

Design and Effective Synthesis of the First 4-Aza-2,3-didehydropodophyllotoxin Rigid Aminologue: a *N*-methyl-4-[(3,4,5-trimethoxyphenyl)amino)]-1,2-dihydroquinoline-lactone

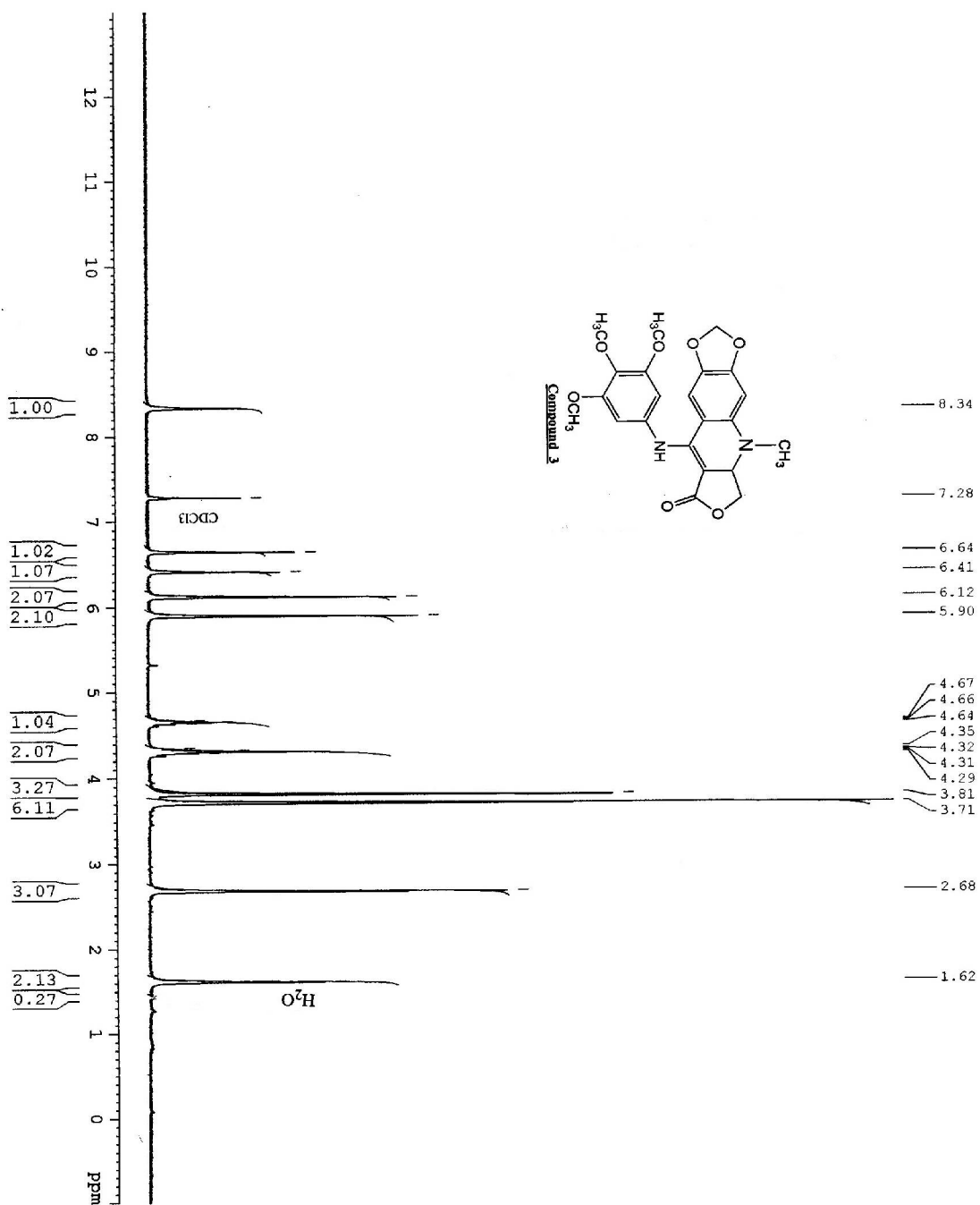
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Current Data Parameters

NAME	sgm130.2
EXNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20071107
Time	15:07
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	2
DS	4
SWH	6172.833 Hz
FIDRES	0.094190 Hz
AQ	5.308460 sec
RG	812.7
DM	81.000 usec
DE	3.000
TE	300.0
TD0	1.00000000 sec

CHANNEL f1

NUC1	1H
PL1	8.50 usec
PL1	-5.00 dB
SEOL	300.321534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300000 MHz
WDW	no
SSB	0
CB	0.00 Hz
SC	1.00

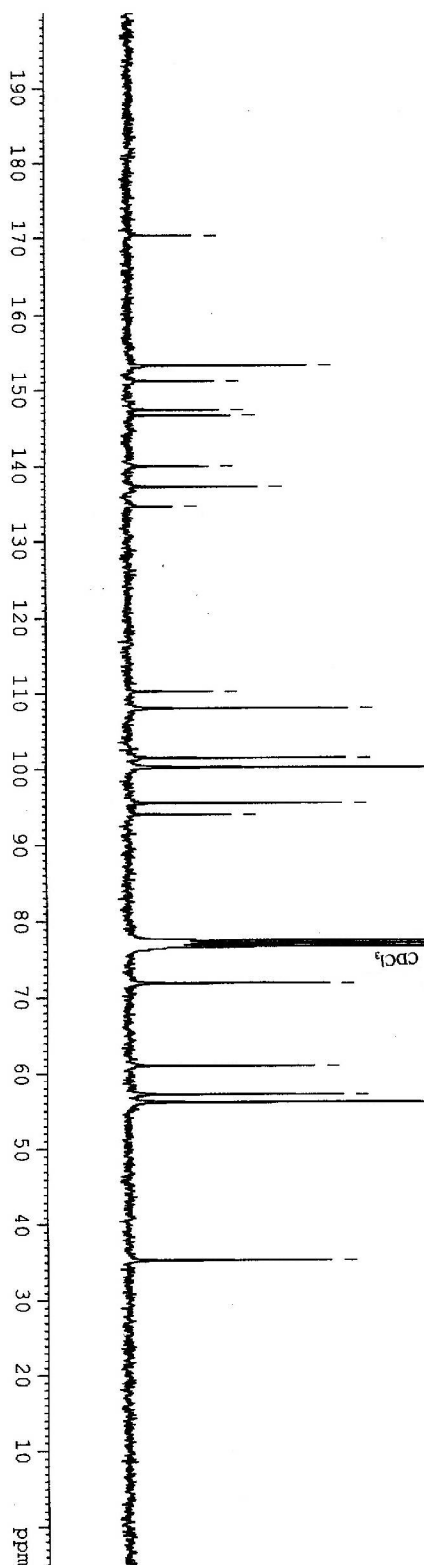
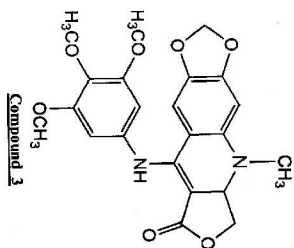
Current Data Parameters
NAME: nmr compound3
EXPNO: 2
PROCNO: 1
F2 - Acquisition Parameters
Date_ : 20070414
Time : 4.14
INSTRUM : spect
PROBHD : 5 mm QNP 1H/13
PULPROG : zgpg30
TD : 32768
SOLVENT : CDCl3
NS : 1226
DS : 4
SWH : 22679.736 Hz
FIDRES : 0.692009 Hz
AQ : 0.725804 sec
RG : 320.00
DW : 22.050 usec
DE : 9.50 usec
TE : 300.2 K
D1 : 2.50000000 sec
d11 : 0.03000000 sec
DELTA : 2.40000010 sec
TD0 : 1

===== CHANNEL f1 =====
NUC1 : 13C
P1 : 12.00 usec
PL1 : -2.00 dB
SFO1 : 75.4768051 MHz

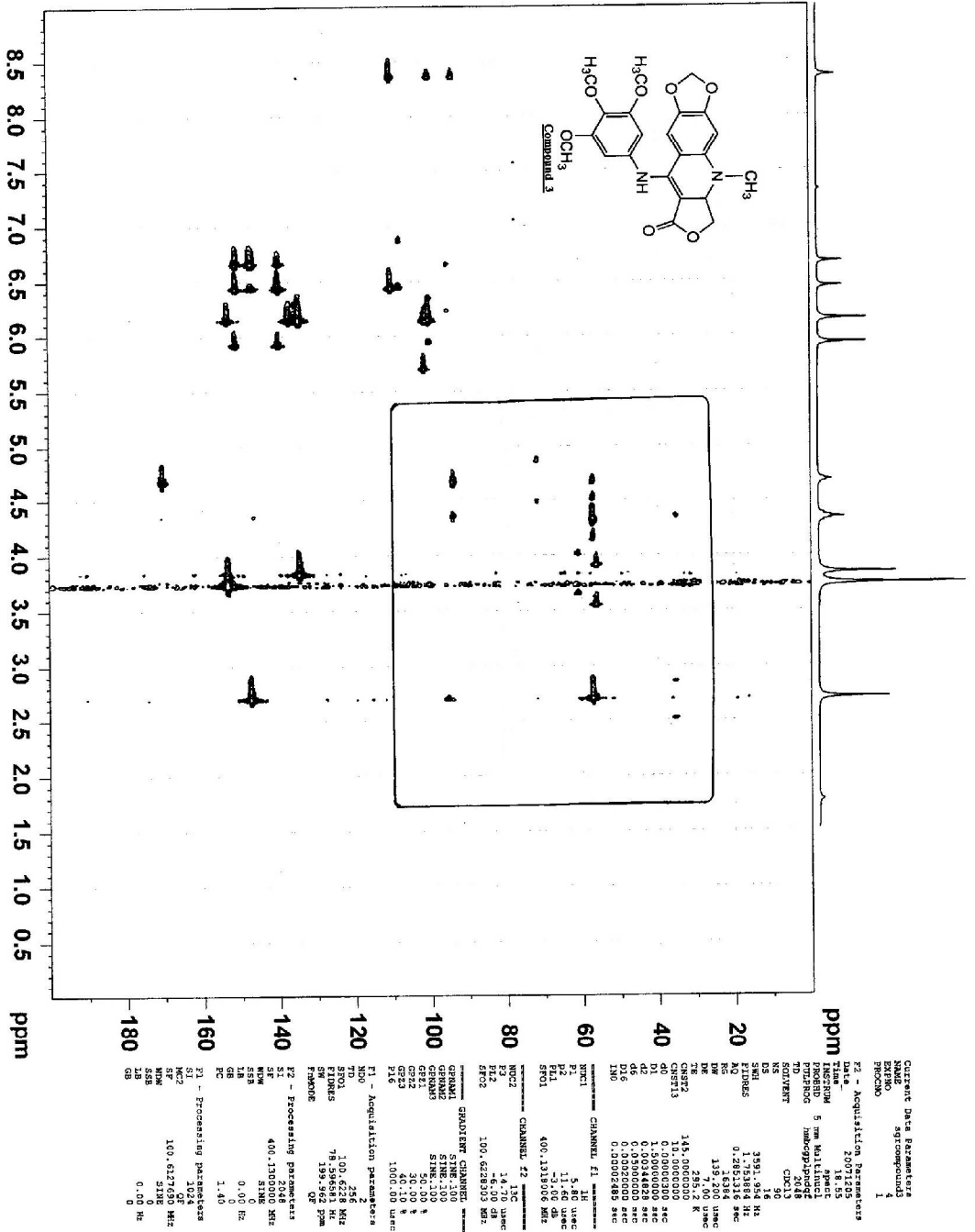
===== CHANNEL f2 =====
CPDPRG2 : waltz16
NUC2 : 1H
P2 : 100.00 usec
PL2 : -5.00 dB
PL12 : 15.00 dB
PL13 : 15.00 dB
SFO2 : 300.1350000 MHz

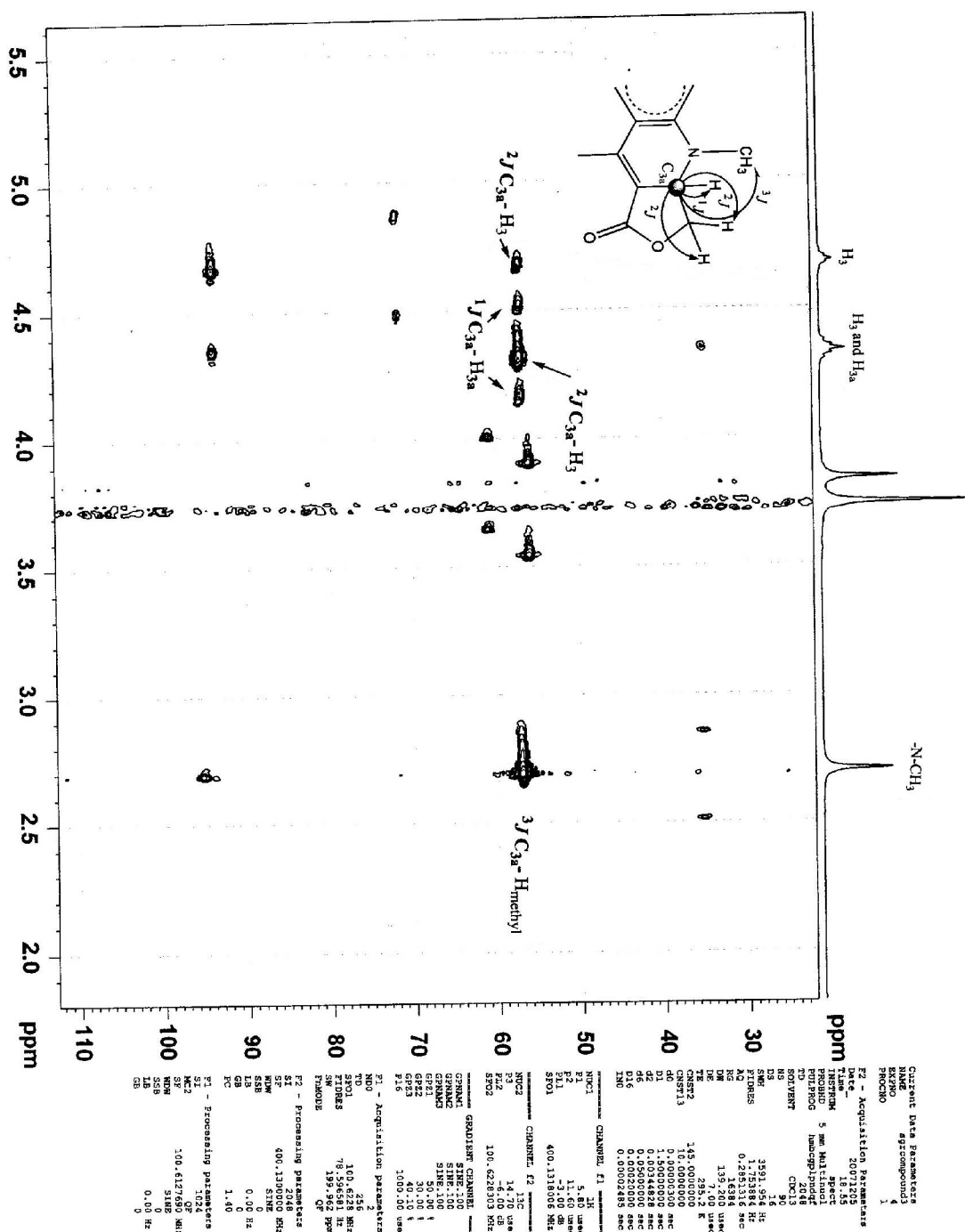
F2 - Processing parameters
SI : 32768
SF : 75.4677490 MHz
WDW : EM
SSB : 0
GB : 0
PC : 1.40

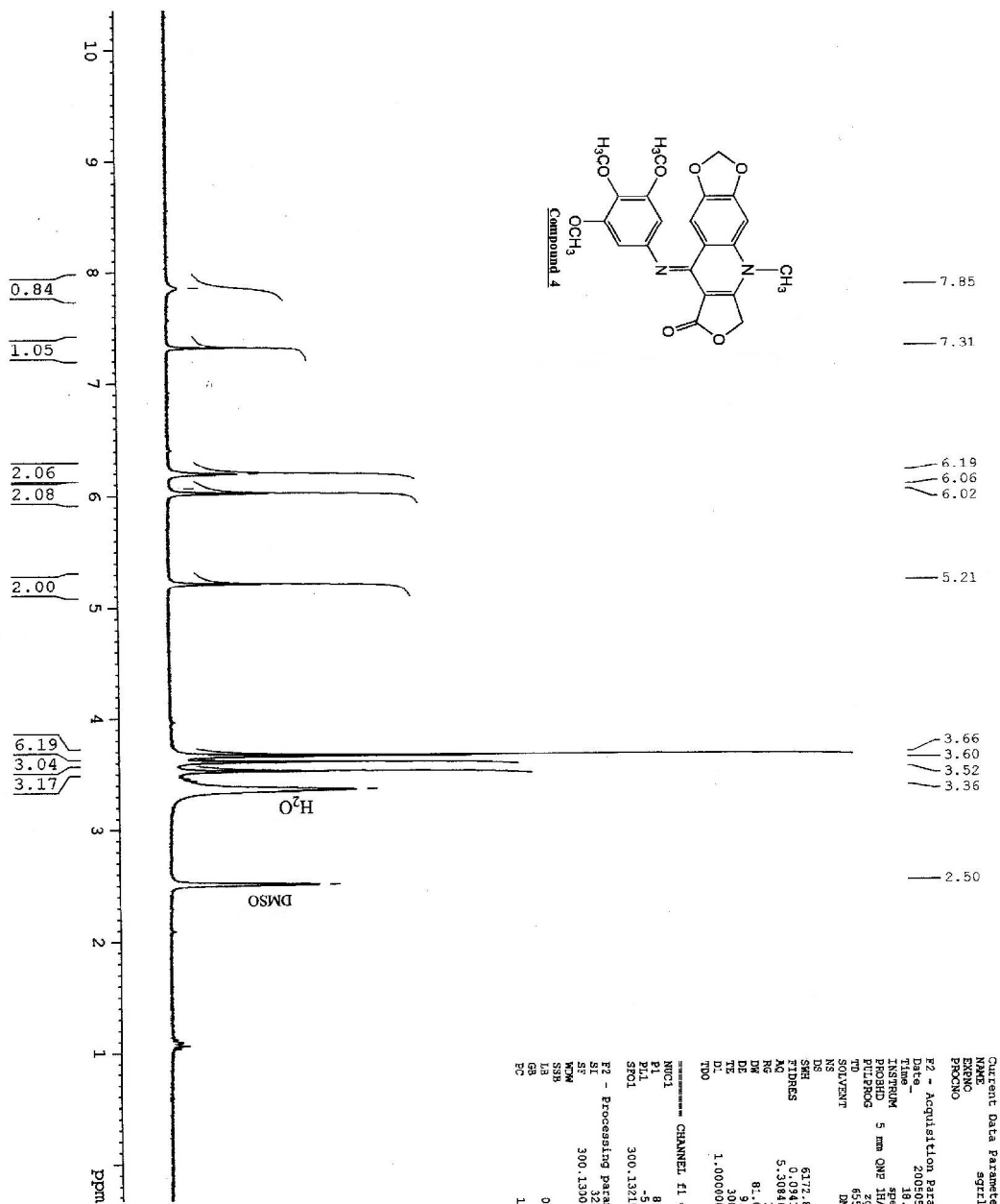
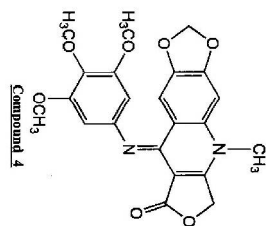
170.44
153.34
151.10
147.38
146.64
139.98
137.23
134.64
110.27
108.03
101.47
100.24
95.40
93.94
77.49
77.06
76.64
71.88
61.03
57.14
56.11
35.26



S-4





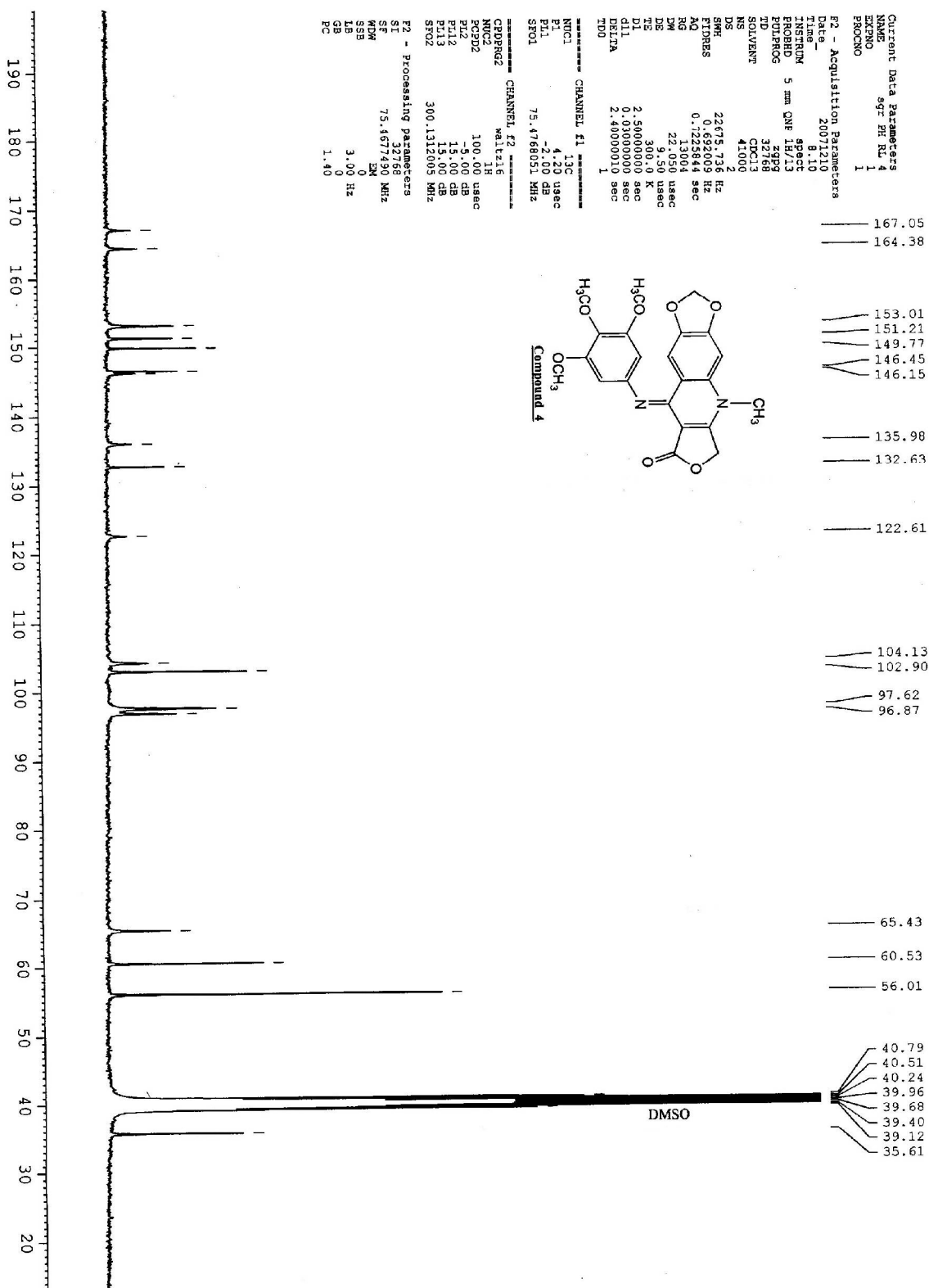
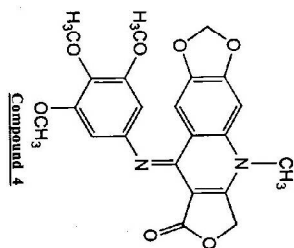


Current Data Parameters
NAME Sqr PE RL 4
EXRNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071210
Time_ 1410
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 41000
DS 2
SWH 22675.726 Hz
FIDRES 0.165410 Hz
AQ 0.122564 sec
RG 13104
DM 22.050 usec
DE 9.50 usec
TE 300.0 K
D1 2.50000000 sec
d11 0.00000000 sec
DELTA 2.40000010 sec
TD0 1

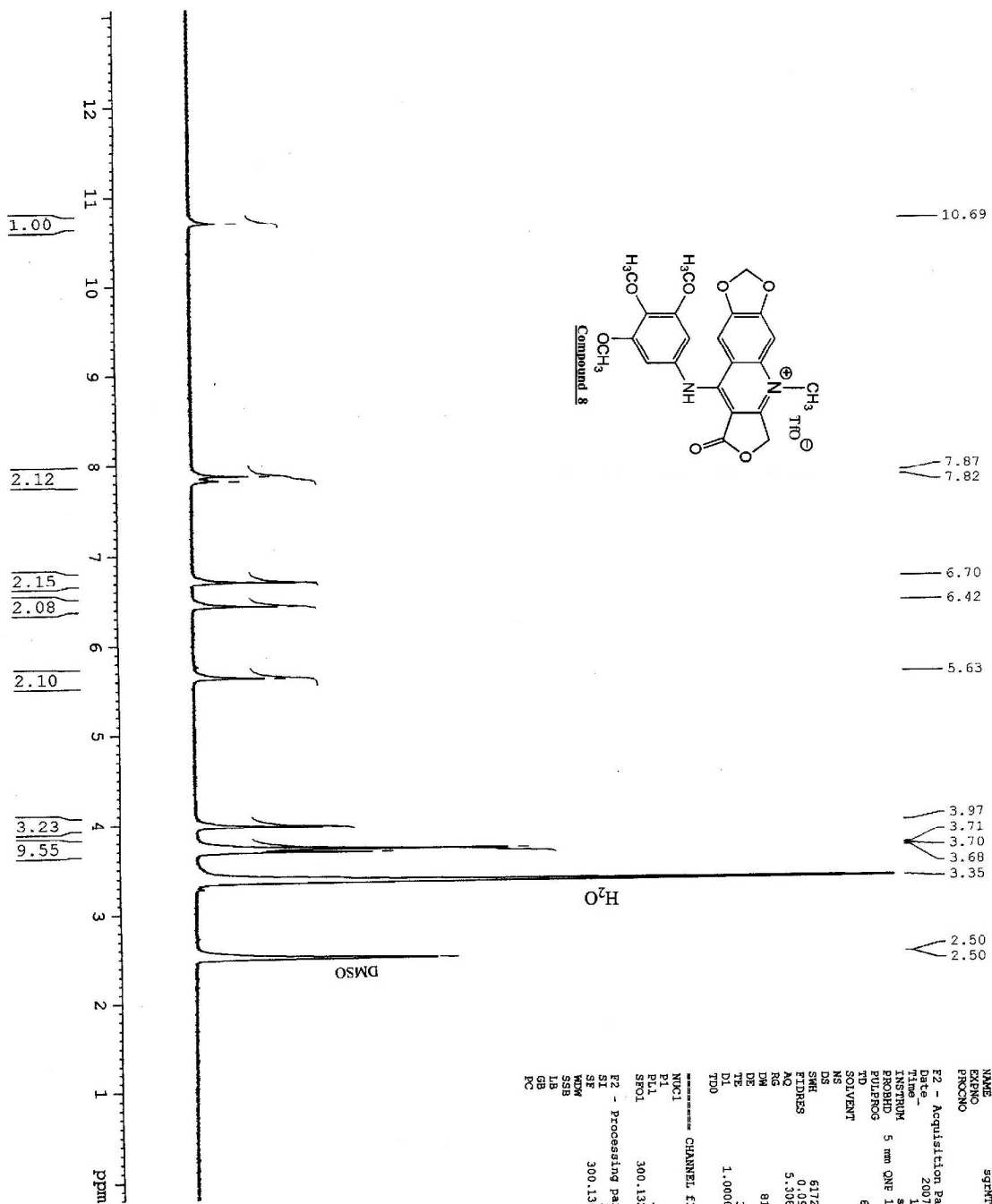
CHANNEL F1
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.476051 MHz

CHANNEL F2
CPDPRG2 waltz16
NUC2 13C
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
S2 2048
SF 75.4677430 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40







Current Data Parameters
NAME sgr compound8
EXFNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071207
Time 7.02

INSTRUM 5 mm QNP 1H/13
PROBHD spect
PULPROG zgpg

TD 32768
SOLVENT CDCl3
NS 13000

DS 2
SMH 22675.736 Hz
AQ 0.692009 Hz

RG 0.7225844 sec
DE 13004

TE 22.050 usec
DE 9.50 usec

DI 300.0 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4768051 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz

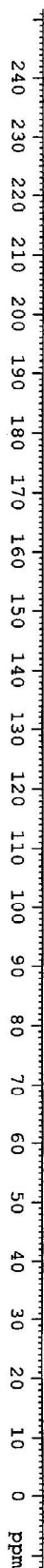
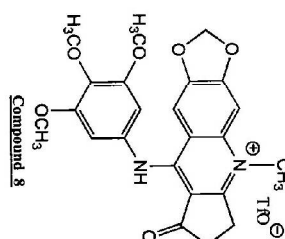
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
MDW EM
SSB 0
LB 0
GB 0
PC 1.40

165.90
163.99
155.66
153.59
150.95
148.63
140.03
136.92
135.97

115.53
105.08
102.38
102.21
100.67
98.04

67.27
60.71
56.67

38.60



Current Data Parameters
 NAME sqcompound9
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071126
 Time 5.30

INSTRUM spect
 PROBD 5 mm QNP 1H/13
 PULPROG zgpg

TD 32768
 SOLVENT DMSO
 NS 65000

DS 2
 SWH 22675.736 Hz
 FIDRES 0.692009 Hz

AQ 0.7225844 sec
 RG 13004
 DW 22.050 usec

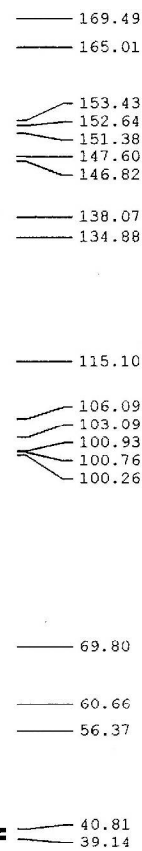
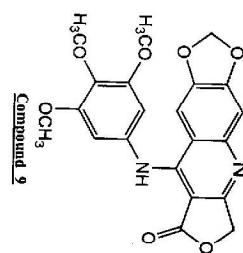
DE 9.50 usec
 TE 300.0 K
 D1 2.50000000 sec

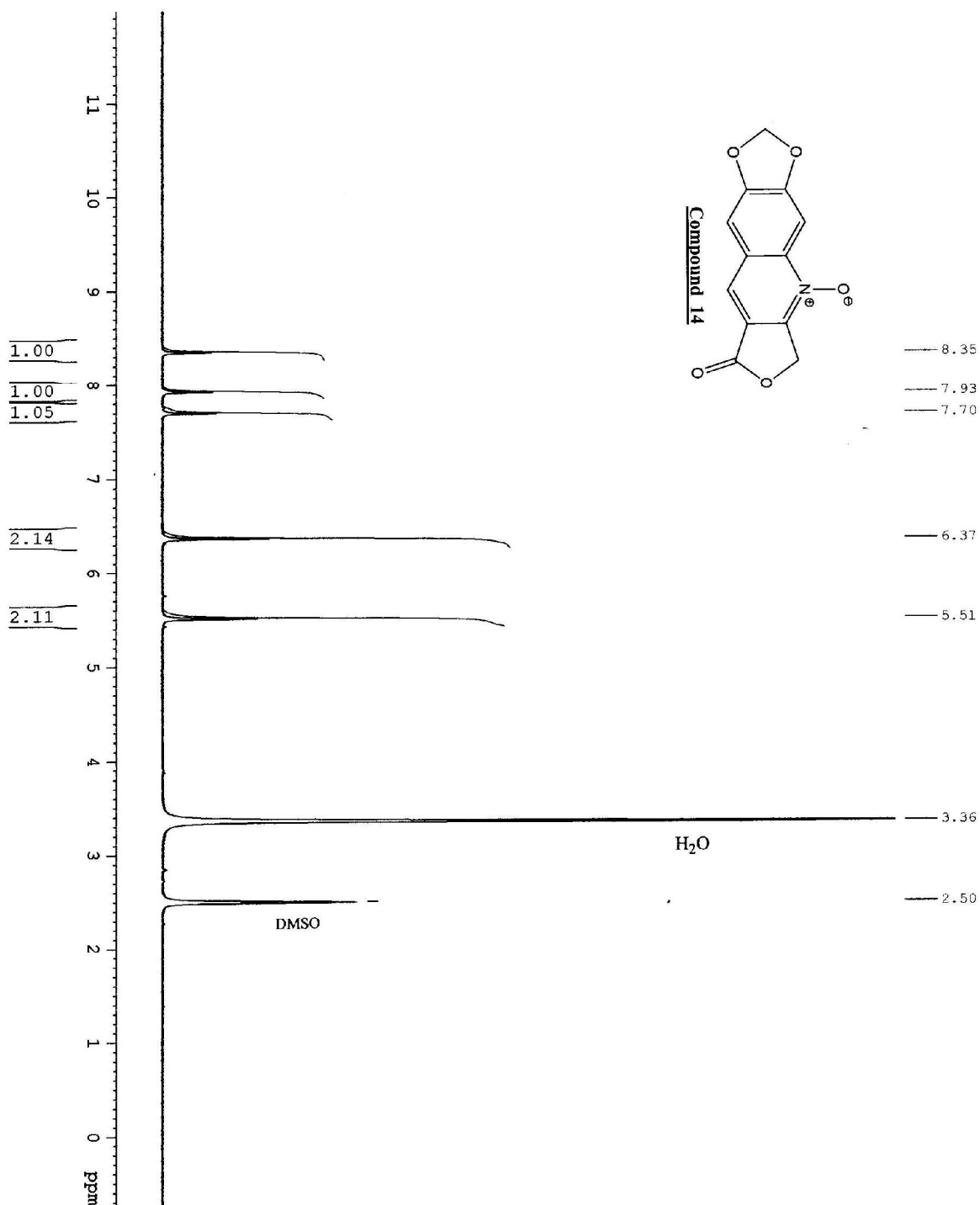
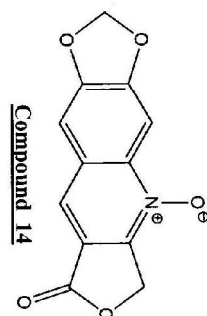
d11 0.03000000 sec
 DELTA 2.40000010 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 4.20 usec
 PL1 -2.00 dB
 SFO1 75.4768051 MHz

===== CHANNEL f2 =====
 CEPRG2 wait16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 -5.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677490 MHz
 WDM EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40





Current Data Parameters
 Name: 5gcomp14
 EXNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20070828
 Time: 11.12
 INSTRUM: spect
 PROBD: 5 mm QNP 1H/13
 PULPROG: zg30
 TD: 65536
 SOLVENT: DMSO
 NS: 18
 DS: 2
 SWH: 6172.833 Hz
 FIDRES: 0.004138 Hz
 AQ: 5.308460 sec
 RG: 574.7
 DE: 81.000 usec
 TE: 300.0 K
 D1: 1.00000000 sec
 TDO: 1

CHANNEL f1
 NUCL1: 1H
 P1: 8.50 usec
 PL1: -5.00 dB
 SFO1: 300.1321534 MHz

F2 - Processing parameters
 SI: 32768
 SF: 300.1300000 MHz
 WDW: TO
 SSB: 0
 LB: 0.00 Hz
 GB: 0
 PC: 1.00

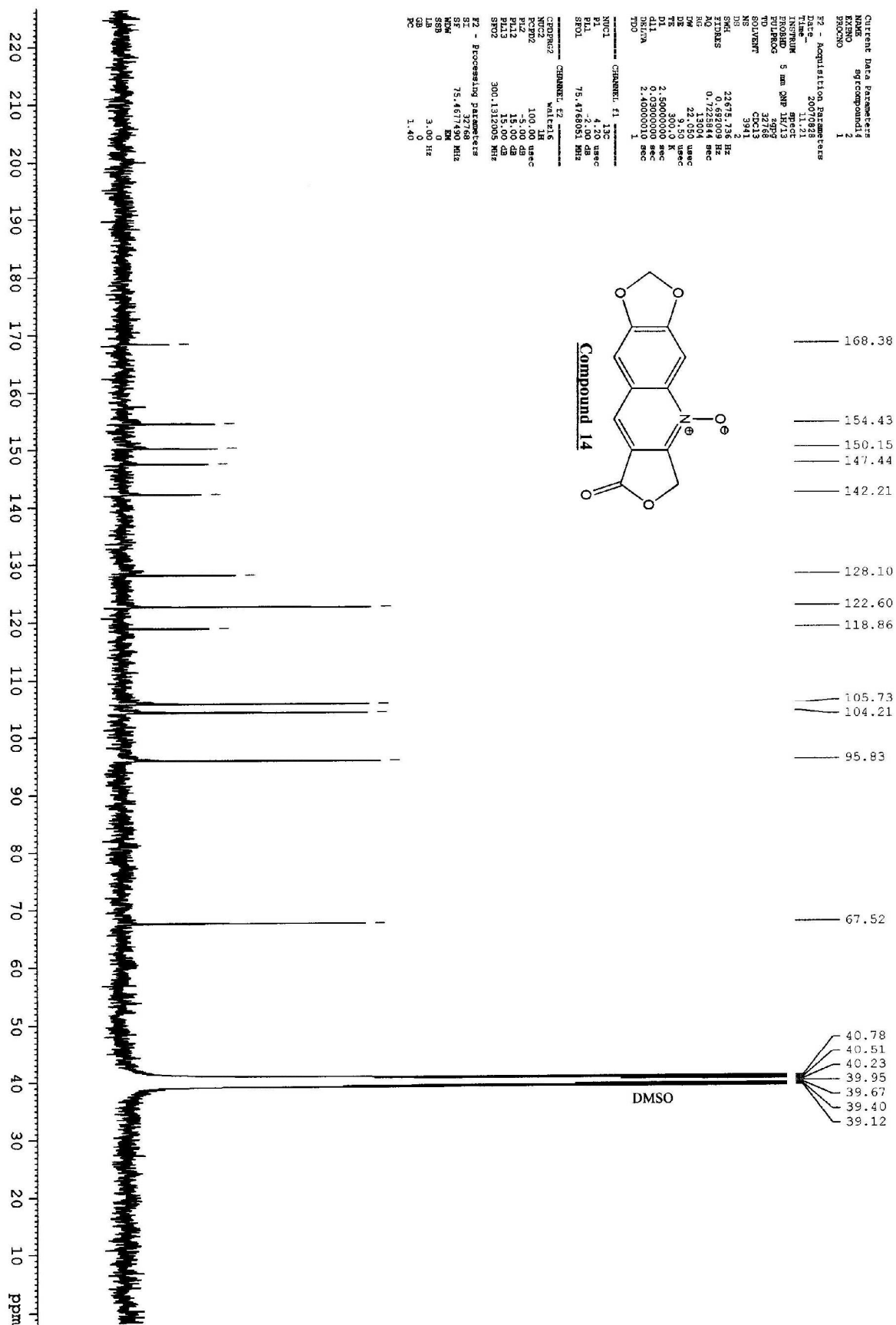
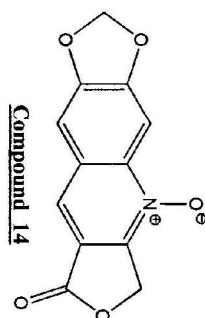
Current Data Parameters
NAME agccompound14
EXNO 2
PROCNO 1

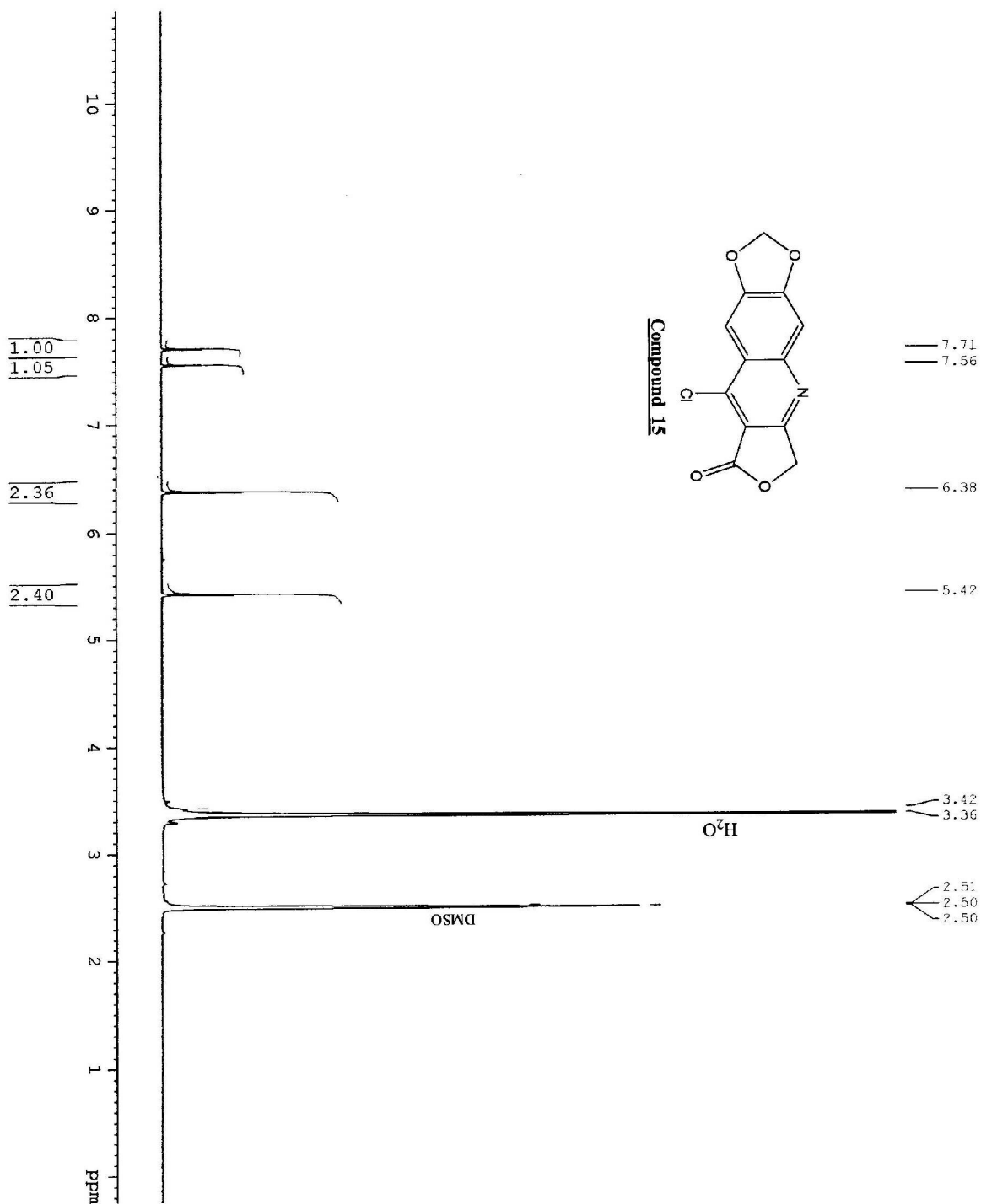
F2 - Acquisition Parameters
Date_ 20070828
Time 11.21
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3544
DS 2
SWH 22575.736 Hz
FIDRES 0.692609 Hz
AQ 0.722544 sec
RG 1304
ZG 22.050 usec
DE 3.00 usec
TE 300.2 K
D1 2.5000000 sec
d11 0.0300000 sec
DELTA 2.4000010 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 4.00 usec
PL 0.00 dB
SFO1 75.476051 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
S 512
SF 75.467490 MHz
WDW EM
SSB 0
GB 0
PC 1.40





Current Data Parameters

NAME	sglcompound15
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20070829
Time	10.58
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	128
DS	2
SWH	6172.833 Hz
FIDRES	0.004130 Hz
AQ	5.3084660 sec
RG	645.1
DE	81.000 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

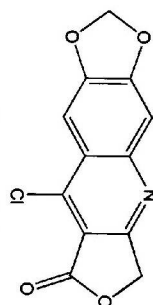
CHANNEL f1

NUC1	1H
FI	8.50 usec
FL1	-5.90 dB
SFO1	300.1321534 MHz

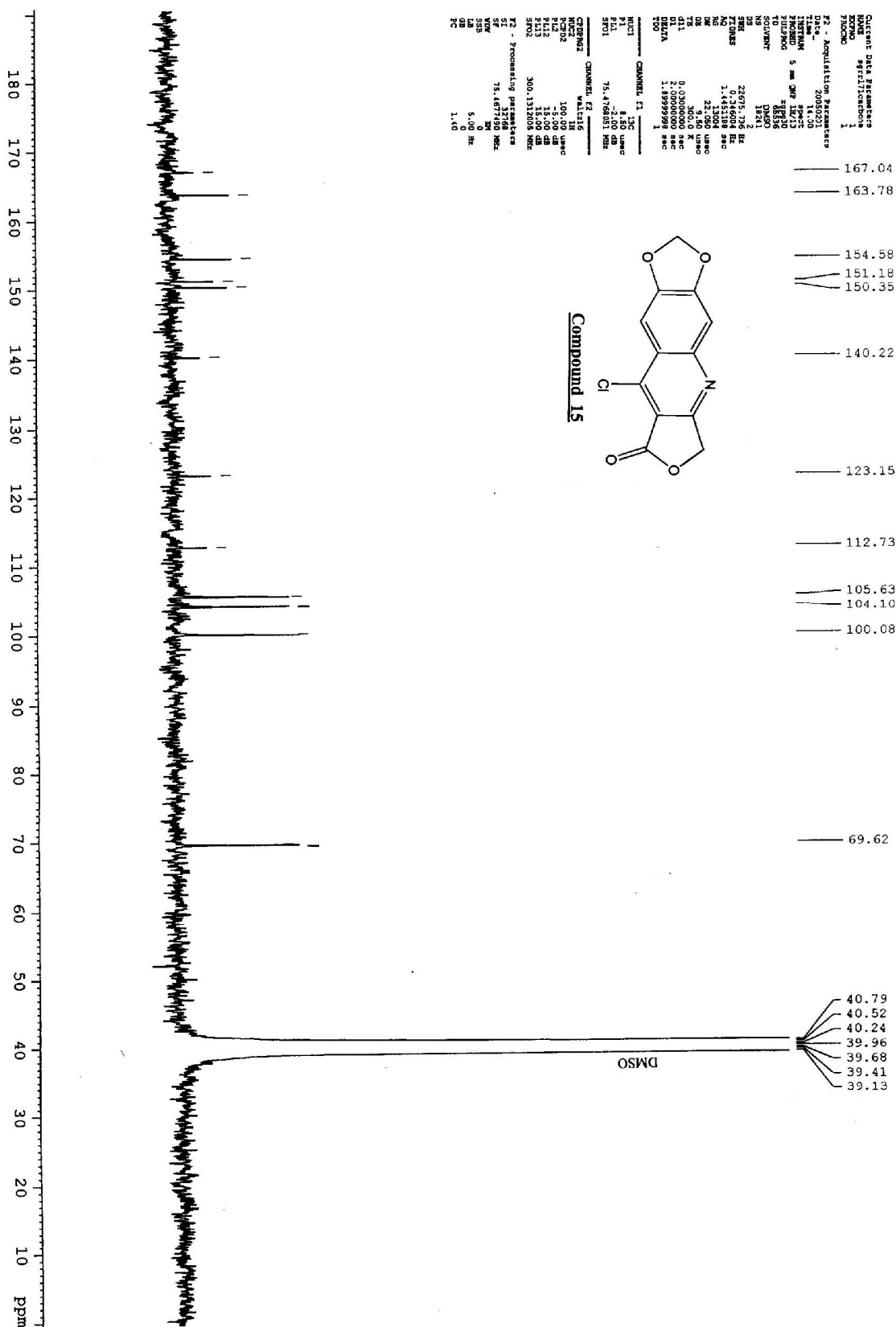
F2 - Processing parameters

SI	32768
SF	300.130000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

Current Data Parameters
 Name: 2-ethyl-2H-chromene
 Date: 20080201
 Time: 14.30
 INSTRUM: spectrometer
 PROBHD: 5 mm QNP 1H/13
 PULPROG: zgpg30
 FIDRES: 0.146004 Hz
 SOLVENT: DMSO
 NS: 18241
 DS: 4
 SWH: 22675.76 Hz
 F2 - Acquisition Parameters
 Date: 20080201
 Time: 14.30
 INSTRUM: spectrometer
 PROBHD: 5 mm QNP 1H/13
 PULPROG: zgpg30
 FIDRES: 0.146004 Hz
 SOLVENT: DMSO
 NS: 18241
 DS: 4
 SWH: 22675.76 Hz
 F2 - Processing parameters
 SI: 32768
 SF: 125.761 MHz
 WID: 15.46713 MHz
 FWHM: 3.00 Hz
 AQ: 0.000000 sec
 SFO2: 300.1312008 MHz
 PC: 1.10



Compound 15



Complete IR peak listings for compounds 3 to 15

Compound 3

IR (KBr, $\nu_{\text{max}}/\text{cm}^{-1}$) 3280, 1697, 1641, 1588, 1508, 1299, 1231, 1209, 1129, 1036, 1004, 926, 862.

Compound 4

IR (KBr, $\nu_{\text{max}}/\text{cm}^{-1}$) 1772, 1645, 1585, 1499, 1482, 1465, 1448, 1412, 1350, 1269, 1226, 1182, 1124, 1030.

Compound 7

IR (KBr, $\nu_{\text{max}}/\text{cm}^{-1}$) 1763, 1625, 1613, 1498, 1465, 1359, 1248, 1208, 1182, 1031, 1001.

Compound 8

IR (KBr, $\nu_{\text{max}}/\text{cm}^{-1}$) 3260, 1756, 1655, 1624, 1597, 1581, 1545, 1503, 1475, 1434, 1384, 1344, 1263, 1223, 1166, 1123, 1034, 930, 863, 790.

Compound 9

IR (KBr, $\nu_{\text{max}}/\text{cm}^{-1}$) 3274, 1749, 1654, 1612, 1592, 1542, 1502, 1467, 1420, 1364, 1324, 1256, 1230, 1132, 1032, 1020.

Compound 14

IR (KBr, $\nu_{\text{max}}/\text{cm}^{-1}$) 1773, 1618, 1498, 1467, 1448, 1419, 1370, 1344, 1306, 1248, 1185, 1152, 1126, 1070, 1034, 1024, 991, 935, 908, 875, 758, 657, 609, 542.

Compound 15

IR (KBr, $\nu_{\text{max}}/\text{cm}^{-1}$) 1774, 1618, 1598, 1508, 1501, 1475, 1366, 1332, 1275, 1259, 1151, 1114, 1042, 1021, 942, 878, 784.