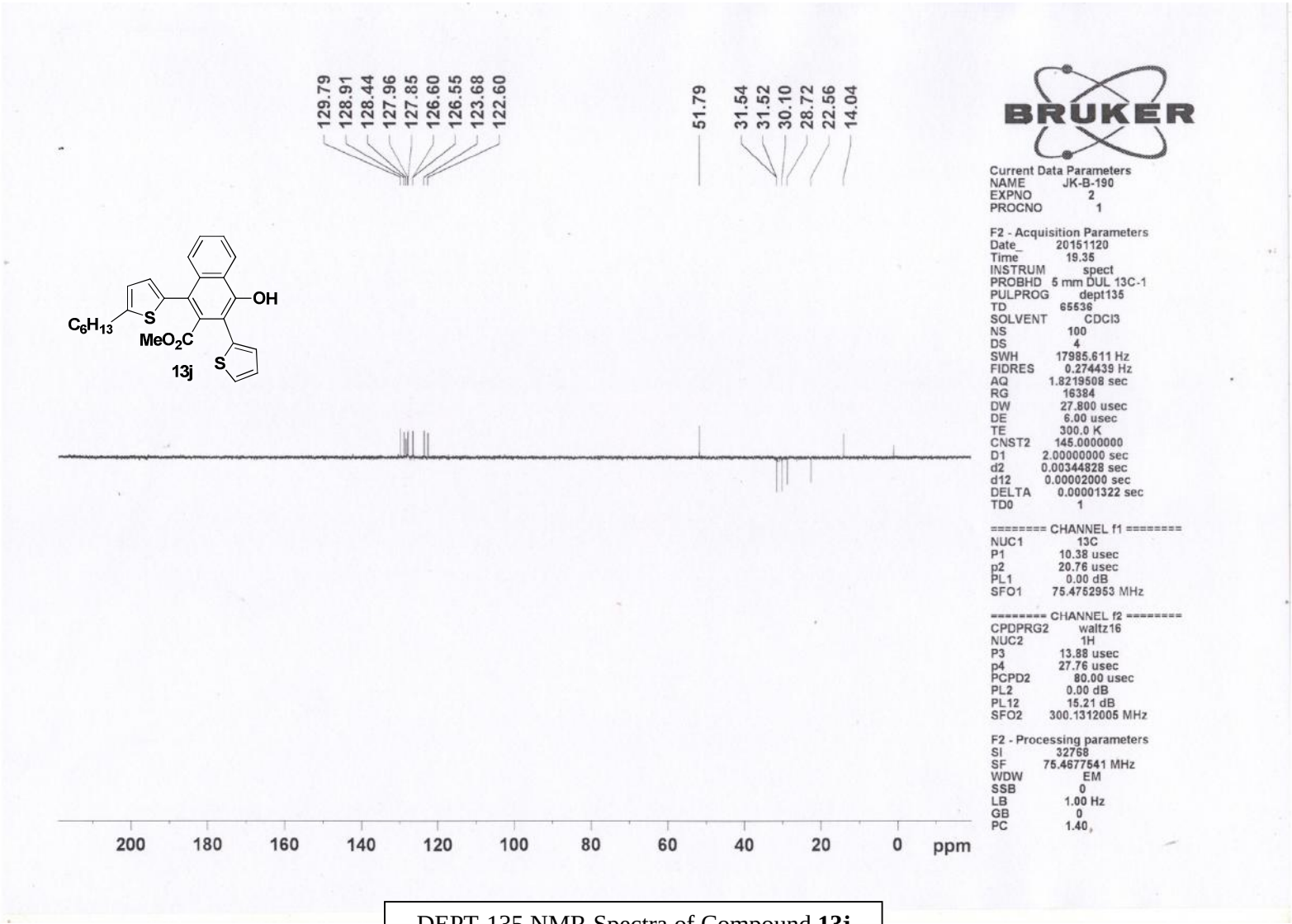
DEPT-135 NMR Spectra of Compound **13i**



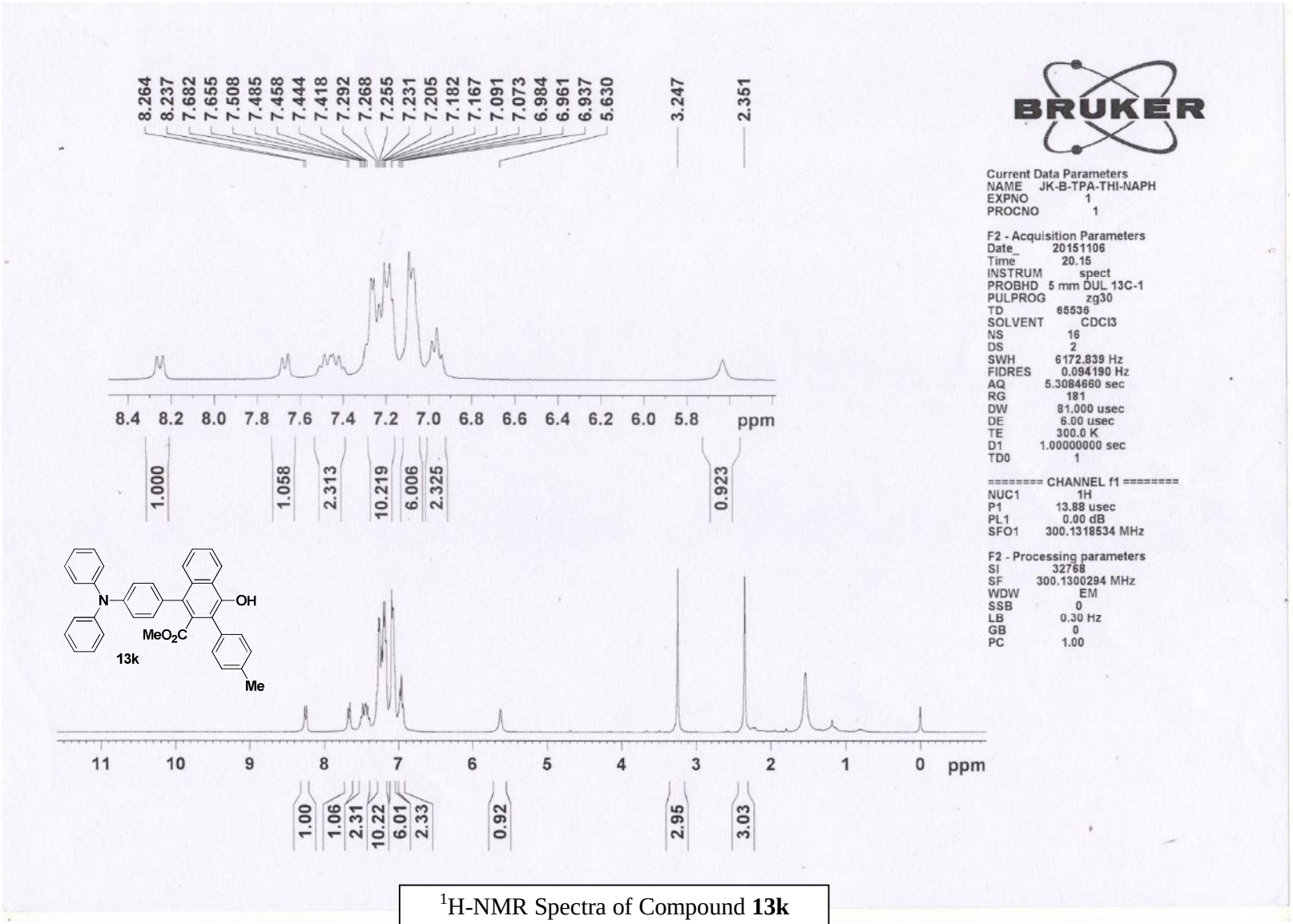
^{1}H-NMR Spectra of Compound **13j**



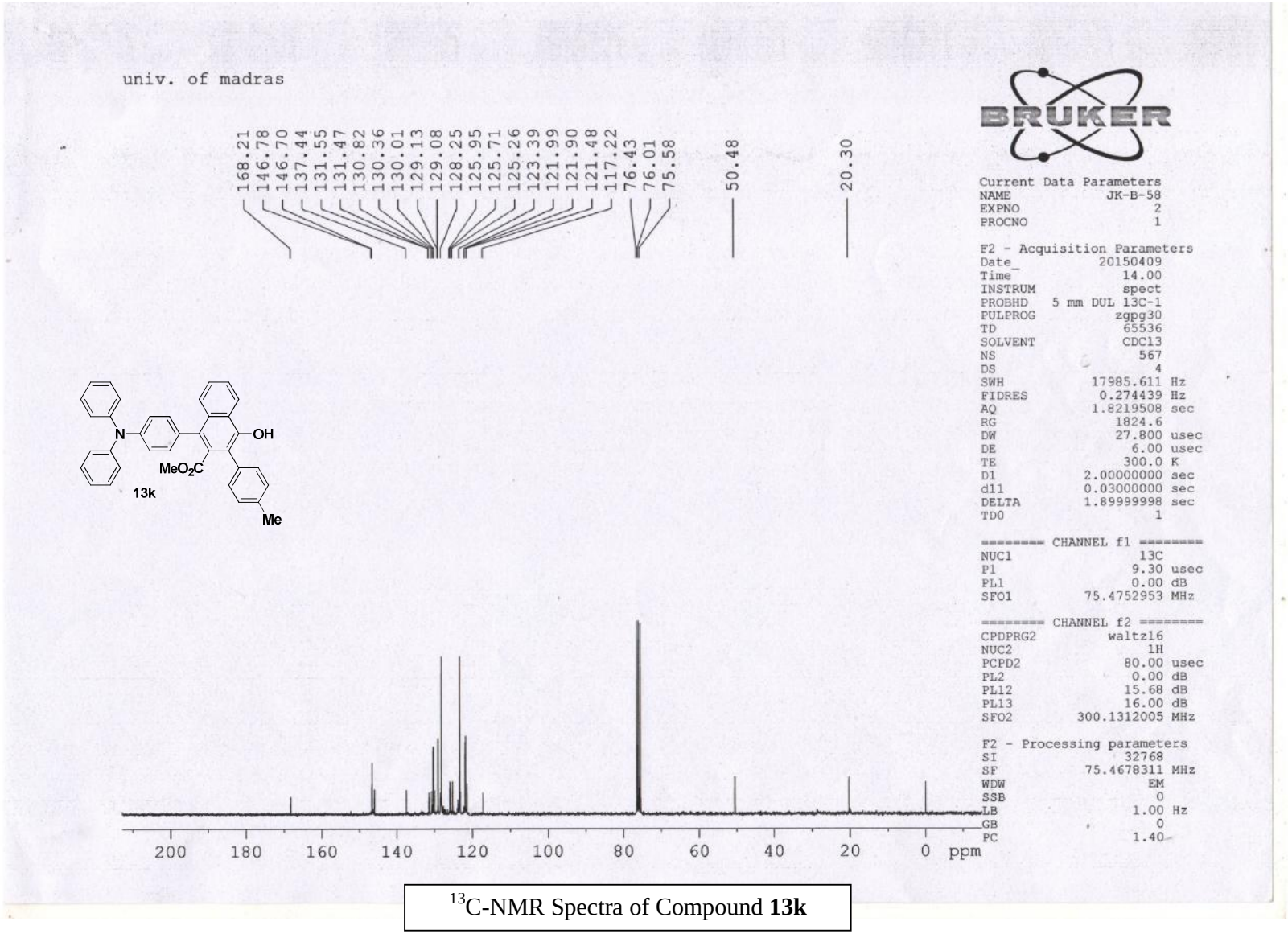
^{13}C-NMR Spectra of Compound **13j**



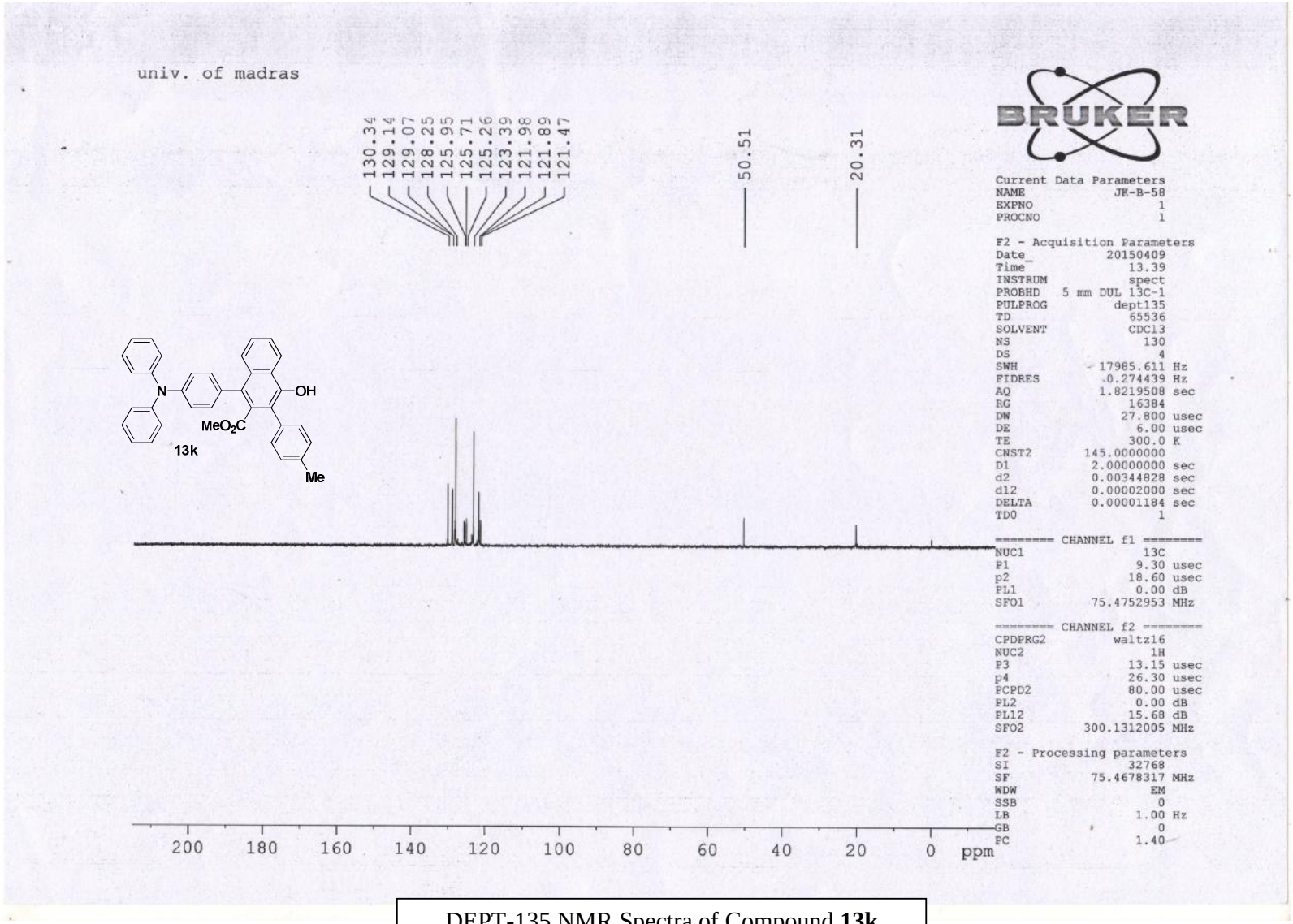
DEPT-135 NMR Spectra of Compound **13j**



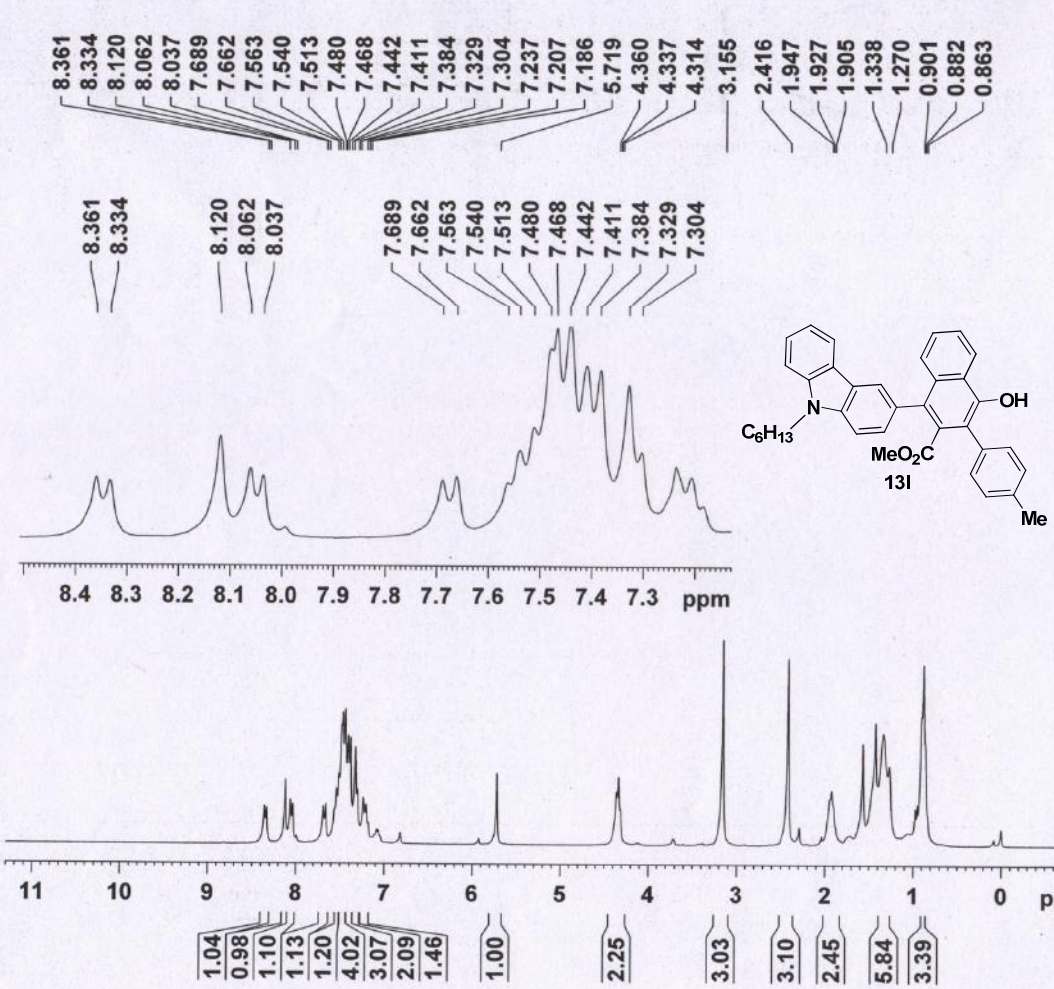
^{1}H-NMR Spectra of Compound **13k**



^{13}C-NMR Spectra of Compound **13k**



DEPT-135 NMR Spectra of Compound **13k**



BRUKER

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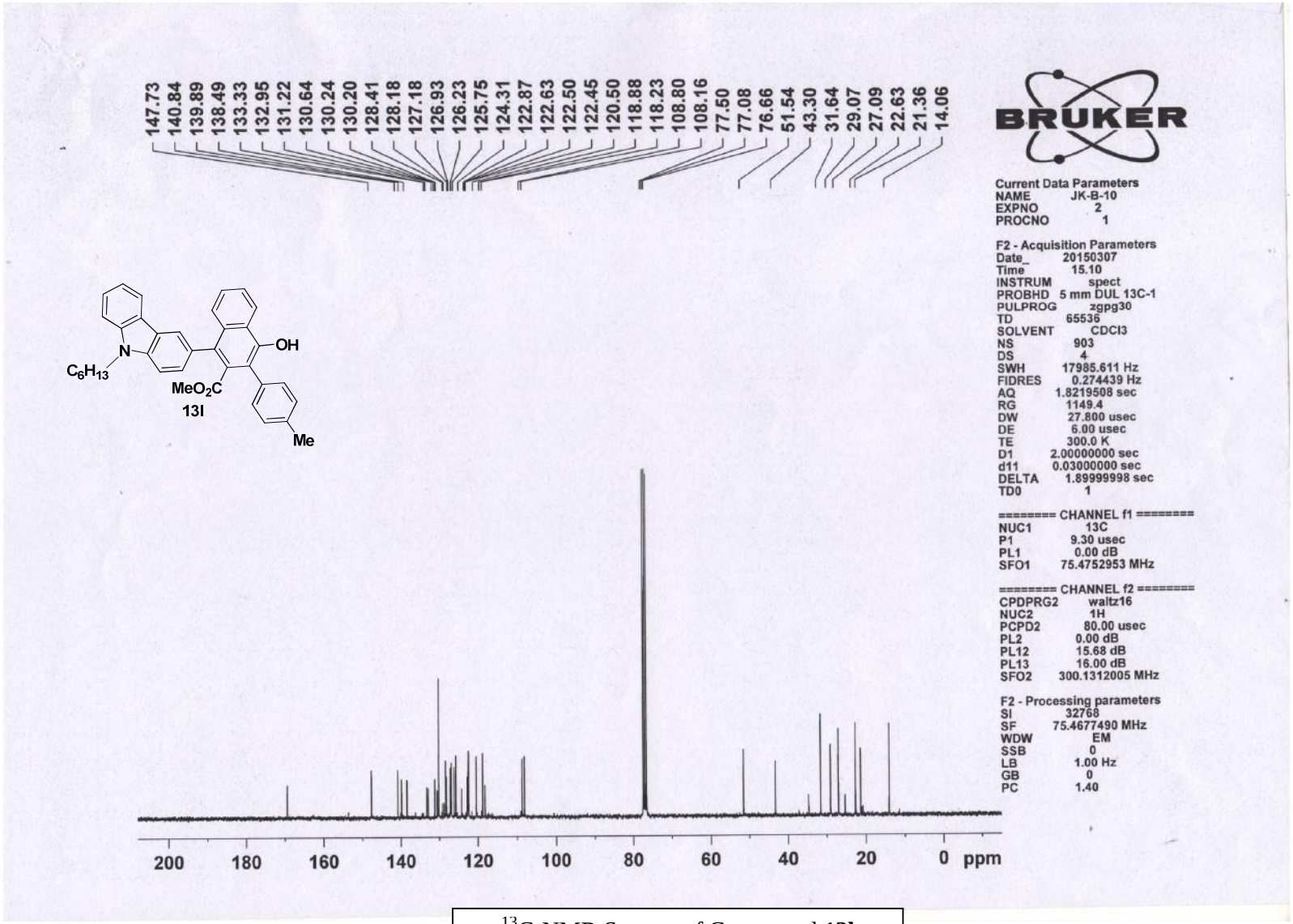
Current Data Parameters
NAME                  JK-B-10
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_                20150306
Time                    21.17
INSTRUM                 spect
PROBHD   5 mm DUL 13C-1
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         12
DS                          2
SWH                  6172.839 Hz
FIDRES               0.094190 Hz
AQ                   5.3084660 sec
RG                        80.6
DW                      81.000 usec
DE                        6.00 usec
TE                       300.0 K
D1                   1.00000000 sec
TD0                         1

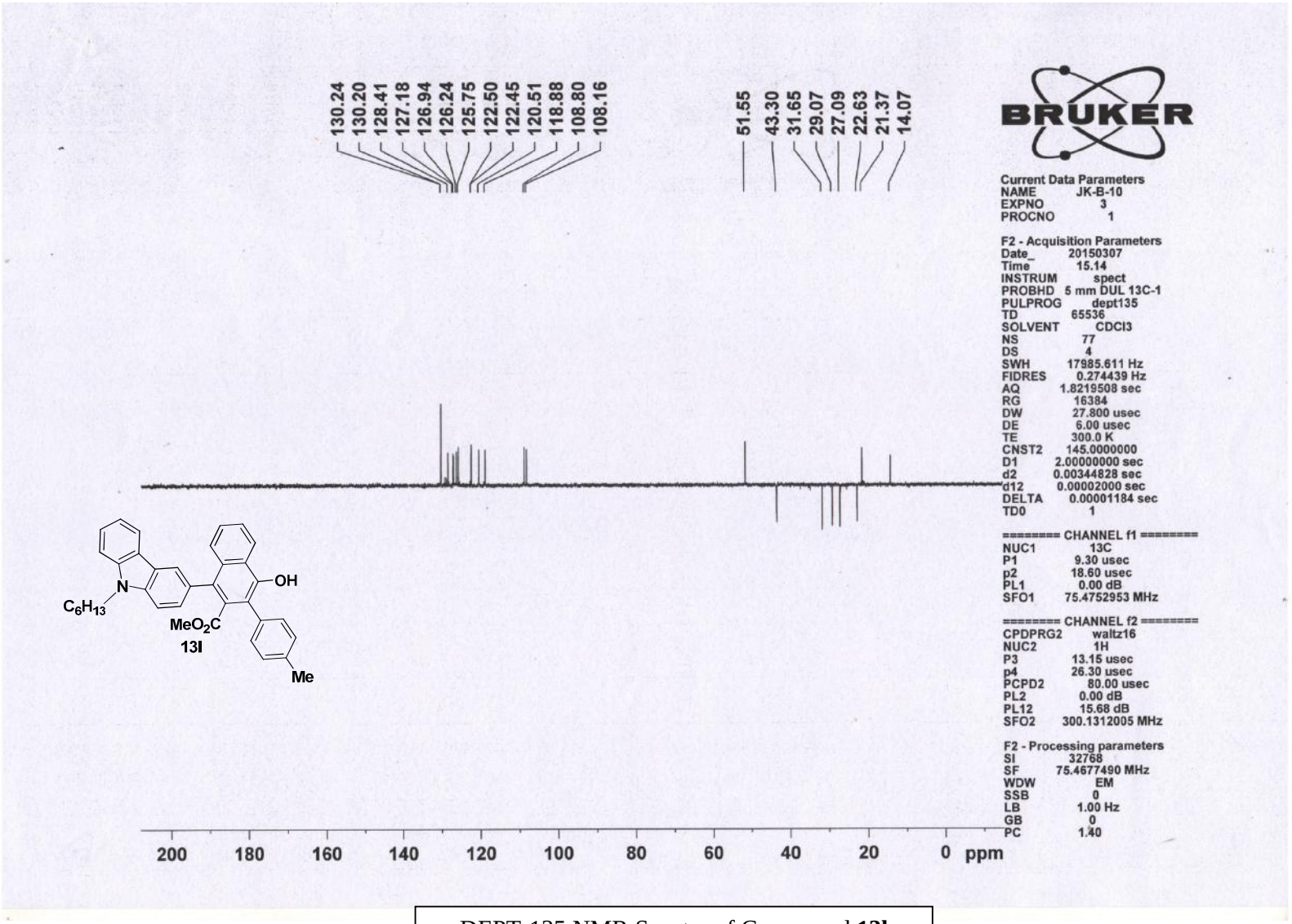
======== CHANNEL f1 ========
NUC1                       1H
P1                      13.15 usec
PL1                      0.00 dB
SFO1            300.1318534 MHz

F2 - Processing parameters
SI                      32768
SF               300.1300136 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

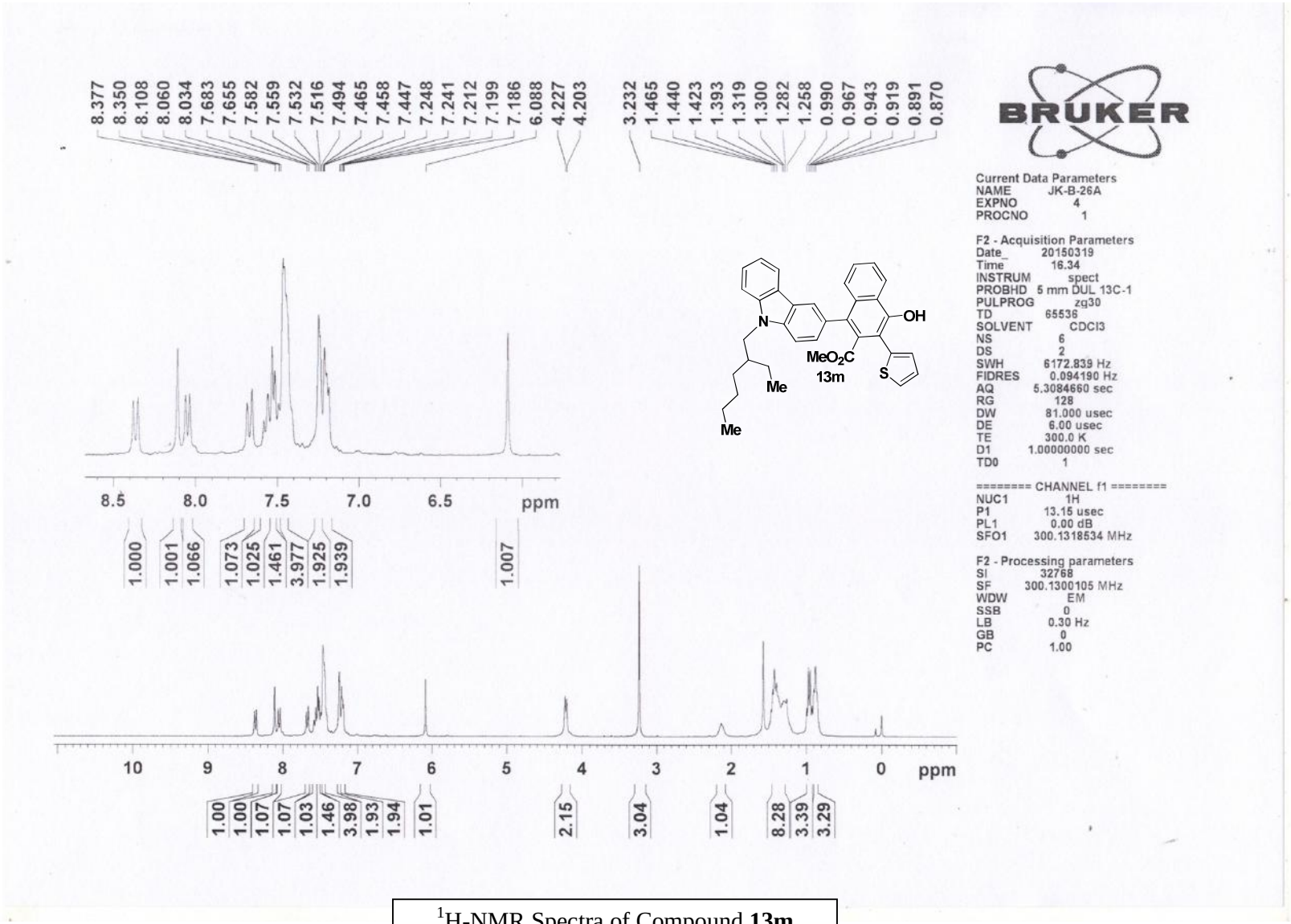
[1]H-NMR Spectra of Compound **13l**



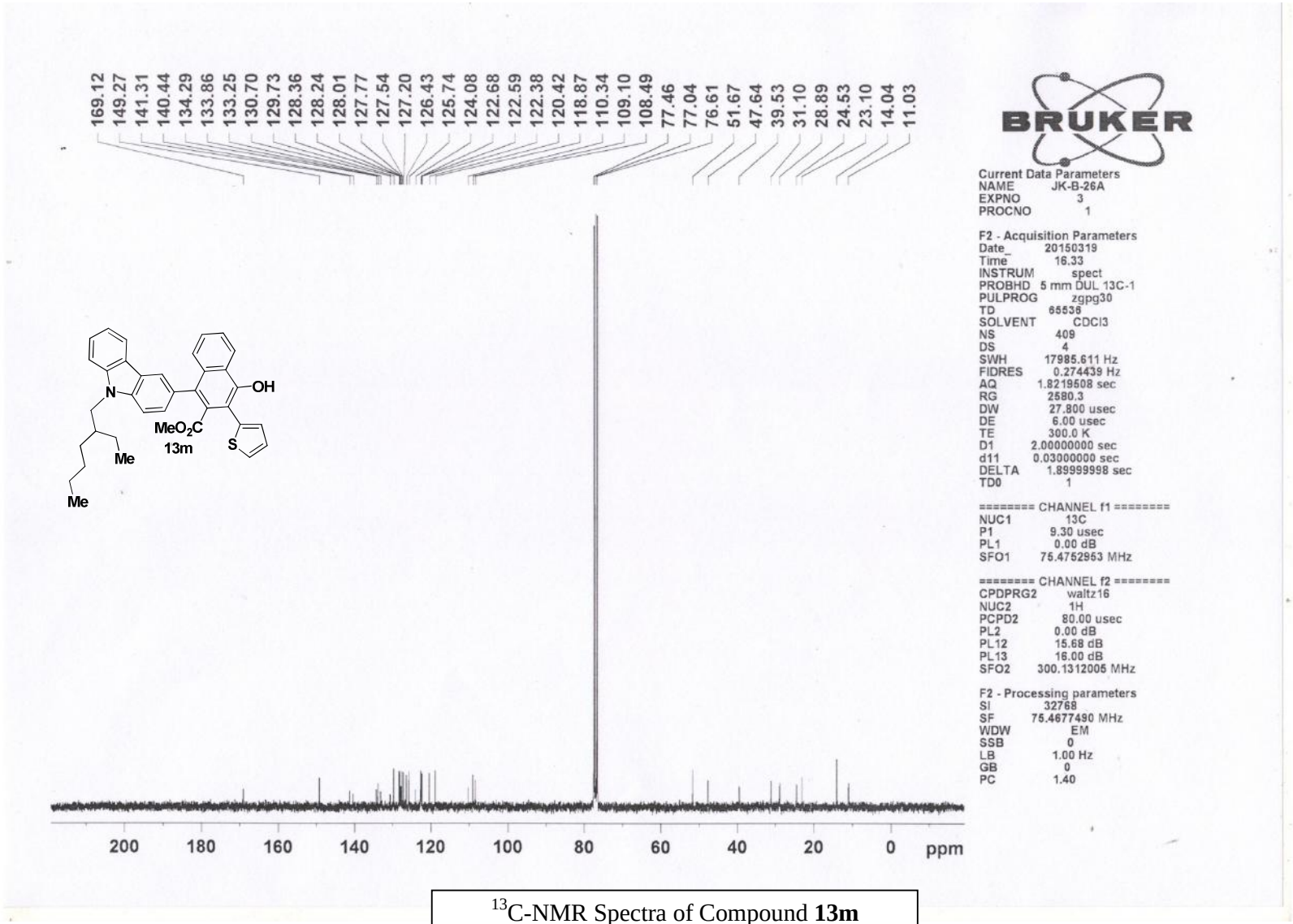
^{13}C-NMR Spectra of Compound **13l**



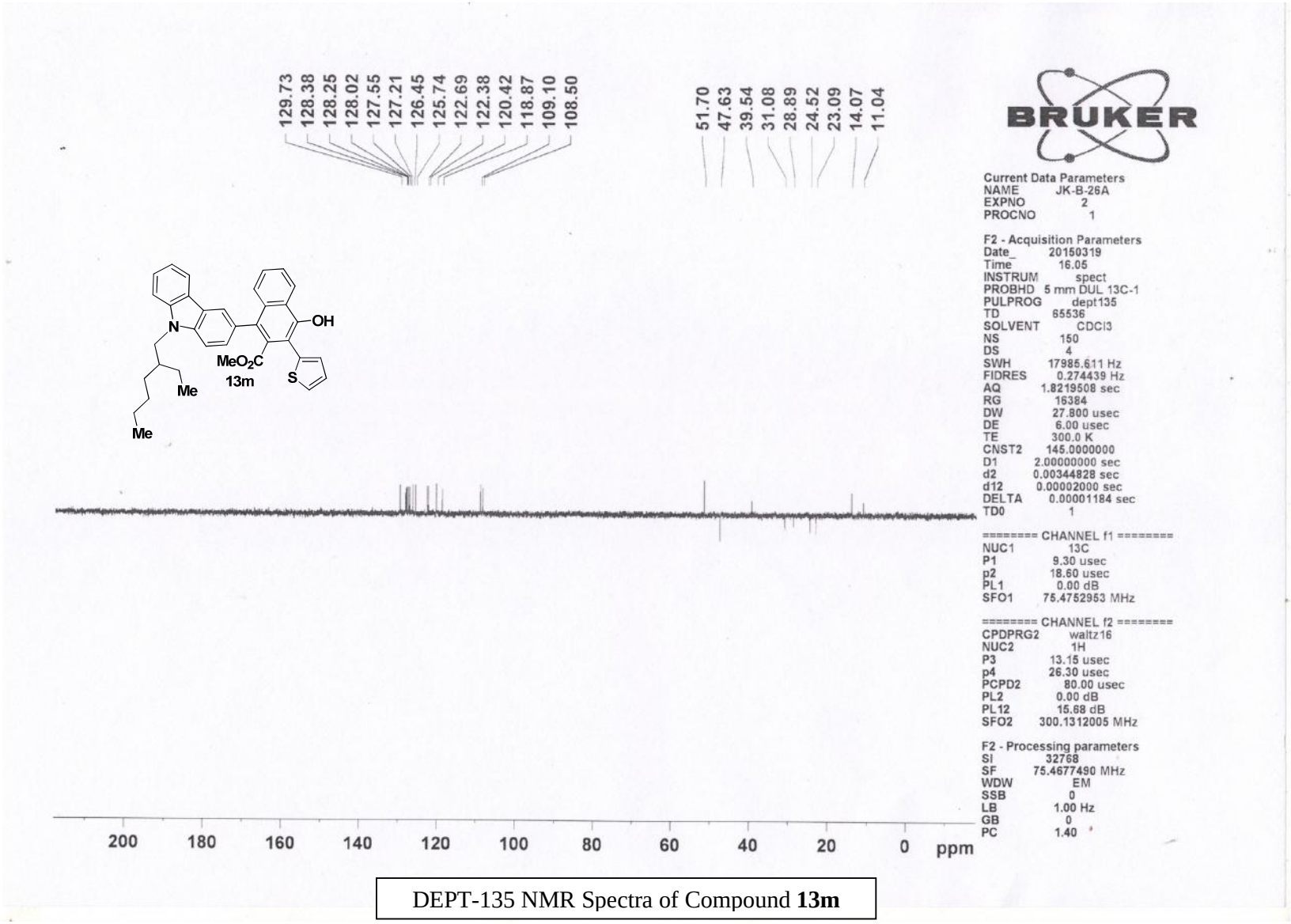
DEPT-135 NMR Spectra of Compound **13l**



^{1}H-NMR Spectra of Compound **13m**



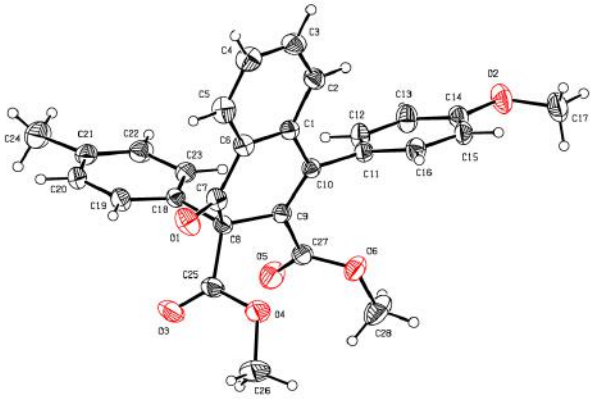
^{13}C-NMR Spectra of Compound **13m**



DEPT-135 NMR Spectra of Compound **13m**

Crystallograpic data of β-keto-ester **3e**:

CCDC Number is **997379**.



Computing details

Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Computer programs: *SHELXS97* (Sheldrick, 1990), *SHELXL97* (Sheldrick, 1997).

Dimethyl-4-(4-methoxyphenyl)-1-oxo-2-*p*-tolyl-1,2-dihydronaphthalene-2,3-dicarboxylate (3e)

Experimental

Crystal data

$C_{28}H_{24}O_6$	V = 1170.06 (6) Å^3
M_r = 456.47	Z = 2
a = 7.5256 (2) Å	F(000) = 480
b = 10.2095 (3) Å	D_x = 1.296 Mg m^{-3}
c = 15.6299 (4) Å	Mo $K\alpha$ radiation, λ = 0.71073 Å
α = 93.990 (1)°	μ = 0.09 mm^{-1}
β = 94.679 (1)°	T = 293 K
γ = 101.089 (2)°	

Data collection

Radiation source: fine-focus sealed tube	R_{int} = 0.027
Graphite monochromator	θ_{max} = 25.0°, θ_{min} = 2.3°
22413 measured reflections	h = −8→8
4119 independent reflections	k = −12→12
3346 reflections with $I > 2\sigma(I)$	l = −18→18

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.040$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.111$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.2906P]$ where $P = (F_o^2 + 2F_c^2)/3$
4119 reflections	$(\Delta/\sigma)_{max} = 0.004$
311 parameters	$\Delta\rho_{max} = 0.19$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Refinement
Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	X	*Y*	*Z*	U_{iso}*/U_{eq}
C1	1.04016 (19)	0.52685 (14)	0.34053 (9)	0.0385 (3)
C2	1.0229 (2)	0.63513 (15)	0.39596 (10)	0.0478 (4)
H2	0.9373	0.6863	0.3807	0.057*
C3	1.1303 (2)	0.66814 (17)	0.47312 (11)	0.0550 (4)
H3	1.1160	0.7407	0.5095	0.066*
C4	1.2578 (2)	0.59471 (18)	0.49645 (11)	0.0587 (5)
H4	1.3297	0.6170	0.5486	0.070*
C5	1.2791 (2)	0.48799 (18)	0.44251 (11)	0.0550 (4)
H5	1.3667	0.4387	0.4581	0.066*
C6	1.17134 (19)	0.45323 (14)	0.36521 (10)	0.0417 (3)

C7	1.1958 (2)	0.33854 (15)	0.30791 (10)	0.0446 (4)
C8	1.0303 (2)	0.27105 (14)	0.24589 (9)	0.0397 (3)
C9	0.92937 (19)	0.37508 (14)	0.21154 (9)	0.0385 (3)
C10	0.92853 (19)	0.49101 (14)	0.25739 (9)	0.0371 (3)
C11	0.8042 (2)	0.58099 (14)	0.23090 (9)	0.0401 (3)
C12	0.8649 (2)	0.70937 (15)	0.20884 (10)	0.0479 (4)
H12	0.9892	0.7416	0.2088	0.058*
C13	0.7446 (3)	0.79111 (15)	0.18672 (10)	0.0521 (4)
H13	0.7878	0.8768	0.1710	0.063*
C14	0.5606 (2)	0.74492 (16)	0.18809 (10)	0.0509 (4)
C15	0.4985 (2)	0.61764 (18)	0.21056 (12)	0.0594 (5)
H15	0.3743	0.5861	0.2118	0.071*
C16	0.6190 (2)	0.53671 (16)	0.23124 (11)	0.0522 (4)
H16	0.5749	0.4504	0.2458	0.063*
C17	0.8254 (2)	0.33536 (15)	0.12560 (10)	0.0457 (4)
C18	0.7520 (3)	0.4012 (2)	−0.01137 (12)	0.0833 (7)
H18A	0.7824	0.3208	−0.0363	0.125*
H18B	0.7902	0.4735	−0.0461	0.125*
H18C	0.6228	0.3879	−0.0088	0.125*
C19	1.0997 (2)	0.19417 (17)	0.17186 (11)	0.0523 (4)
C20	1.2394 (4)	0.2216 (3)	0.04350 (16)	0.1075 (10)
H20A	1.3518	0.1943	0.0597	0.161*
H20B	1.2588	0.2863	0.0018	0.161*
H20C	1.1505	0.1449	0.0190	0.161*
C21	0.4758 (4)	0.9350 (2)	0.12847 (14)	0.0822 (7)
H21A	0.5676	0.9976	0.1647	0.123*
H21B	0.3702	0.9736	0.1181	0.123*
H21C	0.5219	0.9148	0.0746	0.123*
C22	0.9152 (2)	0.17131 (13)	0.29922 (9)	0.0383 (3)
C23	0.7324 (2)	0.16747 (15)	0.30518 (9)	0.0435 (4)
H23	0.6760	0.2297	0.2789	0.052*
C24	0.6325 (2)	0.07153 (17)	0.35007 (11)	0.0536 (4)
H24	0.5091	0.0698	0.3528	0.064*
C25	0.7110 (3)	−0.02137 (16)	0.39079 (11)	0.0569 (5)
C26	0.8952 (3)	−0.01318 (16)	0.38729 (11)	0.0575 (5)
H26	0.9528	−0.0724	0.4162	0.069*
C27	0.9956 (2)	0.08046 (15)	0.34213 (10)	0.0494 (4)
H27	1.1194	0.0828	0.3404	0.059*
C28	0.6003 (4)	−0.1312 (2)	0.43490 (16)	0.0927 (8)
H28A	0.4822	−0.1116	0.4415	0.139*
H28B	0.6608	−0.1372	0.4906	0.139*

H28C	0.5874	−0.2150	0.4007	0.139*
O1	1.33429 (15)	0.29405 (12)	0.31246 (9)	0.0629 (3)
O2	1.0894 (2)	0.07656 (13)	0.16239 (9)	0.0765 (4)
O3	1.17487 (19)	0.28042 (13)	0.11870 (8)	0.0680 (4)
O4	0.7426 (2)	0.22389 (12)	0.10391 (8)	0.0720 (4)
O5	0.84266 (17)	0.43348 (11)	0.07425 (7)	0.0588 (3)
O6	0.42858 (19)	0.81619 (13)	0.16963 (9)	0.0736 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0357 (8)	0.0370 (7)	0.0416 (8)	0.0024 (6)	0.0060 (6)	0.0053 (6)
C2	0.0525 (9)	0.0418 (8)	0.0484 (9)	0.0102 (7)	0.0033 (7)	0.0000 (7)
C3	0.0607 (11)	0.0499 (9)	0.0493 (9)	0.0035 (8)	0.0019 (8)	−0.0061 (7)
C4	0.0537 (10)	0.0613 (11)	0.0525 (10)	−0.0007 (8)	−0.0102 (8)	−0.0034 (8)
C5	0.0391 (9)	0.0574 (10)	0.0646 (11)	0.0051 (7)	−0.0072 (8)	0.0061 (8)
C6	0.0318 (8)	0.0415 (8)	0.0495 (8)	0.0014 (6)	0.0035 (6)	0.0044 (6)
C7	0.0355 (8)	0.0419 (8)	0.0577 (9)	0.0070 (7)	0.0095 (7)	0.0103 (7)
C8	0.0398 (8)	0.0357 (7)	0.0450 (8)	0.0086 (6)	0.0099 (6)	0.0031 (6)
C9	0.0377 (8)	0.0368 (7)	0.0407 (7)	0.0038 (6)	0.0075 (6)	0.0057 (6)
C10	0.0359 (8)	0.0346 (7)	0.0404 (7)	0.0035 (6)	0.0073 (6)	0.0064 (6)
C11	0.0462 (9)	0.0358 (7)	0.0380 (7)	0.0076 (6)	0.0027 (6)	0.0035 (6)
C12	0.0503 (9)	0.0400 (8)	0.0504 (9)	0.0023 (7)	0.0011 (7)	0.0060 (7)
C13	0.0724 (12)	0.0339 (8)	0.0481 (9)	0.0083 (8)	−0.0030 (8)	0.0071 (7)
C14	0.0622 (11)	0.0471 (9)	0.0459 (8)	0.0219 (8)	−0.0059 (7)	0.0024 (7)
C15	0.0471 (10)	0.0586 (11)	0.0754 (12)	0.0143 (8)	0.0035 (8)	0.0188 (9)
C16	0.0472 (10)	0.0434 (9)	0.0674 (10)	0.0084 (7)	0.0058 (8)	0.0178 (8)
C17	0.0515 (9)	0.0404 (8)	0.0432 (8)	0.0041 (7)	0.0068 (7)	0.0026 (7)
C18	0.1029 (17)	0.0871 (15)	0.0472 (10)	−0.0074 (13)	−0.0152 (10)	0.0165 (10)
C19	0.0560 (10)	0.0487 (10)	0.0569 (10)	0.0157 (8)	0.0199 (8)	0.0045 (8)
C20	0.153 (3)	0.1032 (18)	0.0904 (17)	0.0530 (18)	0.0810 (18)	0.0205 (14)
C21	0.1208 (19)	0.0549 (11)	0.0772 (14)	0.0400 (12)	−0.0099 (13)	0.0101 (10)
C22	0.0435 (8)	0.0317 (7)	0.0391 (7)	0.0057 (6)	0.0063 (6)	0.0000 (6)
C23	0.0445 (9)	0.0430 (8)	0.0434 (8)	0.0083 (7)	0.0073 (6)	0.0042 (6)
C24	0.0519 (10)	0.0529 (10)	0.0541 (9)	0.0010 (8)	0.0181 (8)	0.0030 (8)
C25	0.0831 (13)	0.0392 (9)	0.0477 (9)	0.0033 (8)	0.0232 (9)	0.0035 (7)
C26	0.0863 (14)	0.0385 (8)	0.0514 (9)	0.0189 (9)	0.0104 (9)	0.0084 (7)
C27	0.0538 (10)	0.0401 (8)	0.0563 (9)	0.0140 (7)	0.0066 (7)	0.0046 (7)
C28	0.129 (2)	0.0605 (12)	0.0908 (16)	0.0013 (13)	0.0515 (15)	0.0203 (11)
O1	0.0382 (6)	0.0576 (7)	0.0956 (9)	0.0162 (5)	0.0070 (6)	0.0054 (6)
O2	0.1068 (11)	0.0500 (8)	0.0825 (9)	0.0275 (7)	0.0436 (8)	0.0008 (6)

O3	0.0830 (9)	0.0626 (8)	0.0690 (8)	0.0226 (7)	0.0432 (7)	0.0144 (6)
O4	0.0977 (10)	0.0488 (7)	0.0551 (7)	−0.0138 (7)	−0.0069 (7)	0.0012 (6)
O5	0.0773 (8)	0.0510 (7)	0.0415 (6)	−0.0017 (6)	−0.0036 (5)	0.0095 (5)
O6	0.0803 (10)	0.0613 (8)	0.0852 (9)	0.0335 (7)	−0.0090 (7)	0.0141 (7)

Geometric parameters (Å, º)

C1—C2	1.389 (2)	C17—O4	1.1990 (19)
C1—C6	1.397 (2)	C17—O5	1.3204 (19)
C1—C10	1.472 (2)	C18—O5	1.438 (2)
C2—C3	1.379 (2)	C18—H18A	0.9600
C2—H2	0.9300	C18—H18B	0.9600
C3—C4	1.369 (2)	C18—H18C	0.9600
C3—H3	0.9300	C19—O2	1.186 (2)
C4—C5	1.373 (2)	C19—O3	1.331 (2)
C4—H4	0.9300	C20—O3	1.444 (2)
C5—C6	1.383 (2)	C20—H20A	0.9600
C5—H5	0.9300	C20—H20B	0.9600
C6—C7	1.473 (2)	C20—H20C	0.9600
C7—O1	1.2134 (18)	C21—O6	1.412 (3)
C7—C8	1.531 (2)	C21—H21A	0.9600
C8—C9	1.522 (2)	C21—H21B	0.9600
C8—C19	1.535 (2)	C21—H21C	0.9600
C8—C22	1.542 (2)	C22—C23	1.380 (2)
C9—C10	1.342 (2)	C22—C27	1.383 (2)
C9—C17	1.487 (2)	C23—C24	1.384 (2)
C10—C11	1.487 (2)	C23—H23	0.9300
C11—C12	1.380 (2)	C24—C25	1.375 (3)
C11—C16	1.381 (2)	C24—H24	0.9300
C12—C13	1.384 (2)	C25—C26	1.379 (3)
C12—H12	0.9300	C25—C28	1.506 (3)
C13—C14	1.377 (3)	C26—C27	1.373 (2)
C13—H13	0.9300	C26—H26	0.9300
C14—O6	1.364 (2)	C27—H27	0.9300
C14—C15	1.374 (2)	C28—H28A	0.9600
C15—C16	1.374 (2)	C28—H28B	0.9600
C15—H15	0.9300	C28—H28C	0.9600
C16—H16	0.9300		
C2—C1—C6	117.75 (13)	O4—C17—O5	123.66 (15)
C2—C1—C10	122.02 (13)	O4—C17—C9	123.29 (14)
C6—C1—C10	120.21 (13)	O5—C17—C9	112.94 (13)

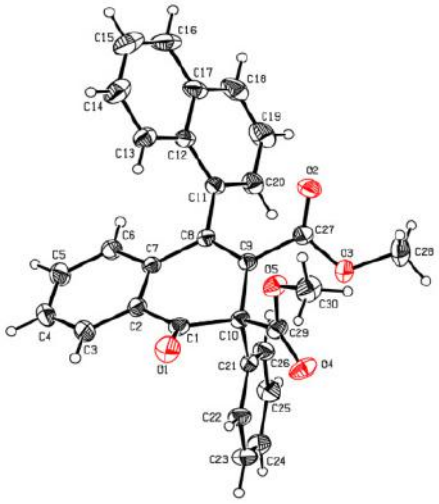
C3—C2—C1	121.18 (15)	O5—C18—H18A	109.5
C3—C2—H2	119.4	O5—C18—H18B	109.5
C1—C2—H2	119.4	H18A—C18—H18B	109.5
C4—C3—C2	120.32 (15)	O5—C18—H18C	109.5
C4—C3—H3	119.8	H18A—C18—H18C	109.5
C2—C3—H3	119.8	H18B—C18—H18C	109.5
C3—C4—C5	119.76 (15)	O2—C19—O3	124.44 (15)
C3—C4—H4	120.1	O2—C19—C8	126.23 (15)
C5—C4—H4	120.1	O3—C19—C8	109.33 (13)
C4—C5—C6	120.48 (16)	O3—C20—H20A	109.5
C4—C5—H5	119.8	O3—C20—H20B	109.5
C6—C5—H5	119.8	H20A—C20—H20B	109.5
C5—C6—C1	120.50 (14)	O3—C20—H20C	109.5
C5—C6—C7	119.92 (14)	H20A—C20—H20C	109.5
C1—C6—C7	119.58 (13)	H20B—C20—H20C	109.5
O1—C7—C6	122.60 (15)	O6—C21—H21A	109.5
O1—C7—C8	121.18 (14)	O6—C21—H21B	109.5
C6—C7—C8	116.07 (12)	H21A—C21—H21B	109.5
C9—C8—C7	110.59 (11)	O6—C21—H21C	109.5
C9—C8—C19	110.62 (12)	H21A—C21—H21C	109.5
C7—C8—C19	107.56 (12)	H21B—C21—H21C	109.5
C9—C8—C22	113.30 (12)	C23—C22—C27	118.00 (14)
C7—C8—C22	104.74 (11)	C23—C22—C8	122.47 (13)
C19—C8—C22	109.74 (12)	C27—C22—C8	119.53 (13)
C10—C9—C17	122.91 (13)	C22—C23—C24	120.40 (15)
C10—C9—C8	122.15 (13)	C22—C23—H23	119.8
C17—C9—C8	114.88 (12)	C24—C23—H23	119.8
C9—C10—C1	120.24 (13)	C25—C24—C23	121.70 (16)
C9—C10—C11	121.87 (13)	C25—C24—H24	119.2
C1—C10—C11	117.65 (12)	C23—C24—H24	119.2
C12—C11—C16	117.89 (14)	C24—C25—C26	117.36 (15)
C12—C11—C10	123.18 (14)	C24—C25—C28	121.8 (2)
C16—C11—C10	118.88 (13)	C26—C25—C28	120.81 (19)
C11—C12—C13	121.32 (15)	C27—C26—C25	121.52 (16)
C11—C12—H12	119.3	C27—C26—H26	119.2
C13—C12—H12	119.3	C25—C26—H26	119.2
C14—C13—C12	119.67 (15)	C26—C27—C22	120.94 (16)
C14—C13—H13	120.2	C26—C27—H27	119.5
C12—C13—H13	120.2	C22—C27—H27	119.5
O6—C14—C15	115.00 (16)	C25—C28—H28A	109.5
O6—C14—C13	125.43 (16)	C25—C28—H28B	109.5

C15—C14—C13	119.57 (15)	H28A—C28—H28B	109.5
C16—C15—C14	120.27 (17)	C25—C28—H28C	109.5
C16—C15—H15	119.9	H28A—C28—H28C	109.5
C14—C15—H15	119.9	H28B—C28—H28C	109.5
C15—C16—C11	121.27 (15)	C19—O3—C20	115.56 (15)
C15—C16—H16	119.4	C17—O5—C18	116.25 (13)
C11—C16—H16	119.4	C14—O6—C21	118.23 (17)
C6—C1—C2—C3	−0.6 (2)	C11—C12—C13—C14	1.2 (2)
C10—C1—C2—C3	−179.19 (14)	C12—C13—C14—O6	178.48 (15)
C1—C2—C3—C4	0.4 (3)	C12—C13—C14—C15	−0.7 (2)
C2—C3—C4—C5	0.3 (3)	O6—C14—C15—C16	−179.47 (16)
C3—C4—C5—C6	−0.7 (3)	C13—C14—C15—C16	−0.2 (3)
C4—C5—C6—C1	0.5 (2)	C14—C15—C16—C11	0.7 (3)
C4—C5—C6—C7	179.96 (15)	C12—C11—C16—C15	−0.2 (2)
C2—C1—C6—C5	0.1 (2)	C10—C11—C16—C15	177.47 (15)
C10—C1—C6—C5	178.74 (14)	C10—C9—C17—O4	−138.46 (18)
C2—C1—C6—C7	−179.29 (13)	C8—C9—C17—O4	38.7 (2)
C10—C1—C6—C7	−0.7 (2)	C10—C9—C17—O5	45.2 (2)
C5—C6—C7—O1	−20.2 (2)	C8—C9—C17—O5	−137.56 (14)
C1—C6—C7—O1	159.22 (15)	C9—C8—C19—O2	−135.20 (19)
C5—C6—C7—C8	155.47 (14)	C7—C8—C19—O2	103.9 (2)
C1—C6—C7—C8	−25.1 (2)	C22—C8—C19—O2	−9.5 (2)
O1—C7—C8—C9	−146.10 (15)	C9—C8—C19—O3	44.87 (18)
C6—C7—C8—C9	38.16 (17)	C7—C8—C19—O3	−76.01 (17)
O1—C7—C8—C19	−25.2 (2)	C22—C8—C19—O3	170.60 (13)
C6—C7—C8—C19	159.05 (13)	C9—C8—C22—C23	7.84 (19)
O1—C7—C8—C22	91.51 (17)	C7—C8—C22—C23	128.45 (14)
C6—C7—C8—C22	−84.24 (15)	C19—C8—C22—C23	−116.35 (15)
C7—C8—C9—C10	−29.46 (18)	C9—C8—C22—C27	−173.06 (12)
C19—C8—C9—C10	−148.52 (14)	C7—C8—C22—C27	−52.45 (16)
C22—C8—C9—C10	87.78 (16)	C19—C8—C22—C27	62.75 (17)
C7—C8—C9—C17	153.32 (12)	C27—C22—C23—C24	−2.6 (2)
C19—C8—C9—C17	34.26 (17)	C8—C22—C23—C24	176.56 (13)
C22—C8—C9—C17	−89.44 (14)	C22—C23—C24—C25	0.8 (2)
C17—C9—C10—C1	−177.56 (13)	C23—C24—C25—C26	1.7 (2)
C8—C9—C10—C1	5.4 (2)	C23—C24—C25—C28	−176.07 (17)
C17—C9—C10—C11	8.2 (2)	C24—C25—C26—C27	−2.5 (2)
C8—C9—C10—C11	−168.83 (13)	C28—C25—C26—C27	175.31 (17)
C2—C1—C10—C9	−170.09 (14)	C25—C26—C27—C22	0.8 (2)
C6—C1—C10—C9	11.4 (2)	C23—C22—C27—C26	1.8 (2)
C2—C1—C10—C11	4.4 (2)	C8—C22—C27—C26	−177.35 (14)

C6—C1—C10—C11	−174.13 (12)	O2—C19—O3—C20	2.7 (3)
C9—C10—C11—C12	−117.13 (17)	C8—C19—O3—C20	−177.38 (18)
C1—C10—C11—C12	68.45 (18)	O4—C17—O5—C18	1.3 (3)
C9—C10—C11—C16	65.36 (19)	C9—C17—O5—C18	177.56 (16)
C1—C10—C11—C16	−109.06 (16)	C15—C14—O6—C21	−166.54 (17)
C16—C11—C12—C13	−0.7 (2)	C13—C14—O6—C21	14.3 (2)
C10—C11—C12—C13	−178.28 (14)		

Crystallograpic data of *β*-keto-ester **9d**:

CCDC Number is **1450132**.



Computing details

Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997).

Dimethyl-4-oxo-3-phenyl-3,4-dihydro-1,1'-binaphthyl-2,3-dicarboxylate (9d)

Experimental

Crystal data

$C_{30}H_{22}O_5$	$F(000)$ = 968
M_r = 462.48	D_x = 1.308 Mg m^{-3}
Monoclinic, $P2/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 4122 reflections
a = 16.2811 (9) Å	θ = 2.5–25.0°
b = 10.0247 (5) Å	μ = 0.09 mm^{-1}

c = 15.5040 (9) Å	T = 296 K
β = 111.902 (2)°	Block, colourless
V = 2347.8 (2) Å^3	0.30 × 0.25 × 0.25 mm
Z = 4	

Data collection

Kappa ApexII CCD Diffractometer	4122 independent reflections
Radiation source: fine-focus sealed tube	3262 reflections with $I > 2\sigma(I)$
Graphite monochromator	R_{int} = 0.042
ω & φ scans	θ_{max} = 25.0°, θ_{min} = 2.5°
Absorption correction: multi-scan *SADABS* (Bruker, 2008)	h = −19→19
T_{min} = 0.974, T_{max} = 0.978	k = −11→11
25692 measured reflections	l = −18→18

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.039	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.119	H-atom parameters constrained
S = 1.04	$w = 1/[\sigma^2(F_o^2) + (0.0706P)^2 + 0.317P]$ where $P = (F_o^2 + 2F_c^2)/3$
4122 reflections	$(\Delta/\sigma)_{max}$ = 0.002
318 parameters	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Special details

Refinement

Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	Y	z	U_{iso}*/U_{eq}
C1	0.16810 (10)	0.79300 (15)	0.08703 (10)	0.0381 (4)
C2	0.15819 (10)	0.64702 (14)	0.07613 (10)	0.0364 (3)
C3	0.10938 (11)	0.59202 (17)	−0.00964 (11)	0.0481 (4)

H3	0.0855	0.6469	−0.0614	0.058*
C4	0.09604 (13)	0.45625 (18)	−0.01856 (12)	0.0551 (5)
H4	0.0629	0.4193	−0.0761	0.066*
C5	0.13226 (12)	0.37531 (17)	0.05851 (12)	0.0518 (4)
H5	0.1224	0.2838	0.0529	0.062*
C6	0.18295 (11)	0.42900 (15)	0.14373 (11)	0.0409 (4)
H6	0.2079	0.3730	0.1947	0.049*
C7	0.19714 (9)	0.56571 (14)	0.15443 (10)	0.0329 (3)
C8	0.25385 (9)	0.62674 (13)	0.24366 (9)	0.0314 (3)
C9	0.24868 (9)	0.75887 (14)	0.25757 (10)	0.0325 (3)
C10	0.17985 (9)	0.84532 (14)	0.18459 (10)	0.0342 (3)
C11	0.31149 (10)	0.53719 (14)	0.31924 (10)	0.0351 (3)
C12	0.38377 (10)	0.46701 (14)	0.30968 (11)	0.0385 (4)
C13	0.40609 (11)	0.47718 (16)	0.23040 (13)	0.0474 (4)
H13	0.3725	0.5317	0.1814	0.057*
C14	0.47591 (13)	0.40870 (19)	0.22422 (17)	0.0665 (6)
H14	0.4887	0.4152	0.1707	0.080*
C15	0.52846 (14)	0.3286 (2)	0.2979 (2)	0.0784 (7)
H15	0.5764	0.2830	0.2934	0.094*
C16	0.51020 (13)	0.31711 (19)	0.37514 (19)	0.0726 (7)
H16	0.5461	0.2639	0.4237	0.087*
C17	0.43703 (11)	0.38464 (16)	0.38425 (13)	0.0513 (4)
C18	0.41625 (14)	0.37321 (19)	0.46430 (14)	0.0659 (6)
H18	0.4509	0.3193	0.5132	0.079*
C19	0.34646 (15)	0.4397 (2)	0.47101 (13)	0.0636 (5)
H19	0.3333	0.4307	0.5242	0.076*
C20	0.29386 (12)	0.52230 (16)	0.39806 (11)	0.0459 (4)
H20	0.2462	0.5675	0.4036	0.055*
C21	0.08688 (9)	0.83404 (14)	0.18891 (10)	0.0349 (3)
C22	0.01590 (11)	0.89851 (16)	0.12212 (12)	0.0467 (4)
H22	0.0255	0.9502	0.0770	0.056*
C23	−0.06846 (11)	0.88684 (18)	0.12188 (13)	0.0535 (4)
H23	−0.1151	0.9318	0.0773	0.064*
C24	−0.08400 (11)	0.80933 (19)	0.18702 (12)	0.0530 (4)
H24	−0.1411	0.8008	0.1864	0.064*
C25	−0.01457 (11)	0.74406 (18)	0.25348 (12)	0.0505 (4)
H25	−0.0248	0.6914	0.2978	0.061*
C26	0.07010 (10)	0.75662 (16)	0.25435 (10)	0.0412 (4)
H26	0.1166	0.7124	0.2996	0.049*
C27	0.30751 (11)	0.82461 (15)	0.34507 (10)	0.0385 (4)
C28	0.31288 (16)	0.9993 (2)	0.44990 (16)	0.0809 (7)
H28A	0.3620	1.0386	0.4391	0.121*
H28B	0.3345	0.9448	0.5047	0.121*

H28C	0.2758	1.0685	0.4583	0.121*
C29	0.21036 (11)	0.99078 (15)	0.18912 (11)	0.0402 (4)
C30	0.33376 (13)	1.12616 (17)	0.20682 (16)	0.0634 (5)
H30A	0.3000	1.1772	0.1528	0.095*
H30B	0.3938	1.1185	0.2106	0.095*
H30C	0.3328	1.1701	0.2615	0.095*
O1	0.16230 (10)	0.86806 (12)	0.02391 (8)	0.0600 (4)
O2	0.38451 (8)	0.80178 (12)	0.38594 (8)	0.0537 (3)
O3	0.26227 (8)	0.91803 (12)	0.37103 (8)	0.0529 (3)
O4	0.16556 (8)	1.08759 (11)	0.18169 (10)	0.0598 (4)
O5	0.29551 (7)	0.99458 (10)	0.20016 (8)	0.0473 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0417 (9)	0.0377 (8)	0.0356 (8)	0.0073 (7)	0.0153 (7)	0.0078 (7)
C2	0.0399 (8)	0.0359 (8)	0.0343 (8)	0.0049 (6)	0.0150 (7)	0.0018 (6)
C3	0.0555 (10)	0.0518 (10)	0.0328 (8)	0.0083 (8)	0.0118 (8)	0.0031 (7)
C4	0.0598 (11)	0.0539 (10)	0.0411 (10)	−0.0014 (9)	0.0067 (8)	−0.0127 (8)
C5	0.0575 (11)	0.0372 (9)	0.0530 (10)	−0.0052 (8)	0.0115 (9)	−0.0079 (8)
C6	0.0449 (9)	0.0340 (8)	0.0417 (9)	0.0027 (7)	0.0136 (7)	0.0023 (7)
C7	0.0337 (8)	0.0323 (7)	0.0331 (8)	0.0044 (6)	0.0129 (6)	0.0016 (6)
C8	0.0311 (7)	0.0316 (7)	0.0329 (7)	0.0031 (6)	0.0136 (6)	0.0037 (6)
C9	0.0326 (8)	0.0327 (7)	0.0332 (8)	0.0023 (6)	0.0133 (6)	0.0031 (6)
C10	0.0371 (8)	0.0284 (7)	0.0372 (8)	0.0049 (6)	0.0140 (7)	0.0040 (6)
C11	0.0357 (8)	0.0286 (7)	0.0364 (8)	−0.0007 (6)	0.0082 (6)	0.0024 (6)
C12	0.0327 (8)	0.0261 (7)	0.0479 (9)	−0.0012 (6)	0.0050 (7)	−0.0009 (6)
C13	0.0415 (9)	0.0391 (9)	0.0622 (11)	0.0002 (7)	0.0201 (8)	−0.0015 (8)
C14	0.0533 (11)	0.0512 (11)	0.1047 (17)	−0.0016 (9)	0.0408 (12)	−0.0132 (11)
C15	0.0455 (12)	0.0478 (12)	0.139 (2)	0.0058 (9)	0.0312 (14)	−0.0103 (13)
C16	0.0403 (11)	0.0400 (10)	0.1085 (18)	0.0096 (8)	−0.0057 (11)	0.0046 (11)
C17	0.0406 (9)	0.0313 (8)	0.0611 (11)	−0.0005 (7)	−0.0053 (8)	0.0022 (8)
C18	0.0685 (13)	0.0484 (11)	0.0542 (12)	0.0012 (10)	−0.0077 (10)	0.0175 (9)
C19	0.0792 (14)	0.0620 (12)	0.0410 (10)	−0.0064 (11)	0.0125 (9)	0.0145 (9)
C20	0.0527 (10)	0.0454 (9)	0.0375 (9)	−0.0008 (8)	0.0144 (8)	0.0053 (7)
C21	0.0377 (8)	0.0307 (7)	0.0349 (8)	0.0035 (6)	0.0120 (6)	−0.0024 (6)
C22	0.0470 (10)	0.0438 (9)	0.0464 (9)	0.0113 (7)	0.0142 (8)	0.0118 (7)
C23	0.0401 (9)	0.0548 (10)	0.0578 (11)	0.0141 (8)	0.0093 (8)	0.0059 (9)
C24	0.0371 (9)	0.0634 (11)	0.0585 (11)	0.0023 (8)	0.0179 (8)	−0.0074 (9)
C25	0.0468 (10)	0.0625 (11)	0.0457 (10)	−0.0034 (8)	0.0215 (8)	0.0029 (8)
C26	0.0387 (9)	0.0479 (9)	0.0351 (8)	0.0044 (7)	0.0116 (7)	0.0052 (7)
C27	0.0418 (9)	0.0338 (8)	0.0395 (8)	0.0000 (7)	0.0146 (7)	0.0019 (6)
C28	0.0880 (16)	0.0793 (15)	0.0690 (14)	−0.0087 (12)	0.0218 (12)	−0.0405 (12)
C29	0.0465 (9)	0.0314 (8)	0.0451 (9)	0.0049 (7)	0.0199 (7)	0.0042 (7)

C30	0.0635 (12)	0.0386 (9)	0.0891 (15)	−0.0128 (9)	0.0297 (11)	−0.0049 (9)
O1	0.0932 (10)	0.0454 (7)	0.0457 (7)	0.0076 (6)	0.0308 (7)	0.0164 (6)
O2	0.0407 (7)	0.0526 (7)	0.0544 (7)	0.0012 (5)	0.0025 (6)	−0.0041 (6)
O3	0.0536 (7)	0.0518 (7)	0.0510 (7)	0.0005 (5)	0.0168 (6)	−0.0182 (6)
O4	0.0595 (8)	0.0306 (6)	0.0944 (10)	0.0112 (5)	0.0346 (7)	0.0078 (6)
O5	0.0446 (7)	0.0303 (5)	0.0693 (8)	−0.0018 (5)	0.0241 (6)	0.0022 (5)

Geometric parameters (Å, º)

C1—O1	1.2100 (17)	C16—H16	0.9300
C1—C2	1.475 (2)	C17—C18	1.408 (3)
C1—C10	1.544 (2)	C18—C19	1.355 (3)
C2—C3	1.384 (2)	C18—H18	0.9300
C2—C7	1.402 (2)	C19—C20	1.405 (3)
C3—C4	1.377 (2)	C19—H19	0.9300
C3—H3	0.9300	C20—H20	0.9300
C4—C5	1.381 (2)	C21—C26	1.383 (2)
C4—H4	0.9300	C21—C22	1.390 (2)
C5—C6	1.379 (2)	C22—C23	1.377 (2)
C5—H5	0.9300	C22—H22	0.9300
C6—C7	1.389 (2)	C23—C24	1.370 (3)
C6—H6	0.9300	C23—H23	0.9300
C7—C8	1.479 (2)	C24—C25	1.378 (2)
C8—C9	1.3496 (19)	C24—H24	0.9300
C8—C11	1.4964 (19)	C25—C26	1.379 (2)
C9—C27	1.491 (2)	C25—H25	0.9300
C9—C10	1.5301 (19)	C26—H26	0.9300
C10—C29	1.534 (2)	C27—O2	1.1970 (18)
C10—C21	1.544 (2)	C27—O3	1.3427 (19)
C11—C20	1.364 (2)	C28—O3	1.444 (2)
C11—C12	1.425 (2)	C28—H28A	0.9600
C12—C13	1.409 (2)	C28—H28B	0.9600
C12—C17	1.422 (2)	C28—H28C	0.9600
C13—C14	1.362 (2)	C29—O4	1.1934 (18)
C13—H13	0.9300	C29—O5	1.3325 (19)
C14—C15	1.397 (3)	C30—O5	1.4458 (19)
C14—H14	0.9300	C30—H30A	0.9600
C15—C16	1.342 (3)	C30—H30B	0.9600
C15—H15	0.9300	C30—H30C	0.9600
C16—C17	1.422 (3)		
O1—C1—C2	123.51 (14)	C18—C17—C12	119.33 (16)
O1—C1—C10	121.66 (14)	C18—C17—C16	122.32 (18)
C2—C1—C10	114.68 (12)	C12—C17—C16	118.35 (19)
C3—C2—C7	120.83 (14)	C19—C18—C17	120.80 (17)

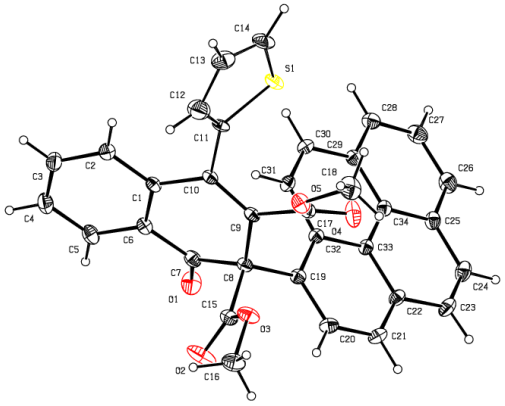
C3—C2—C1	120.29 (14)	C19—C18—H18	119.6
C7—C2—C1	118.87 (13)	C17—C18—H18	119.6
C4—C3—C2	120.18 (15)	C18—C19—C20	120.33 (18)
C4—C3—H3	119.9	C18—C19—H19	119.8
C2—C3—H3	119.9	C20—C19—H19	119.8
C3—C4—C5	119.56 (16)	C11—C20—C19	121.12 (17)
C3—C4—H4	120.2	C11—C20—H20	119.4
C5—C4—H4	120.2	C19—C20—H20	119.4
C6—C5—C4	120.60 (15)	C26—C21—C22	118.07 (14)
C6—C5—H5	119.7	C26—C21—C10	122.55 (13)
C4—C5—H5	119.7	C22—C21—C10	119.31 (13)
C5—C6—C7	120.85 (14)	C23—C22—C21	120.91 (15)
C5—C6—H6	119.6	C23—C22—H22	119.5
C7—C6—H6	119.6	C21—C22—H22	119.5
C6—C7—C2	117.95 (13)	C24—C23—C22	120.28 (16)
C6—C7—C8	122.53 (13)	C24—C23—H23	119.9
C2—C7—C8	119.49 (13)	C22—C23—H23	119.9
C9—C8—C7	120.07 (13)	C23—C24—C25	119.66 (16)
C9—C8—C11	121.46 (13)	C23—C24—H24	120.2
C7—C8—C11	118.33 (12)	C25—C24—H24	120.2
C8—C9—C27	121.47 (13)	C24—C25—C26	120.14 (16)
C8—C9—C10	120.65 (13)	C24—C25—H25	119.9
C27—C9—C10	117.85 (12)	C26—C25—H25	119.9
C9—C10—C29	111.68 (12)	C25—C26—C21	120.94 (15)
C9—C10—C1	108.73 (11)	C25—C26—H26	119.5
C29—C10—C1	106.71 (11)	C21—C26—H26	119.5
C9—C10—C21	112.69 (11)	O2—C27—O3	123.92 (14)
C29—C10—C21	111.94 (11)	O2—C27—C9	125.99 (14)
C1—C10—C21	104.62 (11)	O3—C27—C9	110.06 (13)
C20—C11—C12	119.80 (14)	O3—C28—H28A	109.5
C20—C11—C8	119.15 (13)	O3—C28—H28B	109.5
C12—C11—C8	121.04 (13)	H28A—C28—H28B	109.5
C13—C12—C17	118.27 (15)	O3—C28—H28C	109.5
C13—C12—C11	123.11 (14)	H28A—C28—H28C	109.5
C17—C12—C11	118.60 (15)	H28B—C28—H28C	109.5
C14—C13—C12	121.23 (18)	O4—C29—O5	123.93 (14)
C14—C13—H13	119.4	O4—C29—C10	126.37 (14)
C12—C13—H13	119.4	O5—C29—C10	109.68 (12)
C13—C14—C15	120.3 (2)	O5—C30—H30A	109.5
C13—C14—H14	119.8	O5—C30—H30B	109.5
C15—C14—H14	119.8	H30A—C30—H30B	109.5
C16—C15—C14	120.47 (19)	O5—C30—H30C	109.5
C16—C15—H15	119.8	H30A—C30—H30C	109.5

C14—C15—H15	119.8	H30B—C30—H30C	109.5
C15—C16—C17	121.34 (19)	C27—O3—C28	116.34 (14)
C15—C16—H16	119.3	C29—O5—C30	115.79 (13)
C17—C16—H16	119.3		
O1—C1—C2—C3	−25.2 (2)	C11—C12—C13—C14	179.78 (15)
C10—C1—C2—C3	150.51 (14)	C12—C13—C14—C15	−1.5 (3)
O1—C1—C2—C7	156.37 (15)	C13—C14—C15—C16	0.7 (3)
C10—C1—C2—C7	−27.94 (19)	C14—C15—C16—C17	0.4 (3)
C7—C2—C3—C4	2.1 (2)	C13—C12—C17—C18	179.80 (15)
C1—C2—C3—C4	−176.34 (15)	C11—C12—C17—C18	0.9 (2)
C2—C3—C4—C5	−0.5 (3)	C13—C12—C17—C16	0.2 (2)
C3—C4—C5—C6	−1.2 (3)	C11—C12—C17—C16	−178.67 (15)
C4—C5—C6—C7	1.4 (3)	C15—C16—C17—C18	179.51 (19)
C5—C6—C7—C2	0.2 (2)	C15—C16—C17—C12	−0.9 (3)
C5—C6—C7—C8	−177.57 (14)	C12—C17—C18—C19	−0.1 (3)
C3—C2—C7—C6	−1.9 (2)	C16—C17—C18—C19	179.48 (18)
C1—C2—C7—C6	176.53 (13)	C17—C18—C19—C20	−0.4 (3)
C3—C2—C7—C8	175.92 (13)	C12—C11—C20—C19	0.7 (2)
C1—C2—C7—C8	−5.6 (2)	C8—C11—C20—C19	−179.70 (15)
C6—C7—C8—C9	−163.65 (14)	C18—C19—C20—C11	0.1 (3)
C2—C7—C8—C9	18.63 (19)	C9—C10—C21—C26	1.91 (19)
C6—C7—C8—C11	12.1 (2)	C29—C10—C21—C26	−124.97 (15)
C2—C7—C8—C11	−165.60 (13)	C1—C10—C21—C26	119.85 (14)
C7—C8—C9—C27	−177.39 (12)	C9—C10—C21—C22	−174.90 (13)
C11—C8—C9—C27	7.0 (2)	C29—C10—C21—C22	58.23 (18)
C7—C8—C9—C10	4.44 (19)	C1—C10—C21—C22	−56.95 (16)
C11—C8—C9—C10	−171.20 (12)	C26—C21—C22—C23	0.8 (2)
C8—C9—C10—C29	−153.22 (13)	C10—C21—C22—C23	177.71 (15)
C27—C9—C10—C29	28.54 (17)	C21—C22—C23—C24	−1.0 (3)
C8—C9—C10—C1	−35.74 (17)	C22—C23—C24—C25	0.7 (3)
C27—C9—C10—C1	146.02 (12)	C23—C24—C25—C26	−0.1 (3)
C8—C9—C10—C21	79.77 (16)	C24—C25—C26—C21	−0.2 (3)
C27—C9—C10—C21	−98.47 (14)	C22—C21—C26—C25	−0.2 (2)
O1—C1—C10—C9	−137.75 (15)	C10—C21—C26—C25	−177.01 (14)
C2—C1—C10—C9	46.48 (16)	C8—C9—C27—O2	40.4 (2)
O1—C1—C10—C29	−17.15 (19)	C10—C9—C27—O2	−141.34 (15)
C2—C1—C10—C29	167.07 (12)	C8—C9—C27—O3	−141.53 (14)
O1—C1—C10—C21	101.63 (17)	C10—C9—C27—O3	36.70 (17)
C2—C1—C10—C21	−74.14 (15)	C9—C10—C29—O4	−139.09 (17)
C9—C8—C11—C20	63.14 (19)	C1—C10—C29—O4	102.22 (18)
C7—C8—C11—C20	−112.57 (15)	C21—C10—C29—O4	−11.7 (2)
C9—C8—C11—C12	−117.28 (15)	C9—C10—C29—O5	42.71 (16)
C7—C8—C11—C12	67.01 (18)	C1—C10—C29—O5	−75.98 (15)

C20—C11—C12—C13	179.95 (15)	C21—C10—C29—O5	170.12 (12)
C8—C11—C12—C13	0.4 (2)	O2—C27—O3—C28	3.9 (2)
C20—C11—C12—C17	−1.2 (2)	C9—C27—O3—C28	−174.18 (15)
C8—C11—C12—C17	179.19 (13)	O4—C29—O5—C30	2.5 (2)
C17—C12—C13—C14	1.0 (2)	C10—C29—O5—C30	−179.22 (14)

Crystallograpic data of *β*-keto-ester **9g**:

CCDC Number is **1438503**.



Computing details

Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Computer programs: *SHELXS97* (Sheldrick, 1990), *SHELXL97* (Sheldrick, 1997).

Dimethyl-1-oxo-4-(pyren-4-yl)-2-(thiophen-2-yl)-1,2-dihydronaphthalene-2,3-dicarboxylate (9g)

Experimental

Crystal data

$C_{34}H_{22}O_5S$	V = 2537.9 (4) Å^3
M_r = 542.58	Z = 4
a = 10.9268 (10) Å	F(000) = 1128
b = 18.9670 (14) Å	D_x = 1.420 Mg m^{-3}
c = 12.2628 (9) Å	Mo $K\alpha$ radiation, λ = 0.71073 Å
α = 90°	μ = 0.17 mm^{-1}
β = 93.030 (2)°	T = 296 K
γ = 90°	0.25 × 0.25 × 0.20 mm

Data collection

Radiation source: fine-focus sealed tube	$R_{int} = 0.031$
Graphite monochromator	$\theta_{max} = 25.0°$, $\theta_{min} = 2.0°$
21576 measured reflections	$h = -12 \rightarrow 12$
4457 independent reflections	$k = -22 \rightarrow 22$
3341 reflections with $I > 2\sigma(I)$	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.039$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.102$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 1.1902P]$ where $P = (F_o^2 + 2F_c^2)/3$
4457 reflections	$(\Delta/\sigma)_{max} = 0.035$
409 parameters	$\Delta\rho_{max} = 0.22$ e Å^{-3}
147 restraints	$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Refinement
Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	X	*Y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.96828 (18)	0.11067 (10)	0.90164 (15)	0.0320 (4)	
C2	1.0708 (2)	0.11714 (11)	0.97370 (16)	0.0415 (5)	
H2	1.1396	0.0895	0.9636	0.050*	
C3	1.0716 (2)	0.16390 (12)	1.05966 (17)	0.0477 (6)	
H3	1.1399	0.1664	1.1080	0.057*	

C4	0.9731 (2)	0.20672 (12)	1.07487 (17)	0.0498 (6)
H4	0.9738	0.2374	1.1340	0.060*
C5	0.8731 (2)	0.20401 (11)	1.00231 (17)	0.0451 (5)
H5	0.8077	0.2345	1.0107	0.054*
C6	0.86883 (19)	0.15603 (10)	0.91630 (15)	0.0351 (5)
C7	0.76264 (19)	0.15550 (11)	0.83793 (16)	0.0376 (5)
C8	0.73844 (17)	0.08695 (10)	0.77212 (15)	0.0321 (4)
C9	0.85584 (18)	0.04510 (9)	0.75499 (14)	0.0296 (4)
C10	0.96161 (18)	0.05805 (9)	0.81266 (14)	0.0297 (4)
C15	0.65896 (19)	0.04476 (11)	0.84991 (16)	0.0392 (5)
C16	0.6091 (2)	−0.06628 (13)	0.9181 (2)	0.0595 (7)
H16A	0.6237	−0.0497	0.9916	0.089*
H16B	0.6349	−0.1145	0.9135	0.089*
H16C	0.5232	−0.0629	0.8979	0.089*
C17	0.84616 (18)	−0.01356 (10)	0.67263 (16)	0.0354 (5)
C18	0.9087 (3)	−0.12959 (12)	0.6360 (2)	0.0641 (7)
H18A	0.8318	−0.1402	0.5981	0.096*
H18B	0.9387	−0.1708	0.6743	0.096*
H18C	0.9669	−0.1152	0.5843	0.096*
C19	0.66477 (17)	0.10326 (10)	0.66500 (15)	0.0320 (4)
C20	0.55002 (18)	0.07443 (11)	0.64239 (17)	0.0400 (5)
H20	0.5160	0.0457	0.6942	0.048*
C21	0.48472 (19)	0.08712 (11)	0.54525 (18)	0.0416 (5)
H21	0.4085	0.0660	0.5322	0.050*
C22	0.53065 (18)	0.13070 (10)	0.46670 (16)	0.0350 (5)
C23	0.4643 (2)	0.14590 (12)	0.36525 (18)	0.0458 (5)
H23	0.3898	0.1236	0.3492	0.055*
C24	0.5072 (2)	0.19114 (12)	0.29377 (18)	0.0471 (6)
H24	0.4606	0.2006	0.2298	0.057*
C25	0.62262 (19)	0.22575 (10)	0.31202 (16)	0.0383 (5)
C26	0.6688 (2)	0.27433 (12)	0.23984 (17)	0.0494 (6)
H26	0.6229	0.2855	0.1762	0.059*
C27	0.7806 (2)	0.30624 (12)	0.26021 (18)	0.0507 (6)
H27	0.8085	0.3390	0.2108	0.061*
C28	0.8517 (2)	0.29025 (11)	0.35287 (17)	0.0425 (5)
H28	0.9279	0.3115	0.3651	0.051*
C29	0.80935 (18)	0.24205 (10)	0.42891 (15)	0.0332 (4)
C30	0.87809 (18)	0.22445 (10)	0.52708 (16)	0.0366 (5)
H30	0.9557	0.2438	0.5398	0.044*
C31	0.83406 (18)	0.18065 (10)	0.60193 (16)	0.0346 (5)
H31	0.8818	0.1712	0.6653	0.041*

C32	0.71588 (17)	0.14808 (9)	0.58717 (15)	0.0301 (4)	
C33	0.64720 (17)	0.16243 (9)	0.48784 (15)	0.0305 (4)	
C34	0.69379 (17)	0.20966 (10)	0.40935 (15)	0.0314 (4)	
O1	0.69195 (15)	0.20459 (8)	0.82754 (13)	0.0577 (5)	
O2	0.58943 (18)	0.07161 (9)	0.90895 (16)	0.0782 (6)	
O3	0.67768 (14)	−0.02357 (7)	0.84489 (12)	0.0491 (4)	
O4	0.80019 (18)	−0.00824 (9)	0.58233 (13)	0.0650 (5)	
O5	0.89193 (14)	−0.07340 (7)	0.71283 (11)	0.0444 (4)	
S1	1.12323 (15)	0.02883 (10)	0.65482 (12)	0.0435 (4)	0.691 (3)
C11	1.0763 (11)	0.0203 (8)	0.7846 (7)	0.033 (2)	0.691 (3)
C12	1.1467 (13)	−0.0255 (7)	0.8443 (10)	0.063 (3)	0.691 (3)
H12	1.1326	−0.0365	0.9164	0.076*	0.691 (3)
C13	1.2445 (14)	−0.0553 (8)	0.7873 (9)	0.0561 (18)	0.691 (3)
H13	1.3016	−0.0877	0.8158	0.067*	0.691 (3)
C14	1.2407 (10)	−0.0290 (6)	0.6863 (9)	0.054 (2)	0.691 (3)
H14	1.2979	−0.0413	0.6361	0.064*	0.691 (3)
S1'	1.1578 (8)	−0.0323 (4)	0.8685 (6)	0.0512 (11)	0.309 (3)
C11'	1.071 (2)	0.0236 (17)	0.7853 (15)	0.035 (5)	0.309 (3)
C12'	1.1315 (17)	0.0266 (11)	0.6919 (11)	0.065 (4)	0.309 (3)
H12'	1.1068	0.0607	0.6407	0.077*	0.309 (3)
C13'	1.230 (2)	−0.0186 (15)	0.666 (2)	0.053 (3)	0.309 (3)
H13'	1.2676	−0.0253	0.6010	0.063*	0.309 (3)
C14'	1.252 (3)	−0.049 (2)	0.7639 (19)	0.055 (4)	0.309 (3)
H14'	1.3186	−0.0786	0.7751	0.065*	0.309 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0379 (11)	0.0318 (10)	0.0268 (10)	0.0002 (9)	0.0069 (8)	0.0045 (8)
C2	0.0401 (12)	0.0479 (13)	0.0367 (11)	0.0005 (10)	0.0028 (10)	0.0000 (10)
C3	0.0517 (14)	0.0550 (14)	0.0360 (12)	−0.0084 (12)	−0.0026 (10)	−0.0017 (11)
C4	0.0672 (17)	0.0460 (13)	0.0363 (12)	−0.0052 (12)	0.0043 (12)	−0.0081 (10)
C5	0.0576 (15)	0.0386 (12)	0.0396 (12)	0.0069 (11)	0.0080 (11)	−0.0051 (10)
C6	0.0436 (12)	0.0311 (10)	0.0310 (10)	0.0020 (9)	0.0056 (9)	0.0009 (8)
C7	0.0419 (12)	0.0363 (11)	0.0353 (11)	0.0093 (10)	0.0085 (9)	0.0010 (9)
C8	0.0318 (11)	0.0346 (11)	0.0302 (10)	0.0047 (8)	0.0059 (8)	0.0018 (8)
C9	0.0352 (11)	0.0287 (10)	0.0256 (9)	0.0042 (8)	0.0070 (8)	0.0049 (8)
C10	0.0331 (11)	0.0300 (10)	0.0264 (10)	0.0029 (9)	0.0064 (8)	0.0045 (8)
C15	0.0370 (12)	0.0459 (13)	0.0353 (11)	0.0059 (10)	0.0080 (9)	0.0036 (9)
C16	0.0592 (16)	0.0565 (15)	0.0646 (16)	−0.0105 (13)	0.0200 (13)	0.0145 (12)
C17	0.0346 (11)	0.0371 (11)	0.0348 (11)	0.0043 (9)	0.0056 (9)	−0.0014 (9)

C18	0.0784 (19)	0.0402 (13)	0.0747 (17)	0.0104 (13)	0.0118 (15)	−0.0183 (12)
C19	0.0315 (11)	0.0303 (10)	0.0345 (10)	0.0046 (8)	0.0024 (9)	0.0009 (8)
C20	0.0346 (12)	0.0369 (11)	0.0488 (13)	−0.0015 (9)	0.0049 (10)	0.0053 (10)
C21	0.0279 (11)	0.0397 (12)	0.0566 (14)	−0.0031 (9)	−0.0038 (10)	−0.0009 (10)
C22	0.0298 (11)	0.0310 (10)	0.0435 (12)	0.0027 (9)	−0.0037 (9)	−0.0029 (9)
C23	0.0346 (12)	0.0480 (13)	0.0531 (13)	0.0006 (10)	−0.0138 (10)	−0.0051 (11)
C24	0.0488 (14)	0.0501 (13)	0.0406 (12)	0.0082 (11)	−0.0159 (10)	−0.0002 (10)
C25	0.0411 (12)	0.0385 (11)	0.0346 (11)	0.0090 (10)	−0.0049 (9)	−0.0014 (9)
C26	0.0578 (15)	0.0553 (14)	0.0344 (12)	0.0111 (12)	−0.0027 (11)	0.0091 (10)
C27	0.0558 (16)	0.0533 (14)	0.0437 (13)	0.0039 (12)	0.0098 (11)	0.0148 (11)
C28	0.0393 (12)	0.0454 (12)	0.0432 (12)	−0.0003 (10)	0.0064 (10)	0.0028 (10)
C29	0.0344 (11)	0.0323 (10)	0.0332 (10)	0.0048 (9)	0.0032 (9)	0.0003 (8)
C30	0.0283 (11)	0.0394 (11)	0.0417 (12)	−0.0036 (9)	−0.0007 (9)	−0.0020 (9)
C31	0.0330 (11)	0.0365 (11)	0.0334 (10)	0.0000 (9)	−0.0053 (9)	0.0014 (9)
C32	0.0297 (10)	0.0270 (10)	0.0333 (10)	0.0035 (8)	0.0002 (8)	−0.0011 (8)
C33	0.0298 (10)	0.0271 (9)	0.0341 (10)	0.0056 (8)	−0.0027 (8)	−0.0036 (8)
C34	0.0334 (11)	0.0290 (10)	0.0316 (10)	0.0065 (8)	0.0002 (8)	−0.0033 (8)
O1	0.0629 (11)	0.0457 (9)	0.0633 (10)	0.0259 (8)	−0.0083 (9)	−0.0094 (8)
O2	0.0910 (14)	0.0621 (11)	0.0876 (13)	0.0152 (10)	0.0616 (12)	0.0072 (10)
O3	0.0543 (10)	0.0387 (8)	0.0566 (9)	0.0007 (7)	0.0249 (8)	0.0074 (7)
O4	0.0868 (13)	0.0656 (11)	0.0406 (9)	0.0292 (10)	−0.0160 (9)	−0.0160 (8)
O5	0.0591 (10)	0.0298 (7)	0.0449 (8)	0.0050 (7)	0.0069 (7)	−0.0016 (6)
S1	0.0468 (6)	0.0439 (6)	0.0418 (8)	0.0002 (5)	0.0211 (6)	−0.0049 (6)
C11	0.027 (3)	0.035 (4)	0.037 (4)	0.007 (3)	0.010 (3)	−0.002 (3)
C12	0.054 (5)	0.069 (5)	0.068 (6)	0.004 (4)	0.010 (4)	−0.003 (4)
C13	0.041 (3)	0.049 (3)	0.079 (5)	0.012 (3)	0.003 (3)	0.003 (4)
C14	0.045 (3)	0.050 (4)	0.068 (6)	0.010 (2)	0.026 (4)	−0.009 (4)
S1'	0.0454 (18)	0.0493 (17)	0.058 (2)	0.0157 (14)	−0.0010 (16)	0.0060 (15)
C11'	0.046 (9)	0.029 (8)	0.031 (8)	−0.008 (7)	−0.007 (7)	0.008 (7)
C12'	0.085 (7)	0.057 (6)	0.052 (7)	0.008 (5)	0.002 (6)	0.005 (6)
C13'	0.051 (6)	0.056 (7)	0.051 (6)	−0.010 (5)	0.007 (5)	0.008 (5)
C14'	0.043 (7)	0.059 (8)	0.062 (8)	0.016 (6)	0.003 (6)	0.002 (7)

Geometric parameters (Å, º)

C1—C2	1.395 (3)	C22—C33	1.420 (3)
C1—C6	1.405 (3)	C22—C23	1.435 (3)
C1—C10	1.478 (3)	C23—C24	1.330 (3)
C2—C3	1.377 (3)	C23—H23	0.9300
C2—H2	0.9300	C24—C25	1.429 (3)
C3—C4	1.369 (3)	C24—H24	0.9300

C3—H3	0.9300	C25—C26	1.391 (3)
C4—C5	1.373 (3)	C25—C34	1.423 (3)
C4—H4	0.9300	C26—C27	1.374 (3)
C5—C6	1.392 (3)	C26—H26	0.9300
C5—H5	0.9300	C27—C28	1.376 (3)
C6—C7	1.467 (3)	C27—H27	0.9300
C7—O1	1.212 (2)	C28—C29	1.402 (3)
C7—C8	1.546 (3)	C28—H28	0.9300
C8—C9	1.532 (3)	C29—C34	1.413 (3)
C8—C19	1.535 (3)	C29—C30	1.425 (3)
C8—C15	1.547 (3)	C30—C31	1.346 (3)
C9—C10	1.345 (3)	C30—H30	0.9300
C9—C17	1.503 (3)	C31—C32	1.434 (3)
C10—C11'	1.421 (16)	C31—H31	0.9300
C10—C11	1.500 (6)	C32—C33	1.423 (3)
C15—O2	1.191 (2)	C33—C34	1.428 (3)
C15—O3	1.314 (2)	S1—C11	1.705 (6)
C16—O3	1.448 (2)	S1—C14	1.717 (6)
C16—H16A	0.9600	C11—C12	1.349 (10)
C16—H16B	0.9600	C12—C13	1.425 (12)
C16—H16C	0.9600	C12—H12	0.9300
C17—O4	1.196 (2)	C13—C14	1.334 (6)
C17—O5	1.325 (2)	C13—H13	0.9300
C18—O5	1.441 (2)	C14—H14	0.9300
C18—H18A	0.9600	S1'—C14'	1.714 (10)
C18—H18B	0.9600	S1'—C11'	1.719 (10)
C18—H18C	0.9600	C11'—C12'	1.351 (13)
C19—C20	1.383 (3)	C12'—C13'	1.422 (14)
C19—C32	1.415 (3)	C12'—H12'	0.9300
C20—C21	1.377 (3)	C13'—C14'	1.335 (9)
C20—H20	0.9300	C13'—H13'	0.9300
C21—C22	1.383 (3)	C14'—H14'	0.9300
C21—H21	0.9300		
C2—C1—C6	117.75 (18)	C33—C22—C23	118.67 (18)
C2—C1—C10	122.26 (18)	C24—C23—C22	121.4 (2)
C6—C1—C10	119.98 (18)	C24—C23—H23	119.3
C3—C2—C1	120.9 (2)	C22—C23—H23	119.3
C3—C2—H2	119.5	C23—C24—C25	122.2 (2)
C1—C2—H2	119.5	C23—C24—H24	118.9
C4—C3—C2	120.9 (2)	C25—C24—H24	118.9

C4—C3—H3	119.6	C26—C25—C34	118.4 (2)
C2—C3—H3	119.6	C26—C25—C24	123.47 (19)
C3—C4—C5	119.6 (2)	C34—C25—C24	118.14 (19)
C3—C4—H4	120.2	C27—C26—C25	121.7 (2)
C5—C4—H4	120.2	C27—C26—H26	119.2
C4—C5—C6	120.6 (2)	C25—C26—H26	119.2
C4—C5—H5	119.7	C26—C27—C28	120.7 (2)
C6—C5—H5	119.7	C26—C27—H27	119.7
C5—C6—C1	120.13 (19)	C28—C27—H27	119.7
C5—C6—C7	119.65 (18)	C27—C28—C29	120.1 (2)
C1—C6—C7	120.15 (17)	C27—C28—H28	120.0
O1—C7—C6	122.76 (19)	C29—C28—H28	120.0
O1—C7—C8	120.05 (19)	C28—C29—C34	119.60 (18)
C6—C7—C8	117.09 (16)	C28—C29—C30	122.49 (19)
C9—C8—C19	113.07 (15)	C34—C29—C30	117.91 (17)
C9—C8—C7	112.70 (16)	C31—C30—C29	121.98 (18)
C19—C8—C7	110.16 (15)	C31—C30—H30	119.0
C9—C8—C15	108.54 (15)	C29—C30—H30	119.0
C19—C8—C15	110.24 (16)	C30—C31—C32	122.13 (18)
C7—C8—C15	101.50 (15)	C30—C31—H31	118.9
C10—C9—C17	121.10 (17)	C32—C31—H31	118.9
C10—C9—C8	122.38 (17)	C19—C32—C33	118.90 (17)
C17—C9—C8	116.49 (17)	C19—C32—C31	123.95 (17)
C9—C10—C11'	120.3 (15)	C33—C32—C31	117.15 (17)
C9—C10—C1	121.27 (17)	C22—C33—C32	120.17 (17)
C11'—C10—C1	118.4 (15)	C22—C33—C34	119.40 (17)
C9—C10—C11	119.8 (7)	C32—C33—C34	120.41 (17)
C11'—C10—C11	1.2 (19)	C29—C34—C25	119.54 (18)
C1—C10—C11	118.9 (7)	C29—C34—C33	120.35 (17)
O2—C15—O3	123.79 (19)	C25—C34—C33	120.09 (18)
O2—C15—C8	123.40 (19)	C15—O3—C16	115.84 (16)
O3—C15—C8	112.80 (16)	C17—O5—C18	116.67 (17)
O3—C16—H16A	109.5	C11—S1—C14	89.3 (4)
O3—C16—H16B	109.5	C12—C11—C10	129.9 (8)
H16A—C16—H16B	109.5	C12—C11—S1	112.2 (7)
O3—C16—H16C	109.5	C10—C11—S1	117.7 (4)
H16A—C16—H16C	109.5	C11—C12—C13	114.2 (11)
H16B—C16—H16C	109.5	C11—C12—H12	122.9
O4—C17—O5	123.28 (19)	C13—C12—H12	122.9
O4—C17—C9	124.83 (18)	C14—C13—C12	108.6 (12)
O5—C17—C9	111.87 (17)	C14—C13—H13	125.7

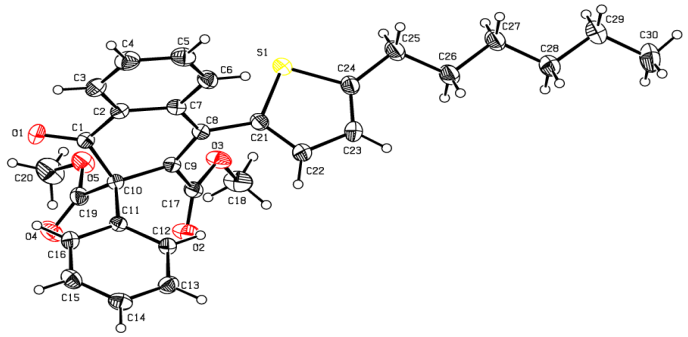
O5—C18—H18A	109.5	C12—C13—H13	125.7
O5—C18—H18B	109.5	C13—C14—S1	115.7 (9)
H18A—C18—H18B	109.5	C13—C14—H14	122.1
O5—C18—H18C	109.5	S1—C14—H14	122.1
H18A—C18—H18C	109.5	C14'—S1'—C11'	90.0 (13)
H18B—C18—H18C	109.5	C12'—C11'—C10	129.4 (12)
C20—C19—C32	119.24 (18)	C12'—C11'—S1'	104.6 (12)
C20—C19—C8	121.38 (17)	C10—C11'—S1'	126.0 (10)
C32—C19—C8	119.37 (17)	C11'—C12'—C13'	124.6 (19)
C21—C20—C19	121.83 (19)	C11'—C12'—H12'	117.7
C21—C20—H20	119.1	C13'—C12'—H12'	117.7
C19—C20—H20	119.1	C14'—C13'—C12'	99 (3)
C20—C21—C22	121.08 (19)	C14'—C13'—H13'	130.3
C20—C21—H21	119.5	C12'—C13'—H13'	130.3
C22—C21—H21	119.5	C13'—C14'—S1'	120 (2)
C21—C22—C33	118.75 (18)	C13'—C14'—H14'	119.8
C21—C22—C23	122.57 (19)	S1'—C14'—H14'	119.8
C6—C1—C2—C3	3.4 (3)	C34—C25—C26—C27	0.4 (3)
C10—C1—C2—C3	−176.23 (18)	C24—C25—C26—C27	−179.7 (2)
C1—C2—C3—C4	−1.9 (3)	C25—C26—C27—C28	0.9 (3)
C2—C3—C4—C5	−1.3 (3)	C26—C27—C28—C29	−1.3 (3)
C3—C4—C5—C6	2.8 (3)	C27—C28—C29—C34	0.5 (3)
C4—C5—C6—C1	−1.3 (3)	C27—C28—C29—C30	−178.90 (19)
C4—C5—C6—C7	−178.23 (19)	C28—C29—C30—C31	176.80 (19)
C2—C1—C6—C5	−1.8 (3)	C34—C29—C30—C31	−2.6 (3)
C10—C1—C6—C5	177.80 (17)	C29—C30—C31—C32	0.9 (3)
C2—C1—C6—C7	175.15 (18)	C20—C19—C32—C33	0.3 (3)
C10—C1—C6—C7	−5.3 (3)	C8—C19—C32—C33	179.54 (16)
C5—C6—C7—O1	18.2 (3)	C20—C19—C32—C31	−179.84 (18)
C1—C6—C7—O1	−158.8 (2)	C8—C19—C32—C31	−0.6 (3)
C5—C6—C7—C8	−158.18 (18)	C30—C31—C32—C19	−178.23 (18)
C1—C6—C7—C8	24.9 (3)	C30—C31—C32—C33	1.7 (3)
O1—C7—C8—C9	154.97 (18)	C21—C22—C33—C32	1.4 (3)
C6—C7—C8—C9	−28.6 (2)	C23—C22—C33—C32	−179.49 (17)
O1—C7—C8—C19	27.7 (2)	C21—C22—C33—C34	−177.63 (17)
C6—C7—C8—C19	−155.90 (16)	C23—C22—C33—C34	1.5 (3)
O1—C7—C8—C15	−89.1 (2)	C19—C32—C33—C22	−1.5 (3)
C6—C7—C8—C15	87.31 (19)	C31—C32—C33—C22	178.59 (17)
C19—C8—C9—C10	140.56 (18)	C19—C32—C33—C34	177.47 (17)
C7—C8—C9—C10	14.8 (2)	C31—C32—C33—C34	−2.4 (3)

C15—C8—C9—C10	−96.8 (2)	C28—C29—C34—C25	0.8 (3)
C19—C8—C9—C17	−41.6 (2)	C30—C29—C34—C25	−179.81 (17)
C7—C8—C9—C17	−167.36 (15)	C28—C29—C34—C33	−177.67 (17)
C15—C8—C9—C17	81.03 (19)	C30—C29—C34—C33	1.7 (3)
C17—C9—C10—C11'	9.6 (11)	C26—C25—C34—C29	−1.2 (3)
C8—C9—C10—C11'	−172.7 (11)	C24—C25—C34—C29	178.87 (18)
C17—C9—C10—C1	−173.57 (16)	C26—C25—C34—C33	177.24 (18)
C8—C9—C10—C1	4.2 (3)	C24—C25—C34—C33	−2.7 (3)
C17—C9—C10—C11	8.3 (5)	C22—C33—C34—C29	179.74 (17)
C8—C9—C10—C11	−174.0 (5)	C32—C33—C34—C29	0.8 (3)
C2—C1—C10—C9	169.54 (18)	C22—C33—C34—C25	1.3 (3)
C6—C1—C10—C9	−10.0 (3)	C32—C33—C34—C25	−177.70 (17)
C2—C1—C10—C11'	−13.6 (10)	O2—C15—O3—C16	−1.0 (3)
C6—C1—C10—C11'	166.8 (10)	C8—C15—O3—C16	178.12 (18)
C2—C1—C10—C11	−12.3 (5)	O4—C17—O5—C18	13.0 (3)
C6—C1—C10—C11	168.1 (5)	C9—C17—O5—C18	−168.71 (18)
C9—C8—C15—O2	151.4 (2)	C9—C10—C11—C12	−116.6 (17)
C19—C8—C15—O2	−84.2 (3)	C11'—C10—C11—C12	131 (81)
C7—C8—C15—O2	32.5 (3)	C1—C10—C11—C12	65 (2)
C9—C8—C15—O3	−27.7 (2)	C9—C10—C11—S1	57.6 (13)
C19—C8—C15—O3	96.7 (2)	C11'—C10—C11—S1	−54 (78)
C7—C8—C15—O3	−146.59 (18)	C1—C10—C11—S1	−120.6 (9)
C10—C9—C17—O4	−133.5 (2)	C14—S1—C11—C12	−1.3 (14)
C8—C9—C17—O4	48.7 (3)	C14—S1—C11—C10	−176.5 (13)
C10—C9—C17—O5	48.3 (2)	C10—C11—C12—C13	175.3 (16)
C8—C9—C17—O5	−129.54 (17)	S1—C11—C12—C13	1 (2)
C9—C8—C19—C20	114.1 (2)	C11—C12—C13—C14	0 (2)
C7—C8—C19—C20	−118.8 (2)	C12—C13—C14—S1	−1.2 (18)
C15—C8—C19—C20	−7.6 (2)	C11—S1—C14—C13	1.5 (13)
C9—C8—C19—C32	−65.2 (2)	C9—C10—C11'—C12'	62 (4)
C7—C8—C19—C32	61.9 (2)	C1—C10—C11'—C12'	−115 (3)
C15—C8—C19—C32	173.14 (16)	C11—C10—C11'—C12'	131 (82)
C32—C19—C20—C21	1.1 (3)	C9—C10—C11'—S1'	−119 (2)
C8—C19—C20—C21	−178.11 (18)	C1—C10—C11'—S1'	65 (3)
C19—C20—C21—C22	−1.3 (3)	C11—C10—C11'—S1'	−50 (76)
C20—C21—C22—C33	0.0 (3)	C14'—S1'—C11'—C12'	−5 (3)
C20—C21—C22—C23	−179.07 (19)	C14'—S1'—C11'—C10	176 (3)
C21—C22—C23—C24	176.0 (2)	C10—C11'—C12'—C13'	−169 (3)
C33—C22—C23—C24	−3.1 (3)	S1'—C11'—C12'—C13'	11 (4)
C22—C23—C24—C25	1.7 (3)	C11'—C12'—C13'—C14'	−11 (4)
C23—C24—C25—C26	−178.7 (2)	C12'—C13'—C14'—S1'	6 (4)

C23—C24—C25—C34	1.2 (3)	C11'—S1'—C14'—C13'	−1 (4)

Crystallograpic data of *β*-keto-ester **9h**:

CCDC Number is **1450133**



Computing details

Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997).

Dimethyl-4-(5-hexylthiophen-2-yl)-1-oxo-2-phenyl-1,2-dihydronaphthalene-2,3-dicarboxylate (9h)

Experimental

Crystal data

$C_{30}H_{30}O_5S$	Z = 2
M_r = 502.60	F(000) = 532
Triclinic, $P\overline{1}$	D_x = 1.287 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, λ = 0.71073 Å
a = 7.4725 (4) Å	Cell parameters from 5097 reflections
b = 12.9073 (7) Å	θ = 2.6–26.0°
c = 14.9503 (8) Å	μ = 0.16 mm^{-1}
α = 105.607 (2)°	T = 296 K
β = 103.985 (4)°	Block, colourless
γ = 100.951 (5)°	0.35 × 0.30 × 0.25 mm
V = 1296.49 (12) Å^3	

Data collection

Kappa ApexII CCD Diffractometer	5097 independent reflections
Radiation source: fine-focus sealed tube	4006 reflections with $I > 2\sigma(I)$

Graphite monochromator	R_{int} = 0.034
ω & φ scans	θ_{max} = 26.0°, θ_{min} = 2.6°
Absorption correction: multi-scan *SADABS* (Bruker, 2008)	h = −9→9
T_{min} = 0.945, T_{max} = 0.960	k = −15→15
26878 measured reflections	l = −18→18

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.040	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.141	H-atom parameters constrained
S = 0.99	$w = 1/[\sigma^2(F_o^2) + (0.0918P)^2 + 0.2678P]$ where $P = (F_o^2 + 2F_c^2)/3$
5097 reflections	$(\Delta/\sigma)_{max} < 0.001$
328 parameters	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Special details

Refinement

Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on*F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*Y*	*z*	U_{iso}*/U_{eq}
C1	0.5780 (2)	0.72824 (15)	0.36737 (12)	0.0379 (4)
C2	0.6857 (2)	0.84143 (14)	0.37843 (13)	0.0376 (4)
C3	0.6631 (3)	0.93389 (16)	0.44287 (15)	0.0494 (5)
H3	0.5780	0.9243	0.4781	0.059*
C4	0.7660 (3)	1.03996 (17)	0.45504 (16)	0.0560 (5)
H4	0.7517	1.1018	0.4990	0.067*
C5	0.8899 (3)	1.05410 (16)	0.40195 (15)	0.0525 (5)
H5	0.9600	1.1257	0.4104	0.063*
C6	0.9110 (3)	0.96248 (15)	0.33594 (14)	0.0446 (4)
H6	0.9935	0.9732	0.2995	0.054*
C7	0.8103 (2)	0.85453 (14)	0.32345 (12)	0.0362 (4)
C8	0.8286 (2)	0.75488 (13)	0.25340 (12)	0.0337 (3)
C9	0.7558 (2)	0.65176 (13)	0.25477 (11)	0.0330 (3)

C10	0.6708 (2)	0.63412 (13)	0.33416 (12)	0.0331 (3)
C11	0.8220 (2)	0.63934 (13)	0.42672 (11)	0.0322 (3)
C12	1.0123 (2)	0.64787 (14)	0.43176 (12)	0.0365 (4)
H12	1.0524	0.6532	0.3785	0.044*
C13	1.1428 (3)	0.64849 (15)	0.51516 (14)	0.0433 (4)
H13	1.2699	0.6541	0.5176	0.052*
C14	1.0859 (3)	0.64085 (15)	0.59430 (13)	0.0469 (4)
H14	1.1734	0.6397	0.6498	0.056*
C15	0.8985 (3)	0.63495 (17)	0.59094 (14)	0.0489 (5)
H15	0.8602	0.6315	0.6451	0.059*
C16	0.7670 (3)	0.63409 (15)	0.50811 (13)	0.0417 (4)
H16	0.6407	0.6300	0.5067	0.050*
C17	0.7642 (2)	0.54771 (14)	0.18483 (12)	0.0372 (4)
C18	0.7152 (4)	0.44620 (19)	0.02138 (15)	0.0641 (6)
H18A	0.8420	0.4365	0.0328	0.096*
H18B	0.6709	0.4512	−0.0428	0.096*
H18C	0.6303	0.3835	0.0263	0.096*
C19	0.5116 (2)	0.52348 (15)	0.29554 (13)	0.0405 (4)
C20	0.2373 (3)	0.4132 (2)	0.16447 (19)	0.0767 (8)
H20A	0.2817	0.3506	0.1739	0.115*
H20B	0.1864	0.4004	0.0958	0.115*
H20C	0.1389	0.4219	0.1947	0.115*
C21	0.9427 (2)	0.77292 (14)	0.18819 (12)	0.0377 (4)
C22	1.1179 (3)	0.75668 (17)	0.19409 (14)	0.0466 (4)
H22	1.1823	0.7290	0.2398	0.056*
C23	1.1931 (3)	0.78627 (18)	0.12325 (15)	0.0507 (5)
H23	1.3127	0.7802	0.1184	0.061*
C24	1.0764 (3)	0.82387 (15)	0.06361 (13)	0.0427 (4)
C25	1.1068 (3)	0.86537 (18)	−0.01800 (16)	0.0536 (5)
H25A	0.9893	0.8352	−0.0726	0.064*
H25B	1.1327	0.9462	0.0041	0.064*
C26	1.2680 (3)	0.83450 (18)	−0.05301 (16)	0.0558 (5)
H26A	1.2400	0.7536	−0.0769	0.067*
H26B	1.3845	0.8625	0.0021	0.067*
C27	1.3039 (3)	0.87873 (18)	−0.13271 (16)	0.0551 (5)
H27A	1.3286	0.9594	−0.1099	0.066*
H27B	1.1895	0.8486	−0.1890	0.066*
C28	1.4713 (3)	0.84909 (19)	−0.16360 (16)	0.0553 (5)
H28A	1.5852	0.8790	−0.1070	0.066*
H28B	1.4462	0.7684	−0.1861	0.066*
C29	1.5111 (3)	0.8920 (2)	−0.24299 (17)	0.0610 (6)
H29A	1.3960	0.8638	−0.2989	0.073*
H29B	1.5397	0.9729	−0.2198	0.073*

C30	1.6739 (4)	0.8599 (3)	−0.2755 (2)	0.0926 (9)
H30A	1.7908	0.8928	−0.2221	0.139*
H30B	1.6858	0.8863	−0.3286	0.139*
H30C	1.6490	0.7799	−0.2968	0.139*
O1	0.43014 (18)	0.70978 (12)	0.38749 (11)	0.0547 (4)
O2	0.7987 (2)	0.46977 (11)	0.20797 (10)	0.0574 (4)
O3	0.7186 (2)	0.54740 (10)	0.09311 (9)	0.0492 (3)
O4	0.4904 (2)	0.45687 (12)	0.33575 (11)	0.0566 (4)
O5	0.39542 (18)	0.51356 (12)	0.20815 (10)	0.0530 (4)
S1	0.86882 (7)	0.82415 (4)	0.09458 (4)	0.04751 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0349 (8)	0.0472 (10)	0.0408 (9)	0.0170 (7)	0.0159 (7)	0.0212 (8)
C2	0.0381 (9)	0.0394 (9)	0.0429 (9)	0.0178 (7)	0.0164 (7)	0.0172 (7)
C3	0.0545 (11)	0.0491 (11)	0.0571 (12)	0.0272 (9)	0.0279 (9)	0.0186 (9)
C4	0.0699 (13)	0.0430 (11)	0.0587 (12)	0.0272 (10)	0.0230 (11)	0.0115 (9)
C5	0.0606 (12)	0.0352 (10)	0.0589 (12)	0.0126 (9)	0.0150 (10)	0.0149 (9)
C6	0.0484 (10)	0.0387 (10)	0.0495 (10)	0.0104 (8)	0.0186 (8)	0.0170 (8)
C7	0.0381 (9)	0.0352 (9)	0.0381 (8)	0.0124 (7)	0.0115 (7)	0.0150 (7)
C8	0.0336 (8)	0.0358 (8)	0.0355 (8)	0.0111 (7)	0.0121 (7)	0.0150 (7)
C9	0.0336 (8)	0.0366 (9)	0.0336 (8)	0.0127 (6)	0.0121 (6)	0.0151 (7)
C10	0.0314 (8)	0.0348 (8)	0.0380 (8)	0.0098 (6)	0.0139 (7)	0.0163 (7)
C11	0.0343 (8)	0.0293 (8)	0.0361 (8)	0.0091 (6)	0.0128 (7)	0.0135 (7)
C12	0.0364 (8)	0.0375 (9)	0.0392 (9)	0.0113 (7)	0.0154 (7)	0.0146 (7)
C13	0.0352 (9)	0.0442 (10)	0.0493 (10)	0.0122 (7)	0.0094 (8)	0.0158 (8)
C14	0.0517 (11)	0.0432 (10)	0.0404 (10)	0.0106 (8)	0.0040 (8)	0.0161 (8)
C15	0.0592 (12)	0.0539 (11)	0.0390 (10)	0.0142 (9)	0.0196 (9)	0.0209 (9)
C16	0.0415 (9)	0.0486 (10)	0.0431 (9)	0.0149 (8)	0.0199 (8)	0.0197 (8)
C17	0.0412 (9)	0.0360 (9)	0.0385 (9)	0.0126 (7)	0.0145 (7)	0.0155 (7)
C18	0.0969 (18)	0.0569 (13)	0.0391 (11)	0.0328 (12)	0.0200 (11)	0.0097 (9)
C19	0.0347 (9)	0.0444 (10)	0.0436 (9)	0.0075 (7)	0.0134 (7)	0.0175 (8)
C20	0.0569 (14)	0.0750 (16)	0.0681 (15)	−0.0176 (12)	−0.0063 (11)	0.0239 (13)
C21	0.0418 (9)	0.0360 (9)	0.0387 (9)	0.0101 (7)	0.0160 (7)	0.0150 (7)
C22	0.0460 (10)	0.0589 (12)	0.0478 (10)	0.0213 (9)	0.0211 (8)	0.0273 (9)
C23	0.0482 (11)	0.0621 (12)	0.0550 (11)	0.0203 (9)	0.0292 (9)	0.0250 (10)
C24	0.0501 (10)	0.0399 (9)	0.0446 (10)	0.0124 (8)	0.0240 (8)	0.0158 (8)
C25	0.0675 (13)	0.0540 (12)	0.0554 (12)	0.0197 (10)	0.0336 (10)	0.0286 (10)
C26	0.0707 (13)	0.0589 (12)	0.0582 (12)	0.0254 (10)	0.0377 (11)	0.0302 (10)
C27	0.0681 (13)	0.0556 (12)	0.0558 (12)	0.0184 (10)	0.0332 (11)	0.0271 (10)
C28	0.0588 (12)	0.0629 (13)	0.0520 (11)	0.0161 (10)	0.0243 (10)	0.0252 (10)
C29	0.0646 (13)	0.0654 (14)	0.0583 (13)	0.0095 (11)	0.0280 (11)	0.0266 (11)
C30	0.0860 (19)	0.125 (3)	0.096 (2)	0.0299 (18)	0.0607 (17)	0.0537 (19)

O1	0.0428 (7)	0.0646 (9)	0.0781 (10)	0.0244 (6)	0.0364 (7)	0.0349 (8)
O2	0.0915 (11)	0.0443 (8)	0.0473 (8)	0.0326 (7)	0.0234 (7)	0.0212 (6)
O3	0.0749 (9)	0.0404 (7)	0.0343 (6)	0.0215 (6)	0.0157 (6)	0.0129 (5)
O4	0.0525 (8)	0.0520 (8)	0.0623 (9)	−0.0024 (6)	0.0120 (7)	0.0311 (7)
O5	0.0421 (7)	0.0566 (8)	0.0491 (8)	−0.0028 (6)	0.0029 (6)	0.0211 (6)
S1	0.0513 (3)	0.0581 (3)	0.0509 (3)	0.0232 (2)	0.0252 (2)	0.0319 (2)

Geometric parameters (Å, º)

C1—O1	1.211 (2)	C18—H18B	0.9600
C1—C2	1.473 (2)	C18—H18C	0.9600
C1—C10	1.538 (2)	C19—O4	1.182 (2)
C2—C3	1.385 (3)	C19—O5	1.341 (2)
C2—C7	1.398 (2)	C20—O5	1.446 (2)
C3—C4	1.377 (3)	C20—H20A	0.9600
C3—H3	0.9300	C20—H20B	0.9600
C4—C5	1.375 (3)	C20—H20C	0.9600
C4—H4	0.9300	C21—C22	1.350 (3)
C5—C6	1.384 (3)	C21—S1	1.7216 (17)
C5—H5	0.9300	C22—C23	1.417 (3)
C6—C7	1.392 (2)	C22—H22	0.9300
C6—H6	0.9300	C23—C24	1.345 (3)
C7—C8	1.477 (2)	C23—H23	0.9300
C8—C9	1.346 (2)	C24—C25	1.506 (2)
C8—C21	1.477 (2)	C24—S1	1.7227 (18)
C9—C17	1.486 (2)	C25—C26	1.503 (3)
C9—C10	1.523 (2)	C25—H25A	0.9700
C10—C11	1.537 (2)	C25—H25B	0.9700
C10—C19	1.538 (2)	C26—C27	1.510 (3)
C11—C12	1.387 (2)	C26—H26A	0.9700
C11—C16	1.389 (2)	C26—H26B	0.9700
C12—C13	1.382 (2)	C27—C28	1.513 (3)
C12—H12	0.9300	C27—H27A	0.9700
C13—C14	1.371 (3)	C27—H27B	0.9700
C13—H13	0.9300	C28—C29	1.505 (3)
C14—C15	1.376 (3)	C28—H28A	0.9700
C14—H14	0.9300	C28—H28B	0.9700
C15—C16	1.378 (3)	C29—C30	1.503 (3)
C15—H15	0.9300	C29—H29A	0.9700
C16—H16	0.9300	C29—H29B	0.9700
C17—O2	1.200 (2)	C30—H30A	0.9600
C17—O3	1.329 (2)	C30—H30B	0.9600
C18—O3	1.441 (2)	C30—H30C	0.9600
C18—H18A	0.9600		

O1—C1—C2	122.82 (16)	O4—C19—O5	124.12 (16)
O1—C1—C10	121.24 (16)	O4—C19—C10	126.43 (16)
C2—C1—C10	115.85 (13)	O5—C19—C10	109.45 (14)
C3—C2—C7	120.52 (16)	O5—C20—H20A	109.5
C3—C2—C1	119.96 (16)	O5—C20—H20B	109.5
C7—C2—C1	119.52 (15)	H20A—C20—H20B	109.5
C4—C3—C2	120.36 (18)	O5—C20—H20C	109.5
C4—C3—H3	119.8	H20A—C20—H20C	109.5
C2—C3—H3	119.8	H20B—C20—H20C	109.5
C5—C4—C3	119.76 (18)	C22—C21—C8	126.54 (16)
C5—C4—H4	120.1	C22—C21—S1	110.42 (13)
C3—C4—H4	120.1	C8—C21—S1	123.00 (13)
C4—C5—C6	120.42 (18)	C21—C22—C23	112.88 (17)
C4—C5—H5	119.8	C21—C22—H22	123.6
C6—C5—H5	119.8	C23—C22—H22	123.6
C5—C6—C7	120.73 (17)	C24—C23—C22	114.13 (17)
C5—C6—H6	119.6	C24—C23—H23	122.9
C7—C6—H6	119.6	C22—C23—H23	122.9
C6—C7—C2	118.19 (16)	C23—C24—C25	129.83 (17)
C6—C7—C8	122.23 (15)	C23—C24—S1	109.91 (13)
C2—C7—C8	119.58 (15)	C25—C24—S1	120.24 (15)
C9—C8—C21	121.43 (14)	C26—C25—C24	114.06 (17)
C9—C8—C7	120.59 (14)	C26—C25—H25A	108.7
C21—C8—C7	117.78 (14)	C24—C25—H25A	108.7
C8—C9—C17	123.49 (14)	C26—C25—H25B	108.7
C8—C9—C10	121.59 (14)	C24—C25—H25B	108.7
C17—C9—C10	114.77 (13)	H25A—C25—H25B	107.6
C9—C10—C11	113.10 (12)	C25—C26—C27	114.55 (18)
C9—C10—C1	109.64 (13)	C25—C26—H26A	108.6
C11—C10—C1	105.49 (13)	C27—C26—H26A	108.6
C9—C10—C19	111.14 (13)	C25—C26—H26B	108.6
C11—C10—C19	110.29 (13)	C27—C26—H26B	108.6
C1—C10—C19	106.84 (13)	H26A—C26—H26B	107.6
C12—C11—C16	118.48 (15)	C26—C27—C28	112.92 (18)
C12—C11—C10	122.27 (14)	C26—C27—H27A	109.0
C16—C11—C10	119.24 (14)	C28—C27—H27A	109.0
C13—C12—C11	120.59 (16)	C26—C27—H27B	109.0
C13—C12—H12	119.7	C28—C27—H27B	109.0
C11—C12—H12	119.7	H27A—C27—H27B	107.8
C14—C13—C12	120.41 (17)	C29—C28—C27	114.27 (19)
C14—C13—H13	119.8	C29—C28—H28A	108.7
C12—C13—H13	119.8	C27—C28—H28A	108.7
C13—C14—C15	119.50 (17)	C29—C28—H28B	108.7

C13—C14—H14	120.3	C27—C28—H28B	108.7
C15—C14—H14	120.3	H28A—C28—H28B	107.6
C14—C15—C16	120.62 (17)	C30—C29—C28	114.2 (2)
C14—C15—H15	119.7	C30—C29—H29A	108.7
C16—C15—H15	119.7	C28—C29—H29A	108.7
C15—C16—C11	120.38 (16)	C30—C29—H29B	108.7
C15—C16—H16	119.8	C28—C29—H29B	108.7
C11—C16—H16	119.8	H29A—C29—H29B	107.6
O2—C17—O3	123.48 (16)	C29—C30—H30A	109.5
O2—C17—C9	123.51 (15)	C29—C30—H30B	109.5
O3—C17—C9	112.89 (14)	H30A—C30—H30B	109.5
O3—C18—H18A	109.5	C29—C30—H30C	109.5
O3—C18—H18B	109.5	H30A—C30—H30C	109.5
H18A—C18—H18B	109.5	H30B—C30—H30C	109.5
O3—C18—H18C	109.5	C17—O3—C18	115.33 (14)
H18A—C18—H18C	109.5	C19—O5—C20	114.84 (16)
H18B—C18—H18C	109.5	C21—S1—C24	92.67 (9)
O1—C1—C2—C3	23.0 (3)	C16—C11—C12—C13	1.5 (2)
C10—C1—C2—C3	−153.45 (16)	C10—C11—C12—C13	−177.77 (15)
O1—C1—C2—C7	−156.92 (18)	C11—C12—C13—C14	−0.1 (3)
C10—C1—C2—C7	26.6 (2)	C12—C13—C14—C15	−1.4 (3)
C7—C2—C3—C4	−1.3 (3)	C13—C14—C15—C16	1.5 (3)
C1—C2—C3—C4	178.77 (18)	C14—C15—C16—C11	−0.1 (3)
C2—C3—C4—C5	0.9 (3)	C12—C11—C16—C15	−1.4 (3)
C3—C4—C5—C6	0.4 (3)	C10—C11—C16—C15	177.89 (16)
C4—C5—C6—C7	−1.2 (3)	C8—C9—C17—O2	139.04 (19)
C5—C6—C7—C2	0.8 (3)	C10—C9—C17—O2	−36.6 (2)
C5—C6—C7—C8	179.70 (17)	C8—C9—C17—O3	−44.7 (2)
C3—C2—C7—C6	0.4 (3)	C10—C9—C17—O3	139.66 (15)
C1—C2—C7—C6	−179.61 (16)	C9—C10—C19—O4	135.44 (19)
C3—C2—C7—C8	−178.49 (16)	C11—C10—C19—O4	9.2 (2)
C1—C2—C7—C8	1.5 (2)	C1—C10—C19—O4	−105.0 (2)
C6—C7—C8—C9	168.58 (16)	C9—C10—C19—O5	−45.47 (18)
C2—C7—C8—C9	−12.5 (2)	C11—C10—C19—O5	−171.72 (13)
C6—C7—C8—C21	−6.3 (2)	C1—C10—C19—O5	74.11 (17)
C2—C7—C8—C21	172.56 (15)	C9—C8—C21—C22	−67.3 (3)
C21—C8—C9—C17	−7.0 (2)	C7—C8—C21—C22	107.6 (2)
C7—C8—C9—C17	178.29 (15)	C9—C8—C21—S1	115.25 (17)
C21—C8—C9—C10	168.39 (15)	C7—C8—C21—S1	−69.88 (19)
C7—C8—C9—C10	−6.3 (2)	C8—C21—C22—C23	−177.36 (17)
C8—C9—C10—C11	−85.16 (18)	S1—C21—C22—C23	0.3 (2)
C17—C9—C10—C11	90.60 (17)	C21—C22—C23—C24	−0.4 (3)
C8—C9—C10—C1	32.3 (2)	C22—C23—C24—C25	178.37 (19)

C17—C9—C10—C1	−151.98 (14)	C22—C23—C24—S1	0.3 (2)
C8—C9—C10—C19	150.16 (16)	C23—C24—C25—C26	16.4 (3)
C17—C9—C10—C19	−34.09 (18)	S1—C24—C25—C26	−165.73 (16)
O1—C1—C10—C9	142.13 (17)	C24—C25—C26—C27	−178.19 (18)
C2—C1—C10—C9	−41.33 (19)	C25—C26—C27—C28	178.12 (19)
O1—C1—C10—C11	−95.79 (19)	C26—C27—C28—C29	−179.97 (19)
C2—C1—C10—C11	80.76 (16)	C27—C28—C29—C30	−178.4 (2)
O1—C1—C10—C19	21.6 (2)	O2—C17—O3—C18	−0.8 (3)
C2—C1—C10—C19	−161.87 (14)	C9—C17—O3—C18	−177.08 (17)
C9—C10—C11—C12	−6.2 (2)	O4—C19—O5—C20	0.0 (3)
C1—C10—C11—C12	−125.99 (16)	C10—C19—O5—C20	−179.14 (18)
C19—C10—C11—C12	118.99 (16)	C22—C21—S1—C24	−0.15 (15)
C9—C10—C11—C16	174.60 (14)	C8—C21—S1—C24	177.65 (15)
C1—C10—C11—C16	54.77 (18)	C23—C24—S1—C21	−0.09 (16)
C19—C10—C11—C16	−60.25 (19)	C25—C24—S1—C21	−178.37 (16)

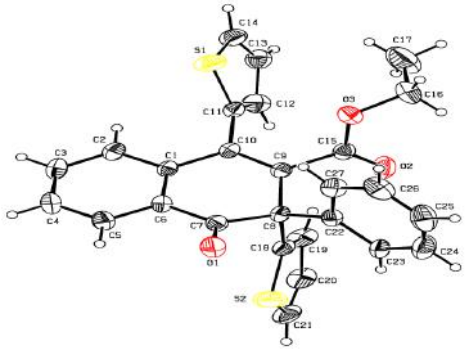
Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C18—H18*B*···O5	0.96	2.58	3.533 (3)	171
C22—H22···O1^{i}	0.93	2.62	3.520 (2)	163

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) *x*+1, *y*, *z*.

Crystallograpic data of *β*-keto-ester **10b**:

CCDC Number is **1438209**.



Computing details

Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Computer programs: *SHELXS97* (Sheldrick, 1990), *SHELXL97* (Sheldrick, 1997).

Ethyl-4-oxo-3-phenyl-1,3-di(thiophen-2-yl)-3,4-dihydronaphthalene-2-carboxylate (10b)

Experimental

Crystal data

$C_{27}H_{20}O_3S_2$	V = 2262.8 (3) Å^3
M_r = 456.55	Z = 4
a = 12.1263 (11) Å	F(000) = 952
b = 11.8009 (11) Å	D_x = 1.340 Mg m^{-3}
c = 16.0657 (13) Å	Mo $K\alpha$ radiation, λ = 0.71073 Å
α = 90°	μ = 0.26 mm^{-1}
β = 100.181 (2)°	T = 296 K
γ = 90°	0.25 × 0.25 × 0.15 mm

Data collection

Radiation source: fine-focus sealed tube	R_{int} = 0.044
Graphite monochromator	θ_{max} = 25.4°, θ_{min} = 2.2°
29905 measured reflections	h = −14→14
4111 independent reflections	k = −14→14
2686 reflections with $I > 2\sigma(I)$	l = −19→18

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.061	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.148	H atoms treated by a mixture of independent and constrained

	refinement
S = 1.09	$w = 1/[\sigma^2(F_o^2) + (0.0338P)^2 + 2.9594P]$ where $P = (F_o^2 + 2F_c^2)/3$
4111 reflections	$(\Delta/\sigma)_{max} = 0.004$
364 parameters	$\Delta\rho_{max} = 0.24$ e Å^{-3}
100 restraints	$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Refinement
Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*Z*	U_{iso}*/U_{eq}	Occ. (<1)
C7	0.6147 (3)	0.7554 (3)	0.11948 (19)	0.0430 (7)	
C6	0.5745 (2)	0.7440 (3)	0.02783 (18)	0.0394 (7)	
C5	0.4953 (3)	0.6614 (3)	−0.0013 (2)	0.0519 (8)	
H5	0.4686	0.6142	0.0371	0.062*	
C4	0.4560 (3)	0.6490 (3)	−0.0866 (2)	0.0600 (10)	
H4	0.4034	0.5934	−0.1059	0.072*	
C3	0.4950 (3)	0.7196 (3)	−0.1430 (2)	0.0575 (9)	
H3	0.4687	0.7111	−0.2006	0.069*	
C2	0.5725 (3)	0.8026 (3)	−0.11538 (19)	0.0480 (8)	
H2	0.5976	0.8498	−0.1544	0.058*	
C1	0.6138 (2)	0.8163 (3)	−0.02929 (18)	0.0369 (7)	
C10	0.6948 (2)	0.9062 (2)	0.00282 (17)	0.0352 (7)	
C9	0.7444 (2)	0.9089 (3)	0.08433 (18)	0.0368 (7)	
C8	0.7274 (2)	0.8171 (3)	0.14761 (17)	0.0385 (7)	
C22	0.7307 (3)	0.8700 (3)	0.23535 (18)	0.0424 (7)	
C23	0.8062 (3)	0.8363 (3)	0.30967 (19)	0.0465 (8)	
H23	0.8569	0.7774	0.3082	0.056*	
C24	0.8016 (4)	0.8933 (4)	0.3831 (2)	0.0773 (13)	
H24	0.8507	0.8724	0.4319	0.093*	
C25	0.7296 (4)	0.9778 (4)	0.3875 (3)	0.0817 (14)	
H25	0.7297	1.0143	0.4388	0.098*	

C26	0.6570 (4)	1.0107 (4)	0.3185 (3)	0.0729 (12)	
H26	0.6065	1.0689	0.3225	0.087*	
C27	0.6573 (3)	0.9589 (3)	0.2429 (2)	0.0555 (9)	
H27	0.6076	0.9831	0.1953	0.067*	
C15	0.8246 (3)	1.0013 (3)	0.11779 (19)	0.0450 (8)	
C16	0.8442 (4)	1.2005 (4)	0.1385 (3)	0.0862 (14)	
H16A	0.8015	1.2542	0.1657	0.103*	
H16B	0.9088	1.1771	0.1796	0.103*	
C17	0.8812 (6)	1.2540 (5)	0.0680 (4)	0.123 (2)	
H17A	0.9295	1.2032	0.0445	0.185*	
H17B	0.9214	1.3221	0.0866	0.185*	
H17C	0.8174	1.2724	0.0256	0.185*	
C11	0.7228 (2)	0.9919 (3)	−0.05704 (19)	0.0408 (7)	
S1'	0.8517 (4)	0.9931 (5)	−0.0827 (3)	0.0550 (9)	0.471 (4)
C12'	0.6541 (15)	1.0805 (16)	−0.0905 (16)	0.073 (5)	0.471 (4)
H12'	0.5824	1.0924	−0.0794	0.088*	0.471 (4)
C13'	0.7086 (15)	1.154 (2)	−0.1465 (17)	0.069 (4)	0.471 (4)
H13'	0.6782	1.2174	−0.1769	0.083*	0.471 (4)
C14'	0.8128 (17)	1.1090 (17)	−0.1434 (15)	0.054 (3)	0.471 (4)
H14'	0.8623	1.1416	−0.1744	0.065*	0.471 (4)
S1	0.6308 (4)	1.0925 (5)	−0.1018 (4)	0.0707 (12)	0.529 (4)
C12	0.8222 (10)	1.0026 (18)	−0.0858 (12)	0.072 (5)	0.529 (4)
H12	0.8807	0.9517	−0.0699	0.086*	0.529 (4)
C13	0.8325 (16)	1.0987 (17)	−0.1432 (16)	0.062 (3)	0.529 (4)
H13	0.8942	1.1207	−0.1664	0.074*	0.529 (4)
C14	0.7296 (12)	1.1464 (19)	−0.1533 (15)	0.060 (3)	0.529 (4)
H14	0.7129	1.2089	−0.1886	0.072*	0.529 (4)
C18	0.8130 (3)	0.7231 (3)	0.14620 (19)	0.0448 (8)	
S2	0.8058 (3)	0.5957 (2)	0.1929 (2)	0.0788 (10)	0.633 (5)
C19	0.9074 (11)	0.7381 (13)	0.1097 (12)	0.065 (4)	0.633 (5)
H19	0.9267	0.8030	0.0827	0.078*	0.633 (5)
C20	0.9724 (10)	0.6298 (8)	0.1220 (8)	0.067 (2)	0.633 (5)
H20	1.0377	0.6190	0.1001	0.081*	0.633 (5)
C21	0.9295 (7)	0.5468 (11)	0.1679 (8)	0.077 (2)	0.633 (5)
H21	0.9619	0.4765	0.1826	0.093*	0.633 (5)
S2'	0.9240 (7)	0.7245 (7)	0.0999 (7)	0.084 (2)	0.367 (5)
C19'	0.795 (2)	0.6208 (14)	0.1853 (19)	0.110 (7)	0.367 (5)
H19'	0.7358	0.6016	0.2126	0.132*	0.367 (5)
C20'	0.8927 (18)	0.549 (3)	0.1726 (18)	0.099 (5)	0.367 (5)
H20'	0.9004	0.4766	0.1950	0.119*	0.367 (5)
C21'	0.973 (2)	0.5911 (13)	0.1276 (19)	0.099 (6)	0.367 (5)

H21'	1.0365	0.5553	0.1157	0.119*	0.367 (5)
O1	0.5657 (2)	0.7135 (2)	0.17142 (14)	0.0627 (7)	
O2	0.9203 (2)	0.9858 (2)	0.14861 (17)	0.0692 (8)	
O3	0.7748 (2)	1.1019 (2)	0.11081 (16)	0.0622 (7)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C7	0.0478 (18)	0.0444 (18)	0.0371 (17)	−0.0016 (15)	0.0082 (14)	−0.0012 (14)
C6	0.0350 (16)	0.0465 (18)	0.0376 (17)	0.0002 (14)	0.0087 (13)	−0.0070 (14)
C5	0.0490 (19)	0.057 (2)	0.051 (2)	−0.0094 (17)	0.0123 (15)	−0.0075 (17)
C4	0.051 (2)	0.068 (2)	0.057 (2)	−0.0145 (18)	0.0020 (17)	−0.017 (2)
C3	0.055 (2)	0.073 (3)	0.042 (2)	−0.0038 (19)	−0.0012 (16)	−0.0138 (18)
C2	0.0465 (19)	0.059 (2)	0.0381 (18)	0.0013 (16)	0.0059 (14)	−0.0005 (16)
C1	0.0296 (14)	0.0449 (18)	0.0357 (16)	0.0059 (13)	0.0041 (12)	−0.0034 (14)
C10	0.0297 (14)	0.0425 (17)	0.0339 (16)	0.0048 (13)	0.0068 (12)	−0.0004 (13)
C9	0.0312 (15)	0.0415 (17)	0.0378 (17)	0.0010 (13)	0.0064 (12)	−0.0004 (13)
C8	0.0385 (16)	0.0446 (18)	0.0314 (16)	−0.0007 (14)	0.0039 (13)	−0.0026 (13)
C22	0.0428 (17)	0.0499 (19)	0.0357 (17)	−0.0068 (15)	0.0100 (13)	−0.0038 (14)
C23	0.0435 (18)	0.060 (2)	0.0357 (18)	−0.0077 (16)	0.0056 (14)	−0.0080 (15)
C24	0.075 (3)	0.107 (4)	0.046 (2)	−0.022 (3)	0.002 (2)	−0.005 (2)
C25	0.083 (3)	0.100 (4)	0.067 (3)	−0.024 (3)	0.027 (3)	−0.033 (3)
C26	0.081 (3)	0.065 (3)	0.081 (3)	−0.008 (2)	0.038 (3)	−0.019 (2)
C27	0.058 (2)	0.062 (2)	0.049 (2)	0.0018 (18)	0.0173 (17)	−0.0053 (18)
C15	0.0444 (19)	0.053 (2)	0.0371 (17)	−0.0041 (16)	0.0067 (14)	0.0016 (15)
C16	0.103 (3)	0.060 (3)	0.089 (3)	−0.021 (2)	−0.003 (3)	−0.018 (2)
C17	0.172 (6)	0.097 (4)	0.107 (4)	−0.066 (4)	0.041 (4)	−0.014 (3)
C11	0.0369 (16)	0.0462 (18)	0.0383 (17)	0.0006 (14)	0.0041 (13)	0.0021 (14)
S1'	0.0550 (18)	0.070 (2)	0.0428 (15)	−0.0058 (15)	0.0164 (13)	0.0081 (13)
C12'	0.078 (10)	0.074 (9)	0.065 (8)	−0.008 (7)	0.003 (7)	0.024 (6)
C13'	0.079 (8)	0.066 (7)	0.056 (6)	−0.008 (6)	−0.004 (6)	0.013 (6)
C14'	0.064 (7)	0.068 (6)	0.033 (5)	−0.017 (5)	0.010 (5)	0.013 (5)
S1	0.0577 (16)	0.0709 (19)	0.080 (3)	0.0160 (13)	0.0030 (15)	0.0266 (15)
C12	0.068 (8)	0.065 (6)	0.079 (8)	−0.004 (6)	0.003 (6)	0.011 (5)
C13	0.053 (5)	0.065 (6)	0.065 (6)	−0.002 (4)	0.006 (5)	0.004 (5)
C14	0.050 (5)	0.064 (5)	0.063 (6)	0.005 (4)	0.002 (4)	0.016 (5)
C18	0.050 (2)	0.048 (2)	0.0340 (17)	0.0095 (16)	0.0014 (14)	−0.0020 (15)
S2	0.103 (2)	0.0537 (14)	0.0784 (15)	0.0134 (14)	0.0120 (14)	0.0163 (12)
C19	0.067 (6)	0.057 (5)	0.068 (6)	0.048 (4)	0.005 (5)	−0.010 (4)
C20	0.059 (4)	0.065 (5)	0.074 (5)	0.042 (4)	0.001 (3)	−0.019 (4)
C21	0.090 (6)	0.059 (5)	0.070 (5)	0.039 (5)	−0.022 (5)	−0.009 (4)

S2'	0.064 (3)	0.084 (4)	0.108 (4)	0.010 (3)	0.029 (3)	−0.027 (3)
C19'	0.113 (11)	0.090 (11)	0.126 (13)	0.074 (9)	0.018 (9)	−0.014 (9)
C20'	0.105 (10)	0.074 (7)	0.113 (10)	0.073 (8)	0.006 (8)	−0.008 (7)
C21'	0.101 (10)	0.084 (10)	0.107 (11)	0.061 (10)	0.005 (8)	−0.020 (9)
O1	0.0706 (16)	0.0788 (18)	0.0416 (14)	−0.0247 (14)	0.0177 (12)	0.0002 (12)
O2	0.0443 (14)	0.0747 (18)	0.0811 (18)	−0.0079 (13)	−0.0092 (13)	−0.0116 (14)
O3	0.0656 (16)	0.0458 (14)	0.0709 (17)	−0.0045 (12)	0.0003 (13)	−0.0055 (12)

Geometric parameters (Å, º)

C7—O1	1.212 (4)	C16—H16B	0.9700
C7—C6	1.473 (4)	C17—H17A	0.9600
C7—C8	1.544 (4)	C17—H17B	0.9600
C6—C5	1.390 (4)	C17—H17C	0.9600
C6—C1	1.397 (4)	C11—C12	1.371 (9)
C5—C4	1.377 (5)	C11—C12'	1.385 (9)
C5—H5	0.9300	C11—S1'	1.686 (4)
C4—C3	1.375 (5)	C11—S1	1.700 (4)
C4—H4	0.9300	S1'—C14'	1.698 (9)
C3—C2	1.375 (5)	C12'—C13'	1.487 (10)
C3—H3	0.9300	C12'—H12'	0.9300
C2—C1	1.395 (4)	C13'—C14'	1.365 (9)
C2—H2	0.9300	C13'—H13'	0.9300
C1—C10	1.476 (4)	C14'—H14'	0.9300
C10—C9	1.341 (4)	S1—C14	1.696 (8)
C10—C11	1.476 (4)	C12—C13	1.481 (9)
C9—C15	1.497 (4)	C12—H12	0.9300
C9—C8	1.524 (4)	C13—C14	1.353 (8)
C8—C18	1.523 (4)	C13—H13	0.9300
C8—C22	1.536 (4)	C14—H14	0.9300
C22—C27	1.395 (5)	C18—C19	1.387 (9)
C22—C23	1.427 (4)	C18—C19'	1.397 (10)
C23—C24	1.368 (5)	C18—S2'	1.648 (6)
C23—H23	0.9300	C18—S2	1.690 (4)
C24—C25	1.335 (6)	S2—C21	1.720 (7)
C24—H24	0.9300	C19—C20	1.496 (9)
C25—C26	1.346 (6)	C19—H19	0.9300
C25—H25	0.9300	C20—C21	1.382 (8)
C26—C27	1.361 (5)	C20—H20	0.9300
C26—H26	0.9300	C21—H21	0.9300
C27—H27	0.9300	S2'—C21'	1.713 (9)

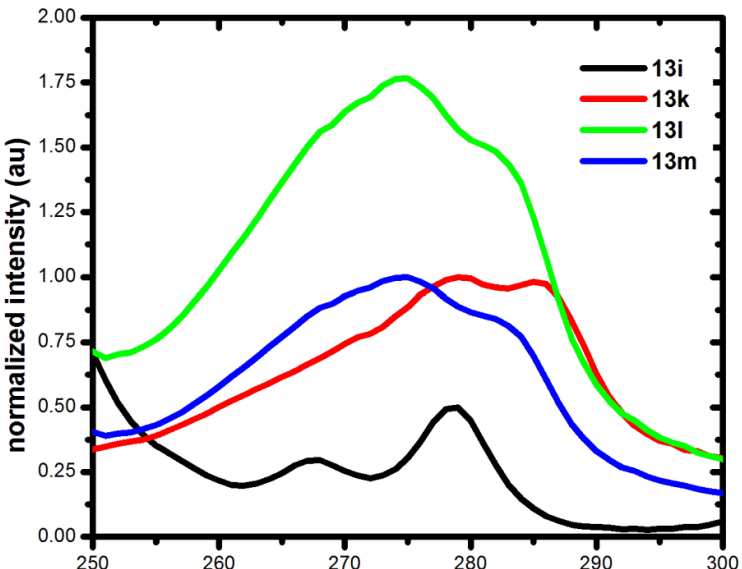
C15—O2	1.192 (4)	C19'—C20'	1.500 (10)
C15—O3	1.328 (4)	C19'—H19'	0.9300
C16—C17	1.437 (6)	C20'—C21'	1.397 (10)
C16—O3	1.459 (4)	C20'—H20'	0.9300
C16—H16A	0.9700	C21'—H21'	0.9300
O1—C7—C6	122.3 (3)	H17A—C17—H17C	109.5
O1—C7—C8	120.5 (3)	H17B—C17—H17C	109.5
C6—C7—C8	117.1 (3)	C12—C11—C12'	107.7 (12)
C5—C6—C1	120.2 (3)	C12—C11—C10	126.8 (8)
C5—C6—C7	119.2 (3)	C12'—C11—C10	125.4 (9)
C1—C6—C7	120.6 (3)	C12—C11—S1'	7.3 (8)
C4—C5—C6	120.4 (3)	C12'—C11—S1'	114.7 (8)
C4—C5—H5	119.8	C10—C11—S1'	119.7 (3)
C6—C5—H5	119.8	C12—C11—S1	110.1 (7)
C3—C4—C5	119.5 (3)	C12'—C11—S1	4.7 (13)
C3—C4—H4	120.2	C10—C11—S1	123.1 (3)
C5—C4—H4	120.2	S1'—C11—S1	117.3 (3)
C2—C3—C4	120.9 (3)	C11—S1'—C14'	88.4 (8)
C2—C3—H3	119.5	C11—C12'—C13'	112.1 (17)
C4—C3—H3	119.5	C11—C12'—H12'	123.9
C3—C2—C1	120.5 (3)	C13'—C12'—H12'	123.9
C3—C2—H2	119.7	C14'—C13'—C12'	105 (2)
C1—C2—H2	119.7	C14'—C13'—H13'	127.6
C2—C1—C6	118.4 (3)	C12'—C13'—H13'	127.6
C2—C1—C10	122.0 (3)	C13'—C14'—S1'	119.9 (16)
C6—C1—C10	119.5 (3)	C13'—C14'—H14'	120.1
C9—C10—C11	120.3 (3)	S1'—C14'—H14'	120.1
C9—C10—C1	120.9 (3)	C14—S1—C11	90.1 (8)
C11—C10—C1	118.7 (2)	C11—C12—C13	117.2 (15)
C10—C9—C15	121.2 (3)	C11—C12—H12	121.4
C10—C9—C8	123.0 (3)	C13—C12—H12	121.4
C15—C9—C8	115.7 (2)	C14—C13—C12	102.5 (18)
C18—C8—C9	109.6 (2)	C14—C13—H13	128.8
C18—C8—C22	113.7 (2)	C12—C13—H13	128.8
C9—C8—C22	109.8 (2)	C13—C14—S1	120.1 (16)
C18—C8—C7	102.9 (2)	C13—C14—H14	119.9
C9—C8—C7	110.9 (2)	S1—C14—H14	120.0
C22—C8—C7	109.8 (2)	C19—C18—C19'	120.6 (10)
C27—C22—C23	117.6 (3)	C19—C18—C8	122.0 (6)
C27—C22—C8	118.1 (3)	C19'—C18—C8	117.3 (9)

C23—C22—C8	124.3 (3)	C19—C18—S2'	6.9 (10)
C24—C23—C22	117.8 (4)	C19'—C18—S2'	114.8 (9)
C24—C23—H23	121.1	C8—C18—S2'	127.8 (4)
C22—C23—H23	121.1	C19—C18—S2	114.4 (6)
C25—C24—C23	122.7 (4)	C19'—C18—S2	6.4 (10)
C25—C24—H24	118.6	C8—C18—S2	123.5 (3)
C23—C24—H24	118.6	S2'—C18—S2	108.6 (4)
C24—C25—C26	120.7 (4)	C18—S2—C21	94.5 (5)
C24—C25—H25	119.7	C18—C19—C20	106.9 (10)
C26—C25—H25	119.7	C18—C19—H19	126.6
C25—C26—C27	120.1 (4)	C20—C19—H19	126.6
C25—C26—H26	120.0	C21—C20—C19	115.9 (11)
C27—C26—H26	120.0	C21—C20—H20	122.0
C26—C27—C22	121.2 (4)	C19—C20—H20	122.0
C26—C27—H27	119.4	C20—C21—S2	108.2 (10)
C22—C27—H27	119.4	C20—C21—H21	125.9
O2—C15—O3	124.6 (3)	S2—C21—H21	125.9
O2—C15—C9	124.1 (3)	C18—S2'—C21'	98.3 (11)
O3—C15—C9	111.3 (3)	C18—C19'—C20'	103.3 (18)
C17—C16—O3	110.7 (4)	C18—C19'—H19'	128.3
C17—C16—H16A	109.5	C20'—C19'—H19'	128.3
O3—C16—H16A	109.5	C21'—C20'—C19'	121 (2)
C17—C16—H16B	109.5	C21'—C20'—H20'	119.6
O3—C16—H16B	109.5	C19'—C20'—H20'	119.6
H16A—C16—H16B	108.1	C20'—C21'—S2'	103 (2)
C16—C17—H17A	109.5	C20'—C21'—H21'	128.7
C16—C17—H17B	109.5	S2'—C21'—H21'	128.7
H17A—C17—H17B	109.5	C15—O3—C16	117.2 (3)
C16—C17—H17C	109.5		
O1—C7—C6—C5	−16.1 (5)	C1—C10—C11—S1	69.7 (5)
C8—C7—C6—C5	160.0 (3)	C12—C11—S1'—C14'	13 (9)
O1—C7—C6—C1	162.8 (3)	C12'—C11—S1'—C14'	−2.9 (18)
C8—C7—C6—C1	−21.1 (4)	C10—C11—S1'—C14'	−177.9 (10)
C1—C6—C5—C4	0.9 (5)	S1—C11—S1'—C14'	1.5 (11)
C7—C6—C5—C4	179.8 (3)	C12—C11—C12'—C13'	1 (3)
C6—C5—C4—C3	−0.5 (5)	C10—C11—C12'—C13'	177.9 (17)
C5—C4—C3—C2	−0.2 (6)	S1'—C11—C12'—C13'	3 (3)
C4—C3—C2—C1	0.4 (5)	S1—C11—C12'—C13'	−121 (16)
C3—C2—C1—C6	0.0 (5)	C11—C12'—C13'—C14'	−2 (3)
C3—C2—C1—C10	−178.5 (3)	C12'—C13'—C14'—S1'	0 (3)

C5—C6—C1—C2	−0.7 (4)	C11—S1'—C14'—C13'	2 (2)
C7—C6—C1—C2	−179.6 (3)	C12—C11—S1—C14	−1.9 (16)
C5—C6—C1—C10	177.8 (3)	C12'—C11—S1—C14	58 (14)
C7—C6—C1—C10	−1.0 (4)	C10—C11—S1—C14	178.9 (10)
C2—C1—C10—C9	−171.3 (3)	S1'—C11—S1—C14	−0.4 (12)
C6—C1—C10—C9	10.3 (4)	C12'—C11—C12—C13	−1 (3)
C2—C1—C10—C11	7.2 (4)	C10—C11—C12—C13	−177.5 (17)
C6—C1—C10—C11	−171.2 (3)	S1'—C11—C12—C13	−166 (11)
C11—C10—C9—C15	3.0 (4)	S1—C11—C12—C13	3 (3)
C1—C10—C9—C15	−178.6 (3)	C11—C12—C13—C14	−3 (3)
C11—C10—C9—C8	−174.3 (3)	C12—C13—C14—S1	2 (3)
C1—C10—C9—C8	4.2 (4)	C11—S1—C14—C13	0 (2)
C10—C9—C8—C18	88.2 (3)	C9—C8—C18—C19	15.8 (11)
C15—C9—C8—C18	−89.2 (3)	C22—C8—C18—C19	−107.5 (11)
C10—C9—C8—C22	−146.3 (3)	C7—C8—C18—C19	133.8 (11)
C15—C9—C8—C22	36.3 (3)	C9—C8—C18—C19'	−165.8 (16)
C10—C9—C8—C7	−24.7 (4)	C22—C8—C18—C19'	70.9 (16)
C15—C9—C8—C7	157.9 (3)	C7—C8—C18—C19'	−47.8 (16)
O1—C7—C8—C18	91.3 (3)	C9—C8—C18—S2'	11.2 (6)
C6—C7—C8—C18	−84.9 (3)	C22—C8—C18—S2'	−112.1 (6)
O1—C7—C8—C9	−151.7 (3)	C7—C8—C18—S2'	129.2 (6)
C6—C7—C8—C9	32.1 (4)	C9—C8—C18—S2	−167.7 (3)
O1—C7—C8—C22	−30.1 (4)	C22—C8—C18—S2	69.1 (4)
C6—C7—C8—C22	153.7 (3)	C7—C8—C18—S2	−49.7 (3)
C18—C8—C22—C27	178.5 (3)	C19—C18—S2—C21	−0.4 (11)
C9—C8—C22—C27	55.3 (4)	C19'—C18—S2—C21	168 (14)
C7—C8—C22—C27	−66.9 (4)	C8—C18—S2—C21	−177.2 (5)
C18—C8—C22—C23	0.1 (4)	S2'—C18—S2—C21	3.8 (6)
C9—C8—C22—C23	−123.0 (3)	C19'—C18—C19—C20	0 (2)
C7—C8—C22—C23	114.8 (3)	C8—C18—C19—C20	178.7 (8)
C27—C22—C23—C24	−0.1 (5)	S2'—C18—C19—C20	−33 (9)
C8—C22—C23—C24	178.2 (3)	S2—C18—C19—C20	1.9 (16)
C22—C23—C24—C25	0.3 (6)	C18—C19—C20—C21	−3.1 (19)
C23—C24—C25—C26	0.2 (7)	C19—C20—C21—S2	2.8 (15)
C24—C25—C26—C27	−0.9 (7)	C18—S2—C21—C20	−1.4 (9)
C25—C26—C27—C22	1.1 (6)	C19—C18—S2'—C21'	146 (10)
C23—C22—C27—C26	−0.6 (5)	C19'—C18—S2'—C21'	−3 (2)
C8—C22—C27—C26	−179.0 (3)	C8—C18—S2'—C21'	−179.9 (11)
C10—C9—C15—O2	−119.3 (4)	S2—C18—S2'—C21'	−0.9 (12)
C8—C9—C15—O2	58.2 (4)	C19—C18—C19'—C20'	−1 (3)
C10—C9—C15—O3	62.5 (4)	C8—C18—C19'—C20'	−179.8 (15)

C8—C9—C15—O3	−120.1 (3)	S2'—C18—C19'—C20'	3 (3)
C9—C10—C11—C12	69.2 (13)	S2—C18—C19'—C20'	−14 (12)
C1—C10—C11—C12	−109.3 (12)	C18—C19'—C20'—C21'	−2 (4)
C9—C10—C11—C12'	−106.8 (15)	C19'—C20'—C21'—S2'	0 (4)
C1—C10—C11—C12'	74.7 (15)	C18—S2'—C21'—C20'	1 (2)
C9—C10—C11—S1'	67.6 (4)	O2—C15—O3—C16	3.7 (5)
C1—C10—C11—S1'	−111.0 (4)	C9—C15—O3—C16	−178.1 (3)
C9—C10—C11—S1	−111.7 (4)	C17—C16—O3—C15	96.7 (5)

Absorption spectra of tri-substituted *α*-naphthols **13i, 13k, 13l** and **13m**



Emission spectra of tri-substituted α-naphthols **13i, 13k, 13l** and **13m**