

Manganese ferrite nanoparticles catalyzed tandem and green synthesis of spirooxindoles

H. Naeimi,^a Z. Rashid,^a A. H. Zarnani,^b and R. Ghahremanzadeh^{c*}

^aDepartment of Organic Chemistry, Faculty of Chemistry, University of Kashan, Kashan, 87317, I.R. Iran.

^bReproductive Immunology Research Center, Avicenna Research Institute, ACECR, Tehran, Iran.

^cNanobiotechnology Research Center, Avicenna Research Institute, ACECR, Tehran, Iran. E-mail: r.ghahremanzadeh@avicenna.ac.ir

SUPPORTING INFORMATION

<i>List of contents</i>	<i>Page</i>	<i>List of contents</i>	<i>Page</i>
Title, author's name, address	1	¹ H NMR of 5h	22
General methods and characterization data	2-8	¹³ C NMR of 5h	23
¹ H NMR of 5a	9	¹ H NMR of 5i	24
¹³ C NMR of 5a	10	¹³ C NMR of 5i	25
¹ H NMR of 5b	11	¹ H NMR of 5j	26
¹³ C NMR of 5b	12	¹³ C NMR of 5j	27
¹ H NMR of 5c	13	¹ H NMR of 5k	28
¹³ C NMR of 5c	14	¹³ C NMR of 5k	29
¹ H NMR of 5d	15	¹ H NMR of 5l	30
¹³ C NMR of 5d	16	¹³ C NMR of 5l	31
¹ H NMR of 5e	17		
¹³ C NMR of 5e	18		
¹ H NMR of 5f	19		
¹³ C NMR of 5f	20		
¹ H NMR of 5g	21		

Experimental Part

The chemicals used in this work were obtained from Fluka and Merck and were used without purification. Melting points were measured on an Electrothermal 9200 apparatus. Mass spectra were recorded on a Shimadzu QP 1100 Ex mass spectrometer operating at an ionization potential of 70 eV. IR spectra were recorded as KBr pellets on a Perkin-Elmer 781 spectrophotometer and an Impact 400 Nicolet FT-IR spectrophotometer. ^1H NMR and ^{13}C NMR spectra were recorded in $\text{DMSO}-d_6$ solvents on a Bruker DRX-400 spectrometer with tetramethylsilane as internal reference. The elemental analyses (C, H, N) were obtained from a Carlo ERBA Model EA 1108 analyzer. X-ray diffraction (XRD) pattern of the as-synthesized material was obtained using a Holland Philips Xpert X-ray powder diffraction (XRD) diffractometer (CuK α , radiation, $\lambda = 0.154056$ nm), at a scanning speed of $2^\circ/\text{min}$ from 10° to 100° (2θ). The nanocatalyst was determined using a FEI Quanta 200 scanning electron microscope (SEM) operated at a 20 kV accelerating voltage. The purity determination of the substrates and reaction monitoring were accomplished by TLC on silica-gel polygram SILG/UV 254 plates (from Merck Company).

Typical experimental procedure for the preparation of catalyst

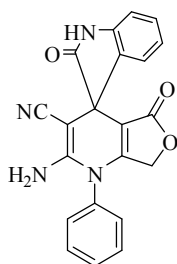
MnFe_2O_4 nanoparticles has been prepared following the reported standard protocol by co-precipitation of MnCl_2 and FeCl_3 in water in the presence of sodium hydroxide. Briefly, $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ were taken in molar ratio of $\text{Mn}^{2+}:\text{Fe}^{3+} = 1:2$ to prepare $0.3 \text{ mol} \cdot \text{L}^{-1}$ metal ion solution of 100 ml containing $0.1 \text{ mol} \cdot \text{L}^{-1}$ Mn^{2+} and $0.2 \text{ mol} \cdot \text{L}^{-1}$ Fe^{3+} , then was slowly dropped into 100 ml NaOH solution of $3 \text{ mol} \cdot \text{L}^{-1}$ at the preheated temperature of 95°C . After aging for 2 h with continuous stirring, the mixture was filtered, washed and dried at 60°C for 12 h.

Typical procedure for the preparation of spiro-furo-pyridine-indoline-carbonitriles **5a-l**:

A mixture of isatin **1** (1 mmol), malononitrile **2** (1 mmol), anilinolactones **3** (1 mmol), and MnFe_2O_4 (5 mol%) were taken in PEG-400 (1 mL). The resulting mixture was stirred at 90°C for an appropriate time. After completion of the reaction as indicated by TLC, the reaction mixture was magnetically concentrated with the aid of an external magnet to separate the catalyst. After separation of the catalyst, H_2O (10 ml) was added to the reaction mixture and was shaken for a few minutes to

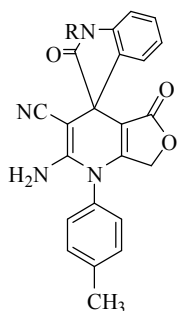
dissolve PEG and precipitate the product. The crude product (insoluble in water) was filtered and re-crystallized by ethanol for more purification. The desired pure product was identified by physical and spectroscopic data.

2-Amino-2',5-dioxo-1-phenyl-5,7-dihydro-1H-spiro[furo[3,4-b]pyridine-4,3'-indoline]-3-carbonitrile (5a):



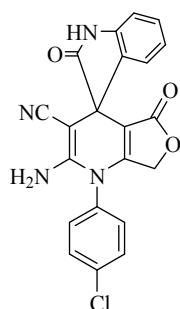
White powder (Yield: 79%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3385, 2180, 1750, 1683. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 4.34-4.65 (2H, m, OCH₂) 6.03 (2H, s, NH₂), 6.81-7.56 (9H, m, ArH), 10.49 (1H, s, NH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 48.2, 60.2, 66.1, 99.2, 109.9, 119.5, 122.5, 125.3, 129.3, 130.7, 134.3, 134.4, 141.8, 152.7, 159.5, 170.0, 178.0. Anal. Calcd for C₂₁H₁₄N₄O₃: C, 68.10; H, 3.81; N, 15.13%;. Found C, 68.16; H, 3.87; N, 15.08%; MS: m/z 370.

2-Amino-2',5-dioxo-1-p-tolyl-5,7-dihydro-1H-spiro[furo[3,4-b]pyridine-4,3'-indoline]-3 carbonitrile (5b):



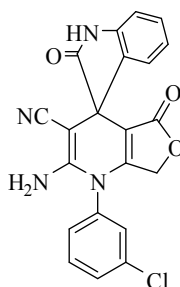
White powder (Yield: 83%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3454, 2185, 1721, 1682. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 2.37 (3H, s, CH₃), 4.47-4.64 (2H, m, OCH₂) 5.99 (2H, s, NH₂), 6.82-7.44 (8H, m, ArH), 10.48 (1H, s, NH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 21.2, 48.2, 60.0, 66.1, 99.0, 109.8, 119.5, 122.5, 125.3, 128.9, 129.3, 131.2, 131.7, 134.3, 140.3, 141.8, 152.8, 159.6, 170.1, 178.0. Anal. Calcd for C₂₂H₁₆N₄O₃: C, 68.74; H, 4.20; N, 14.58%;. Found C,68.69; H, 4.24; N, 14.53%. MS: m/z 384.

2-Amino-1-(4-chlorophenyl)-2',5-dioxo-5,7-dihydro-1H-spiro[furo[3,4-b]pyridine-4,3'-indoline]-3-carbonitrile (5c)



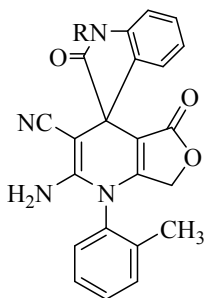
Cream powder (Yield: 80%). mp>300°C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3334, 2185, 1722, 1683. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 4.52-4.71 (2H, m, OCH₂) 6.21 (2H, s, NH₂), 6.81-7.63 (8H, m, ArH), 10.50 (1H, s, NH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 48.2, 60.5, 66.8, 99.3, 109.8, 115.6, 122.5, 125.4, 129.3, 130.7, 131.3, 133.4, 134.3, 135.3, 141.8, 152.7, 159.4, 170.0, 178.0. Anal. Calcd for C₂₁H₁₃ClN₄O₃: C, 62.31; H, 3.24; N%, 13.84;. Found C,62.36; H, 3.30; N, 13.79%. MS: m/z 406, 404.

2-Amino-1-(3-chlorophenyl)-2',5-dioxo-5,7-dihydro-1H-spiro[furo[3,4-b]pyridine-4,3'-indoline]-3-carbonitrile (5d):



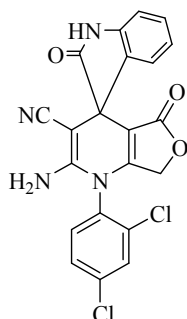
Gray powder (Yield: 77%). mp>300°C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 3454, 2186, 1720, 1686. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 4.51-4.73 (2H, m, OCH₂) 6.24 (2H, s, NH₂), 6.81-7.82 (8H, m, ArH), 10.51 (1H, s, NH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 48.2, 60.2, 66.1, 99.4, 109.8, 119.4, 122.5, 125.4, 128.2, 129.3, 129.7, 130.8, 132.1, 134.2, 134.6, 135.8, 141.8, 152.6, 159.2, 170.0, 177.9. Anal. Calcd for C₂₁H₁₃ClN₄O₃: C, 62.31; H, 3.24; N, 13.84%;. Found C,62.36; H, 3.19; N, 13.78%. MS: m/z 406, 404.

2-Amino-2',5-dioxo-1-p-tolyl-5,7-dihydro-1H-spiro[furo[3,4-b]pyridine-4,3'-indoline]-3-carbonitrile (5e):



White powder (Yield: 80%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3448, 2187, 1713, 1680. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 2.30 (3H, s, CH₃), 4.33-4.70 (2H, m, CH₂O) 6.06 (2H, s, NH₂), 6.83-7.46 (8H, m, ArH), 10.52 (1H, s, NH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 17.2, 48.3, 59.7, 66.0, 99.2, 110.0, 119.5, 122.6, 124.8, 128.3, 129.3, 129.8, 131.1, 132.3, 132.9, 134.4, 137.9, 141.7, 152.5, 159.2, 170.0, 177.9. Anal. Calcd for C₂₂H₁₆N₄O₃: C, 68.74; H, 4.20; N, 14.58%;. Found C,68.69; H, 4.14; N, 14.63%. MS: m/z 384.

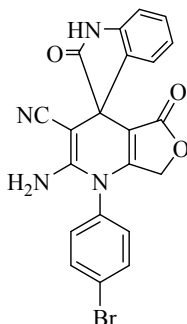
2-Amino-1-(2,4-dichlorophenyl)-2',5-dioxo-5,7-dihydro-1H-spiro[furo[3,4-b]pyridine-4,3'-indoline]-3-carbonitrile (5f):



Cream powder (Yield: 79%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3453, 2186, 1753, 1687. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 4.41-4.80 (2H, m, OCH₂) 6.43 (2H, s, NH₂), 6.83-7.98 (7H, m, ArH), 10.56 (1H, s, NH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 48.2, 59.7, 66.0, 100.1, 110.0, 119.3, 122.8, 124.9, 129.4, 129.8, 130.6, 131.1, 133.4, 134.2, 135.4, 136.7, 141.7, 152.3, 158.7, 169.8, 177.7. Anal. Calcd for C₂₁H₁₂Cl₂N₄O₃: C, 57.42; H, 2.75; N, 12.76%;. Found C,57.48; H, 2.69; N, 12.81%. MS: m/z 440, 438.

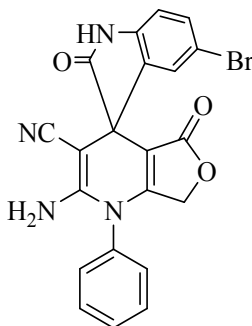
Due to very low solubility of the products **5g**, we cannot report the ^{13}C NMR data for this product.

2-Amino-1-(4-bromophenyl)-2',5-dioxo-5,7-dihydro-1H-spiro[furo[3,4-b]pyridine-4,3'-indoline]-3-carbonitrile (5g):



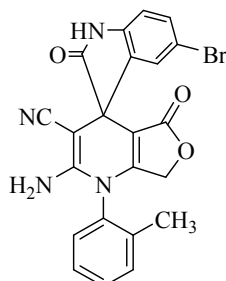
Cream powder (Yield: 75%). mp>300°C. IR (KBr) (ν_{max} / cm^{-1}): 3448, 2192, 1742, 1694. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 4.51-4.72 (2H, m, OCH₂) 6.21 (2H, s, NH₂), 6.81-7.78 (7H, m, ArH), 10.47 (1H, s, NH). Anal. Calcd for C₂₁H₁₃BrN₄O₃: C, 56.14; H, 2.92; N, 12.47%. Found C,56.19; H, 2.87; N, 12.53. Ms: m/z 450, 448

2-Amino-5'-bromo-2',5-dioxo-1-phenyl-5,7-dihydro-1H-spiro[furo[3,4-b]pyridine-4,3'-indoline]-3-carbonitrile (5h):



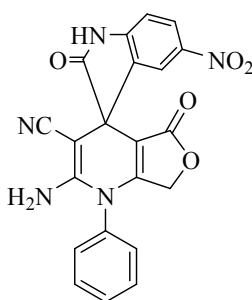
White powder (Yield: 73%). mp>300°C. IR (KBr) (ν_{max} / cm^{-1}): 3340, 2182, 1726, 1675. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 4.45-4.66 (2H, m, OCH₂) 6.12 (2H, s, NH₂), 6.80-7.65 (8H, m, ArH), 10.64 (1H, s, NH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 48.5, 59.4, 66.3, 98.3, 111.9, 114.3, 119.3, 128.2, 129.2, 129.5, 130.7, 132.1, 134.3, 136.5, 141.2, 152.9, 159.9, 170.1, 177.7. Anal. Calcd for C₂₁H₁₃BrN₄O₃: C, 56.14; H, 2.92; N, 12.47%. Found C,56.20; H, 2.87; N, 12.52%. Ms: m/z 450, 448.

2-Amino-5'-bromo-2',5-dioxo-1-o-tolyl-5,7-dihydro-1H-spiro[furo[3,4-b]pyridine-4,3'-indoline]-3-carbonitrile (5i):



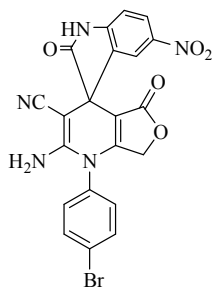
Cream powder (Yield: 76%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3348, 2178, 1718, 1680. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 2.36 (3H, s, CH₃), 4.43-4.84 (2H, m, OCH₂) 6.07 (2H, s, NH₂), 6.78-7.54 (7H, m, ArH), 10.70 (1H, s, NH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 21.3, 48.5, 59.3, 66.2, 98.2, 111.9, 114.3, 119.4, 128.2, 128.9, 129.2, 130.0, 131.2, 131.6, 132.1, 136.6, 140.4, 141.1, 153.0, 160.0, 170.1, 177.7. Anal. Calcd for C₂₂H₁₅BrN₄O₃: C, 57.04; H, 3.26; N, 12.09%. Found C, 57.10; H, 3.32; N, 12.14%. Ms: m/z 464, 462.

2-Amino-5'-nitro-2',5-dioxo-1-phenyl-5,7-dihydro-1H-spiro[furo[3,4-b]pyridine-4,3'-indoline]-3-carbonitrile (5j):



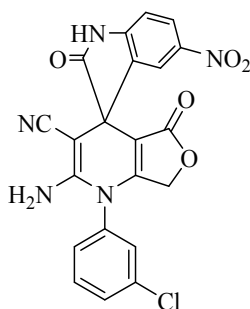
Cream powder (Yield: 81%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3352, 2188, 1735, 1674. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 4.50-4.68 (2H, m, OCH₂) 6.23 (2H, s, NH₂), 7.05-8.31 (8H, m, ArH), 11.25 (1H, s, NH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 48.5, 58.6, 66.5, 97.7, 110.2, 119.2, 121.2, 126.8, 129.2, 129.5, 130.7, 134.2, 135.0, 143.3, 148.4, 153.2, 160.4, 170.1, 178.7. Anal. Calcd for C₂₁H₁₃N₅O₅: C, 60.72; H, 3.15; N, 16.86%. Found C, 60.68; H, 3.20; N, 16.91%. Ms: m/z 415.

2-Amino-1-(4-bromophenyl)-5'-nitro-2',5-dioxo-5,7-dihydro-1H-spiro[furo[3,4-b]pyridine-4,3'-indoline]-3-carbonitrile (5k):



Cream powder (Yield: 84%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3362, 2187, 1735, 1692. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 4.57-4.69 (2H, m, OCH₂) 6.40 (2H, s, NH₂), 7.07-8.33 (7H, m, ArH), 11.24 (1H, s, NH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 48.5, 58.5, 66.5, 97.9, 110.2, 119.1, 121.3, 124.2, 126.8, 131.5, 133.6, 133.7, 135.0, 143.3, 148.4, 153.1, 160.1, 170.0, 178.6. Anal. Calcd for C₂₁H₁₂BrN₅O₅: C, 51.03; H, 2.45; N, 14.17%. Found C,51.09; H, 2.51; N, 14.12%. Ms: m/z 495, 493.

2-Amino-1-(3-chlorophenyl)-5'-nitro-2',5-dioxo-5,7-dihydro-1H-spiro[furo[3,4-b]pyridine-4,3'-indoline]-3-carbonitrile (5l):



Cream powder (Yield: 85%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3362, 2180, 1741, 1689. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 4.56-4.74 (2H, m, OCH₂) 6.42 (2H, s, NH₂), 7.07-8.37 (7H, m, ArH), 11.24 (1H, s, NH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 48.5, 58.5, 66.6, 97.9, 110.3, 119.3, 121.5, 126.9, 128.2, 129.6, 131.0, 132.1, 134.6, 135.0, 135.5, 143.3, 148.4, 153.1, 160.1, 170.1, 178.7. Anal. Calcd for C₂₁H₁₂ClN₅O₅: C, 56.07; H, 2.69; N, 15.57%. Found C,56.12; H, 2.64; N, 15.61%. Ms: m/z 451, 449.



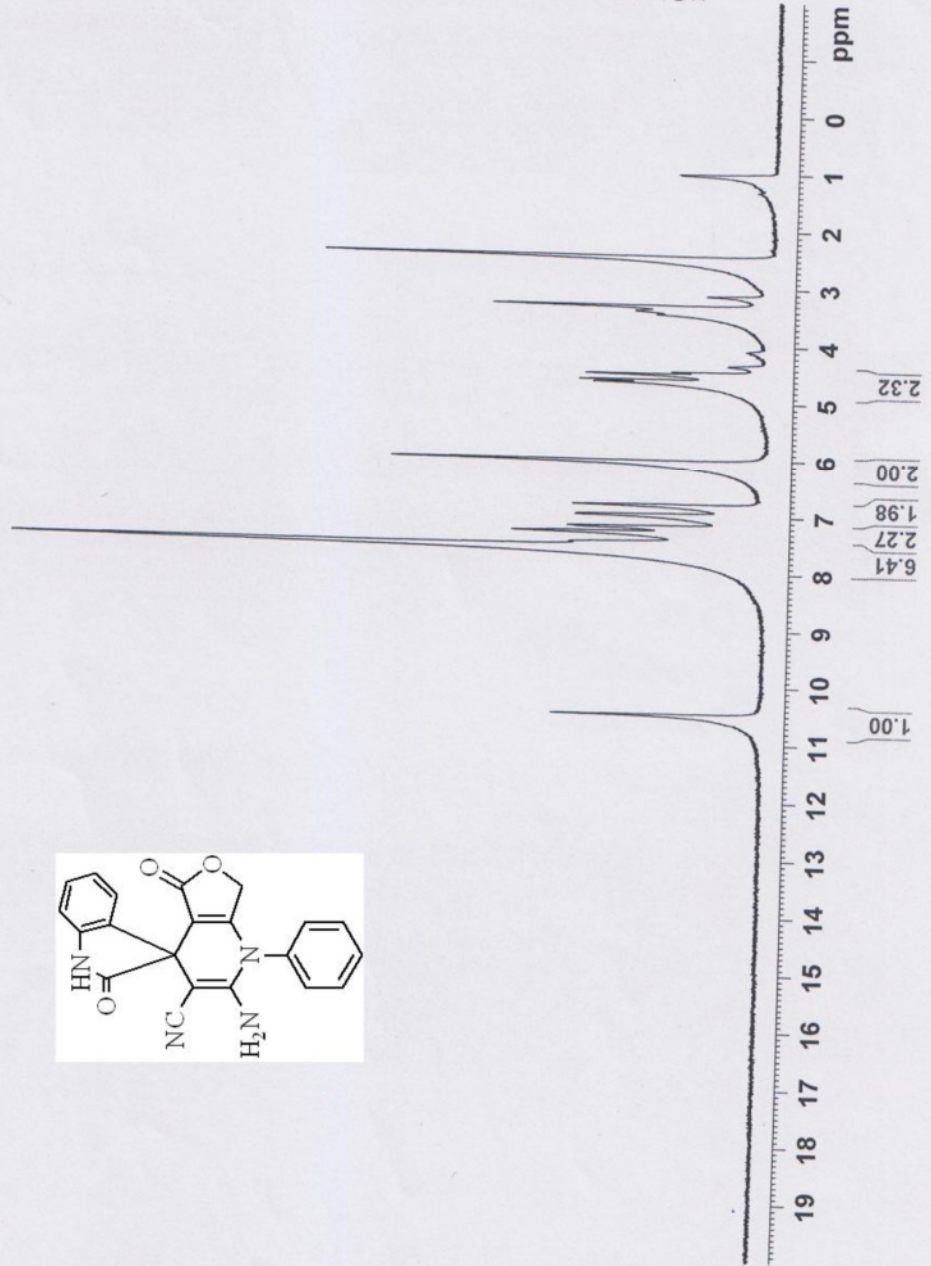
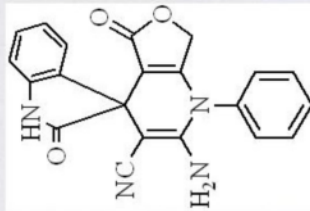
Current Data Parameters
NAME 3H
EXPNO 421
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130217
Time 9.29
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 1
DS 0
SWH 11574.074 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 128
DW 43.200 usec
DE 6.00 usec
TE 300.0 K
D1 5.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10.495
7.562
7.481
7.303
7.197
6.999
6.986
6.819
6.039
4.650
4.611
4.505
4.463
4.345
3.474
3.411
3.327
3.142
2.473
1.027





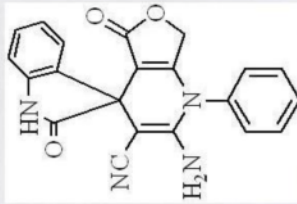
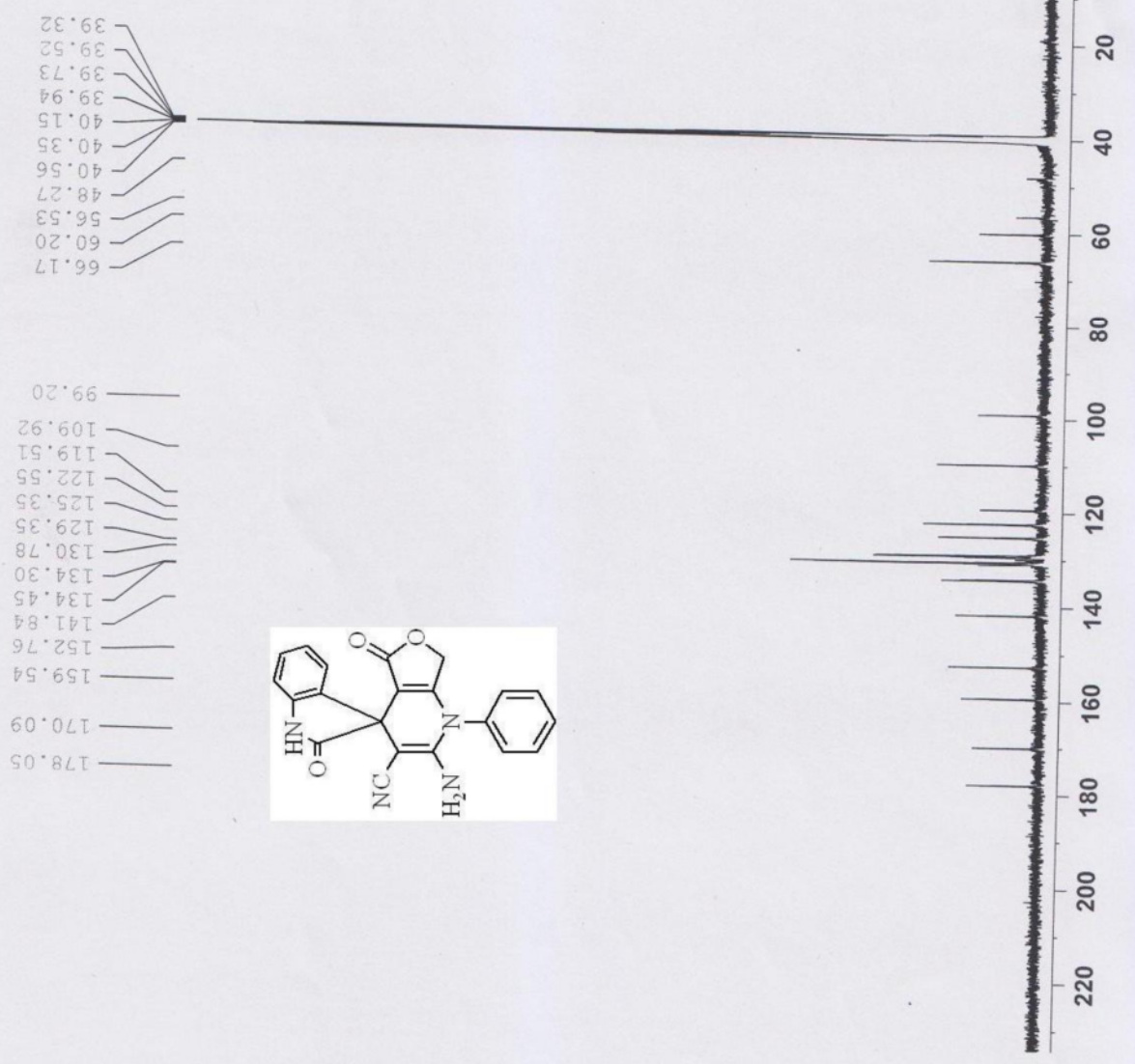
Current Data Parameters
NAME Carbon13
EXPNO 2085
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130217
Time_ 12.38
INSTRUM Spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1000
DS 4
SWH 40404.039 Hz
FIDRES 0.616517 Hz
AQ 0.8110580 sec
RG 32768
DW 12.375 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

==== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SF01 100.6204965 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.50 dB
PL13 18.50 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





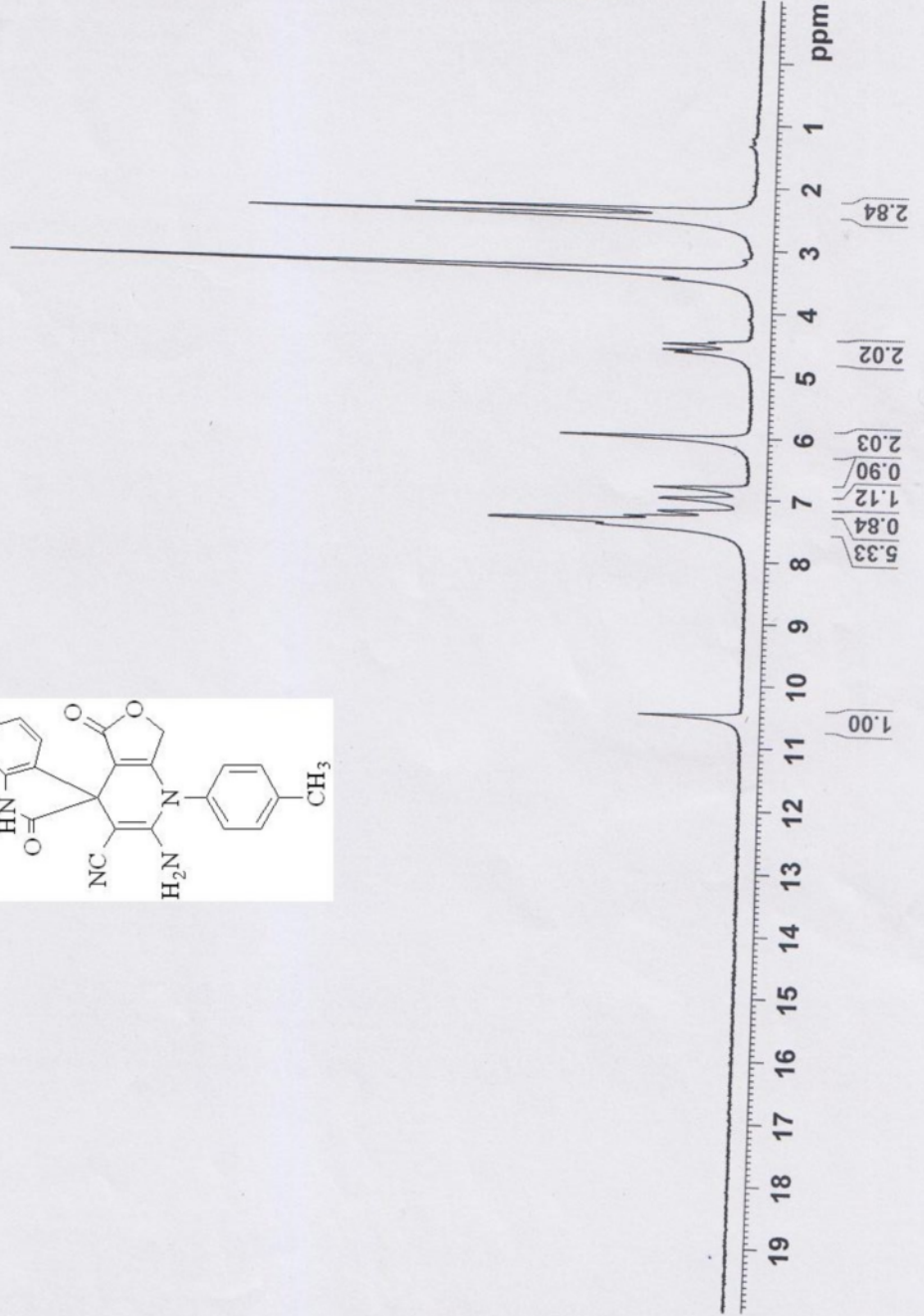
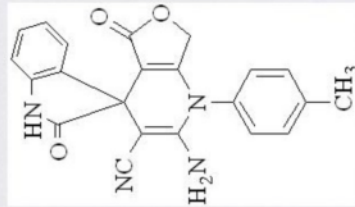
Current Data Parameters
NAME 3H
EXPNO 420
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130217
Time 9.20
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 1
DS 0
SWH 11574.074 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 256
DW 43.200 usec
DE 6.00 usec
TE 300.0 K
D1 5.00000000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10.488
7.445
7.372
7.296
7.197
6.998
6.820
5.997
4.644
4.602
4.510
4.470
3.481
3.330
2.479
2.371





Current Data Parameters
NAME Carbon13
EXPNO 2084
PROCNO 1

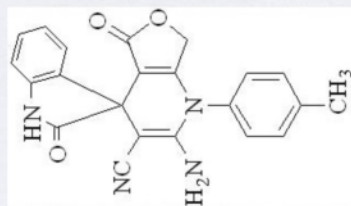
F2 - Acquisition Parameters
Date_ 20130217
Time_ 11.48
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1000
DS 4
SWH 40404.039 Hz
FIDRES 0.616517 Hz
AQ 0.8110580 sec
RG 32768
DW 12.375 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

==== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6204965 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.50 dB
PL13 18.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

178.06
170.10
159.67
152.83
141.81
140.36
134.34
131.77
131.27
129.32
128.95
125.32
122.52
119.51
109.89
99.06
66.13
60.05
48.24
40.56
40.36
40.15
39.94
39.73
39.53
39.32
21.28



ppm



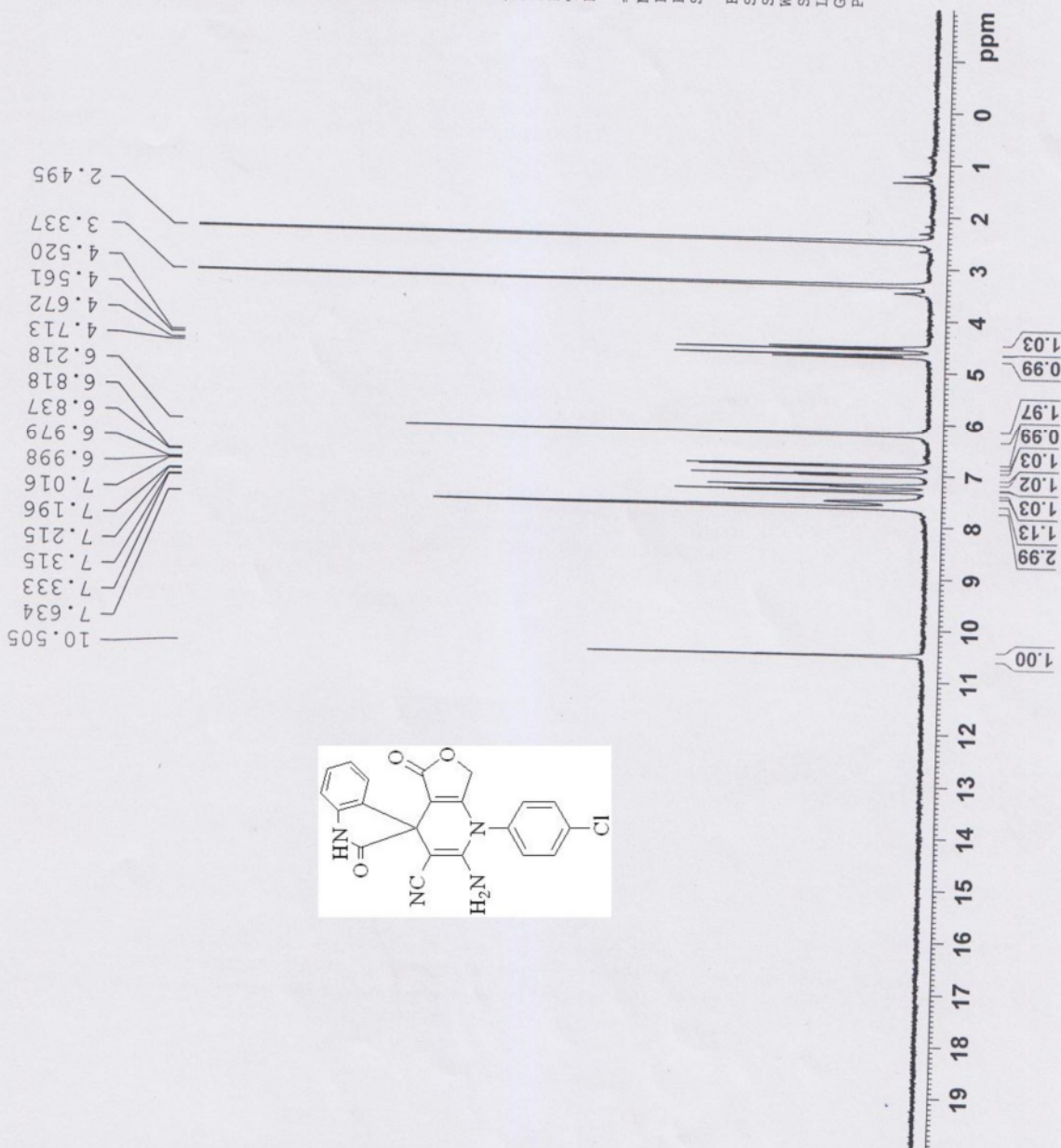
Current Data Parameters
NAME 3H
EXPNO 422
PROCNO 1

F2 - Acquisition Parameters

Date_ 20130217
Time 9.33
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 1
DS 0
SWH 11574.074 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 512
DE 43.200 usec
TE 6.00 usec
D1 300.0 K
D11 5.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





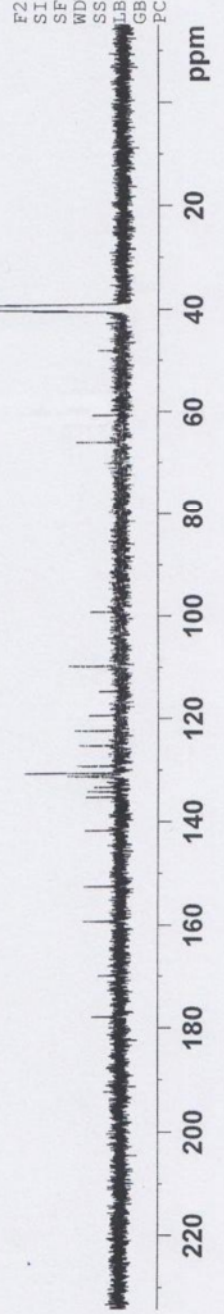
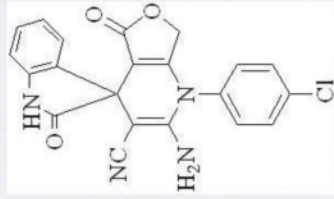
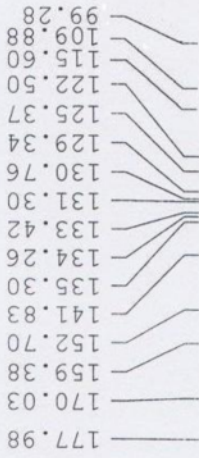
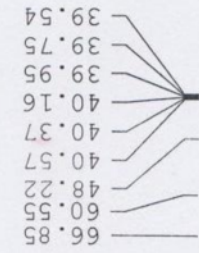
Current Data Parameters
NAME Carbon13
EXPNO 2086
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130218
Time 9.13
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 500
DS 4
SWH 40404.039 Hz
FIDRES 0.616517 Hz
AQ 0.8110580 sec
RG 32768
DW 12.375 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

==== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6204965 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.50 dB
PL13 18.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
EM 0
WDW 0
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





Current Data Parameters
NAME 3H
EXPNO 423
PROCNO 1

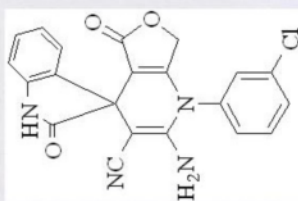
F2 - Acquisition Parameters

Date 20130217
Time 9.58
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 1
DS 0
SWH 11574.074 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 287.4
DW 43.200 usec
DE 6.00 usec
TE 300.0 K
D1 5.00000000 sec

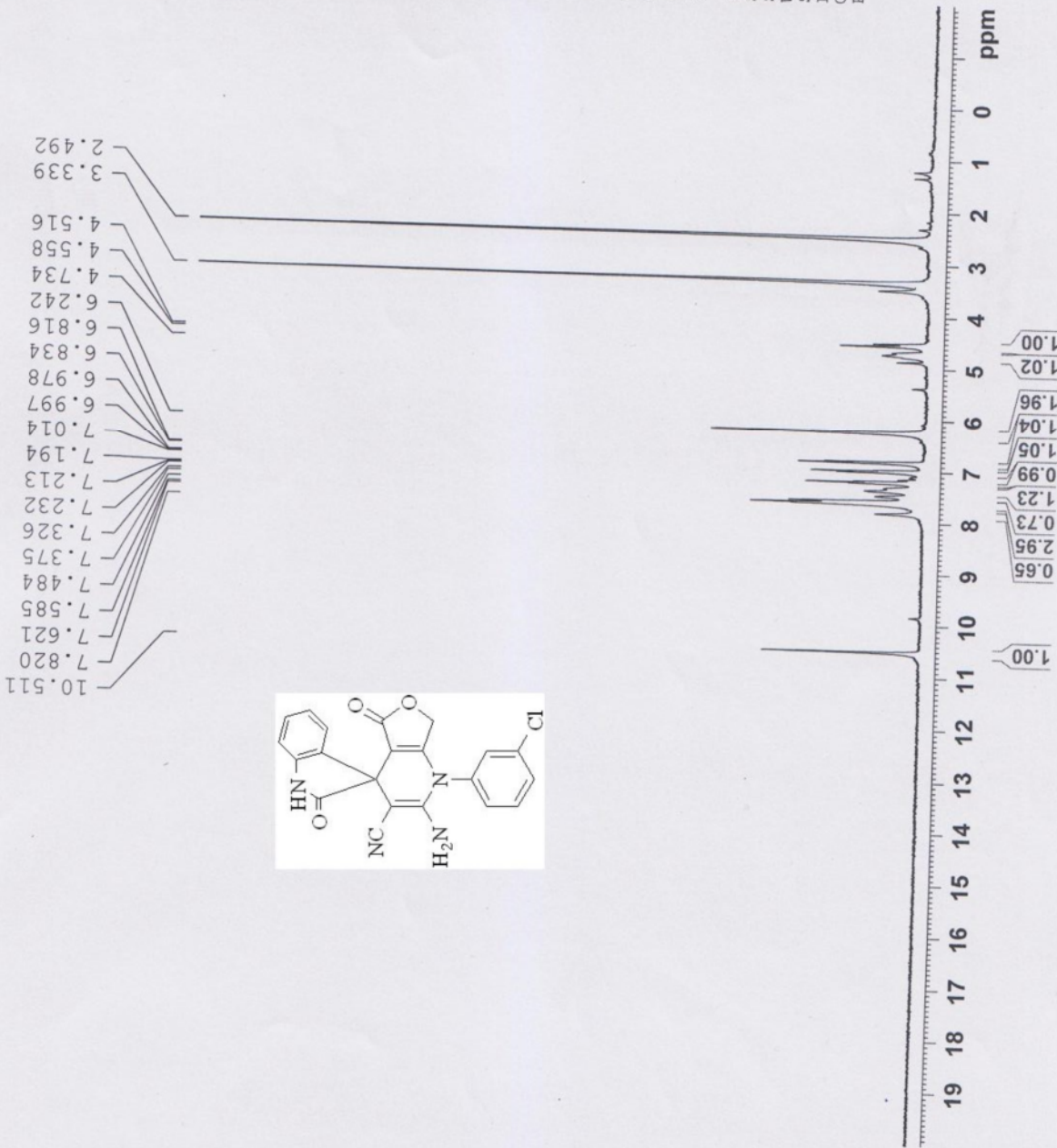
==== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10.511
7.820
7.621
7.585
7.484
7.375
7.326
7.232
7.213
7.194
7.114
6.997
6.834
6.816
6.242
4.734
4.558
4.516
3.339
2.492



1.00
1.02
1.96
1.04
1.05
0.99
1.23
0.73
2.95
0.65





Current Data Parameters
 NAME Carbon13
 EXPNO 2083
 PROCNO 1

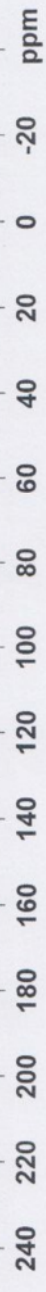
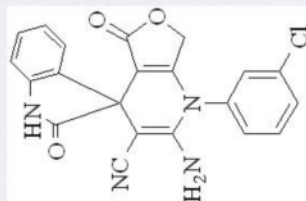
F2 - Acquisition Parameters
 Date_ 20130217
 Time_ 10.51
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 1000
 DS 4
 SWH 40404.039 Hz
 FIDRES 0.616517 Hz
 AQ 0.8110580 sec
 RG 32768
 DW 12.375 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

==== CHANNEL f1 =====
 NUC1 13C
 P1 7.50 usec
 PL1 -2.00 dB
 SFO1 100.6204965 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PL2 -4.00 dB
 PL12 18.50 dB
 PL13 18.50 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

177.95
170.02
159.24
152.63
141.82
135.80
134.63
134.25
132.11
130.82
129.76
129.35
128.26
125.49
122.51
119.45
109.88
99.45
66.19
60.28
48.22
40.58
40.37
40.17
39.96
39.75
39.54
39.33





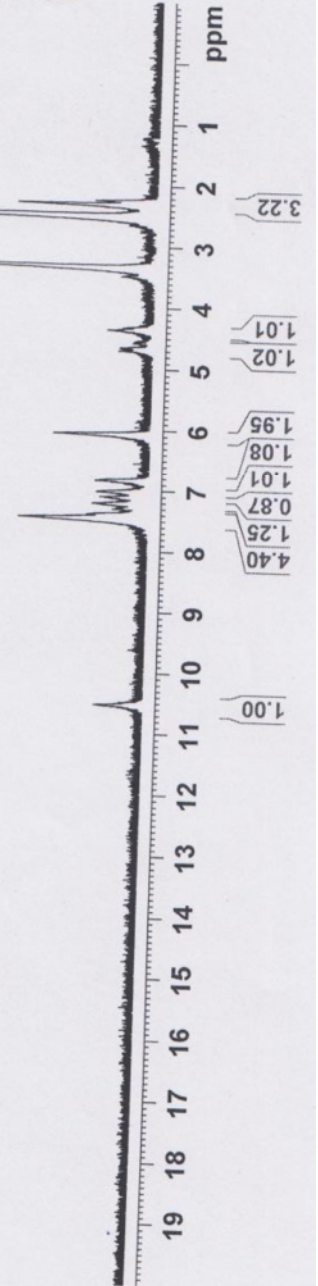
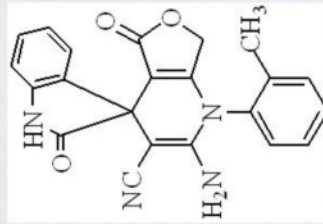
Current Data Parameters
NAME 3H
EXPNO 488
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130227
Time 8.34
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 1
DS 0
SWH 11574.074 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1824.6
DW 43.200 usec
DE 6.00 usec
TE 300.0 K
D1 5.00000000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10.524
7.462
7.307
7.214
7.172
7.106
7.022
6.883
6.836
6.069
4.704
4.674
4.662
4.370
4.334
3.488
3.461
3.328
2.653
2.487
2.396
2.306
2.246





Current Data Parameters
NAME Carbon13
EXPNO 2096
PROCNO 1

F2 - Acquisition Parameters

Date_ 20130227
Time 10.15
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1000
DS 4
SWH 40404.039 Hz
FIDRES 0.616517 Hz
AQ 0.8110580 sec
RG 32768
DW 12.375 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

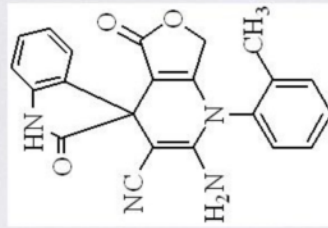
=====
CHANNEL f1
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6204965 MHz

=====
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.50 dB
PL13 18.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

177.92
170.09
159.25
152.53
141.79
137.98
134.44
132.94
132.30
131.15
129.84
129.37
128.38
124.84
122.66
119.50
110.04
99.28

66.02
59.71
48.33
40.58
40.37
40.16
39.96
39.75
39.54
39.33
17.27



220 200 180 160 140 120 100 80 60 40 20 0 ppm



Current Data Parameters
NAME 3H
EXPNO 489
PROCNO 1

F2 - Acquisition Parameters

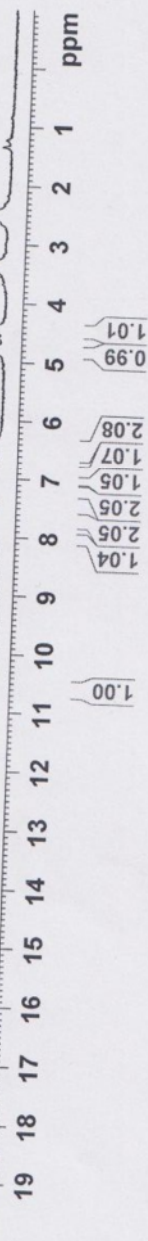
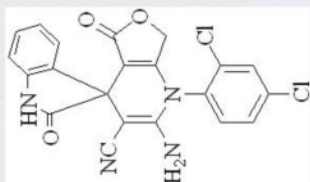
Date_ 20130227
Time_ 12.15
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 1
DS 0
SWH 11574.074 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 203.2
DW 43.200 usec
DE 6.00 usec
TE 300.0 K
D1 5.00000000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10.561
7.980
7.733
7.648
7.217
7.145
7.005
6.838
6.430
4.801
4.761
4.458
4.417
3.352
2.492





Current Data Parameters
NAME Carbon13
EXPNO 2097
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130227
Time 13.03
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1000
DS 4
SWH 40404.039 Hz
FIDRES 0.616517 Hz
AQ 0.8110580 sec
RG 32768
DW 12.375 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

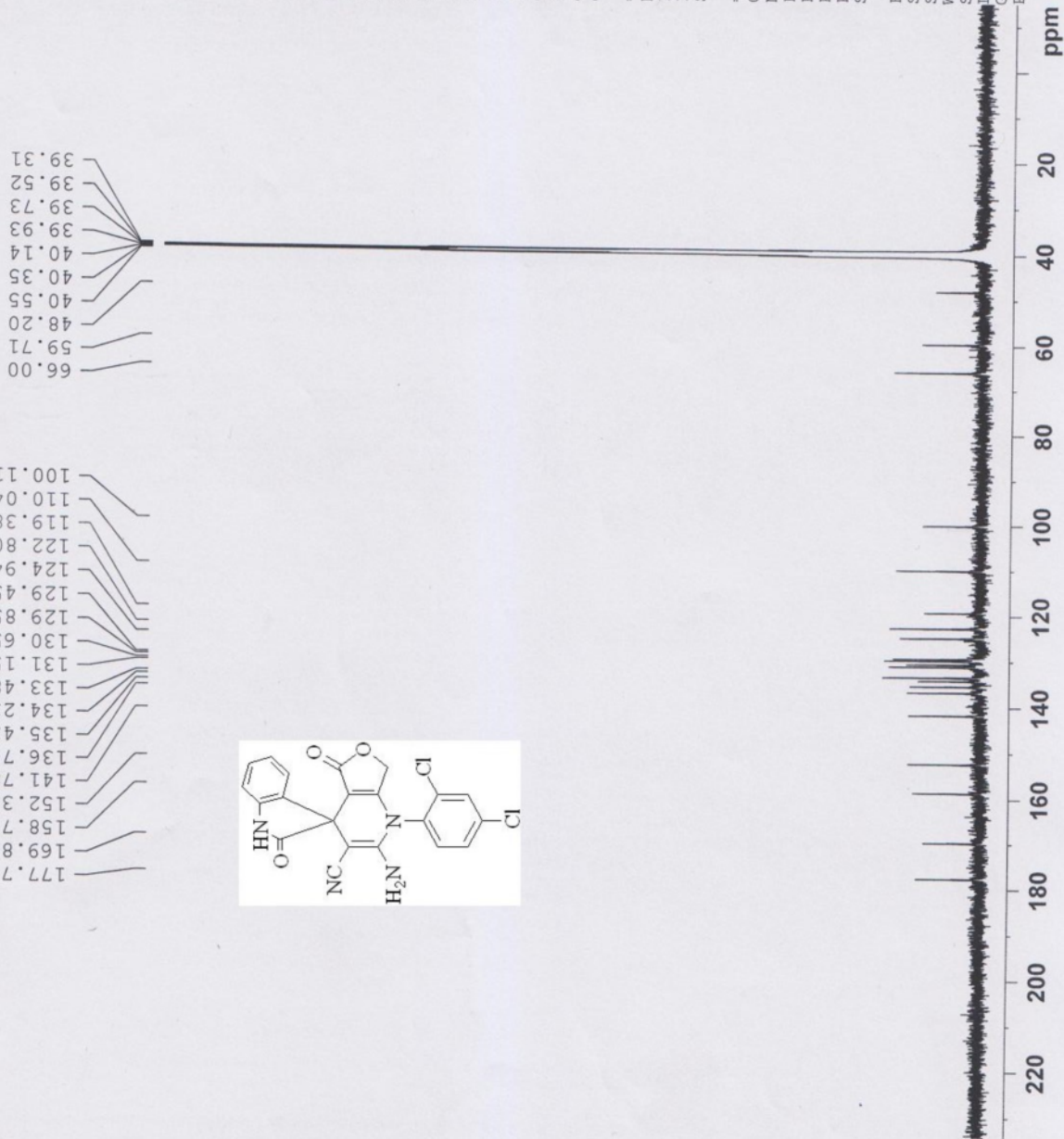
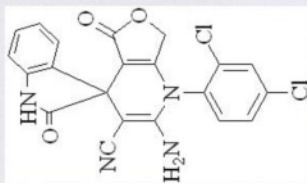
==== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6204965 MHz

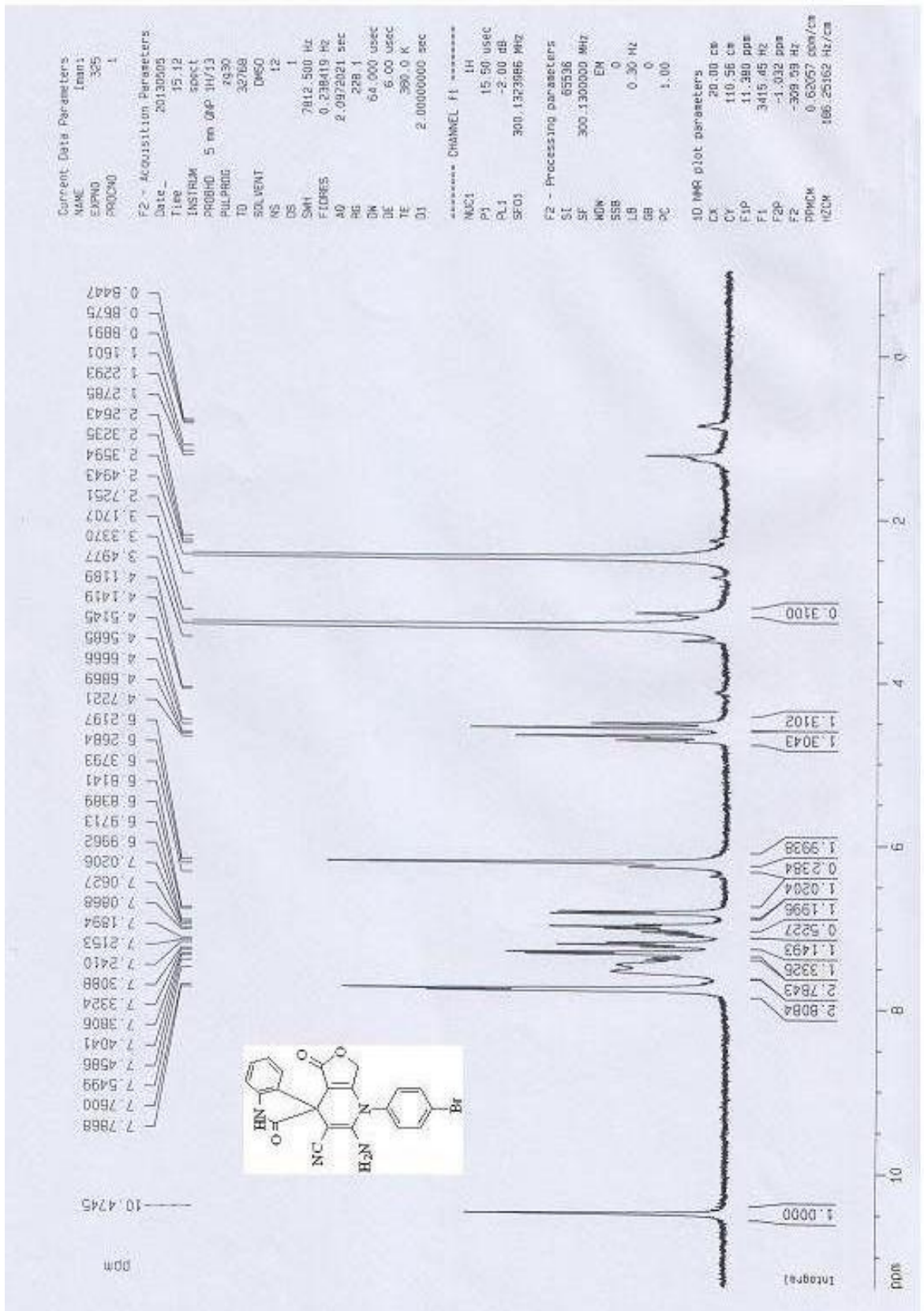
==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.50 dB
PL13 18.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

66.00
59.71
48.20
40.55
40.35
40.14
39.93
39.73
39.52
39.31

177.74
169.82
158.74
152.39
141.78
136.76
135.43
134.23
133.48
131.15
130.65
129.85
129.45
124.94
122.80
119.38
110.04
100.11





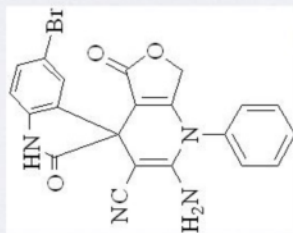
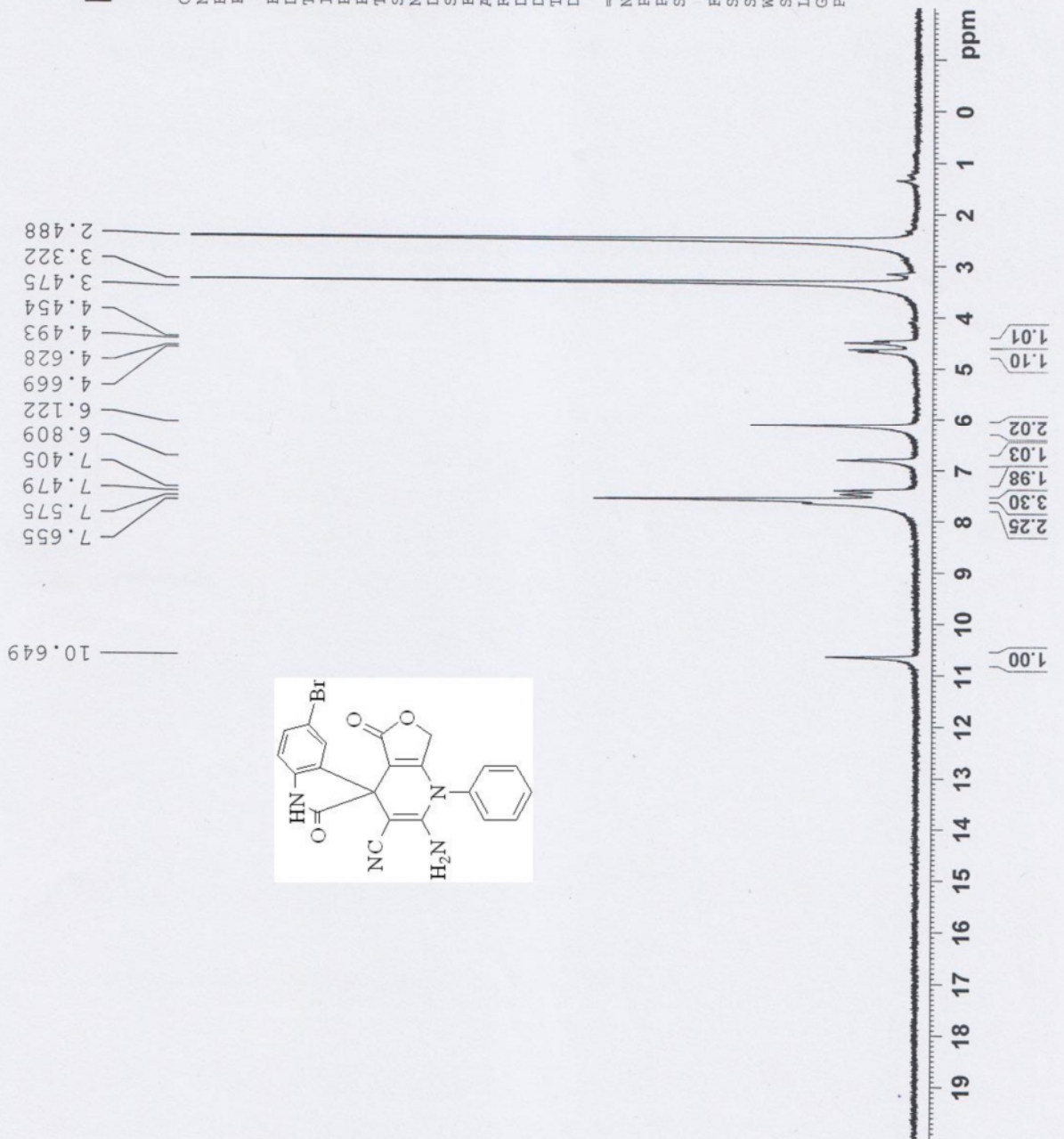


Current Data Parameters
 NAME 3H
 EXPNO 582
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130312
 Time 13.44
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg
 TD 32768
 SOLVENT DMSO
 NS 16
 DS 0
 SWH 11574.074 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 1625.5
 DW 43.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 5.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 -6.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300045 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
NAME Carbon13
EXPNO 2120
PROCNO 1

F2 - Acquisition Parameters

Date_ 20130312
Time 14.40
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1000
DS 4
SWH 40404.039 Hz
FIDRES 0.616517 Hz
AQ 0.8110580 sec
RG 32768
DW 12.375 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

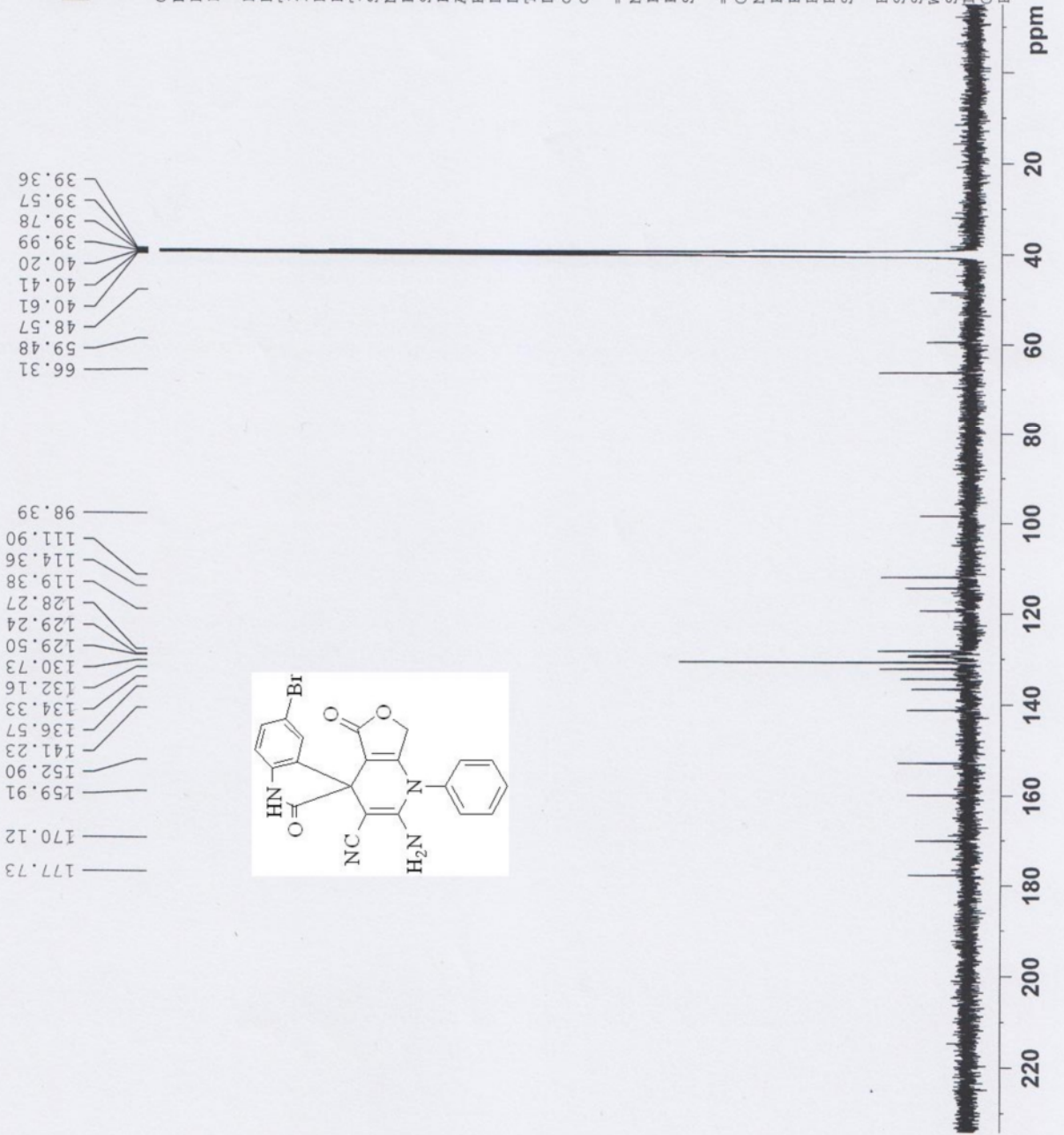
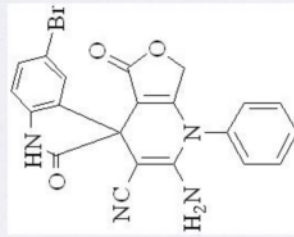
==== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6204965 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.50 dB
PL13 18.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
FC 1.40

66.31
59.48
48.57
40.61
40.41
40.20
39.99
39.78
39.57
39.36

177.73
170.12
159.91
152.90
141.23
136.57
134.33
132.16
130.73
129.50
129.24
128.27
119.38
114.36
111.90
98.39





Current Data Parameters
NAME 3H
EXPNO 583
PROCNO 1

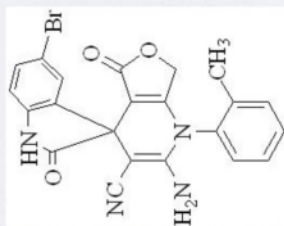
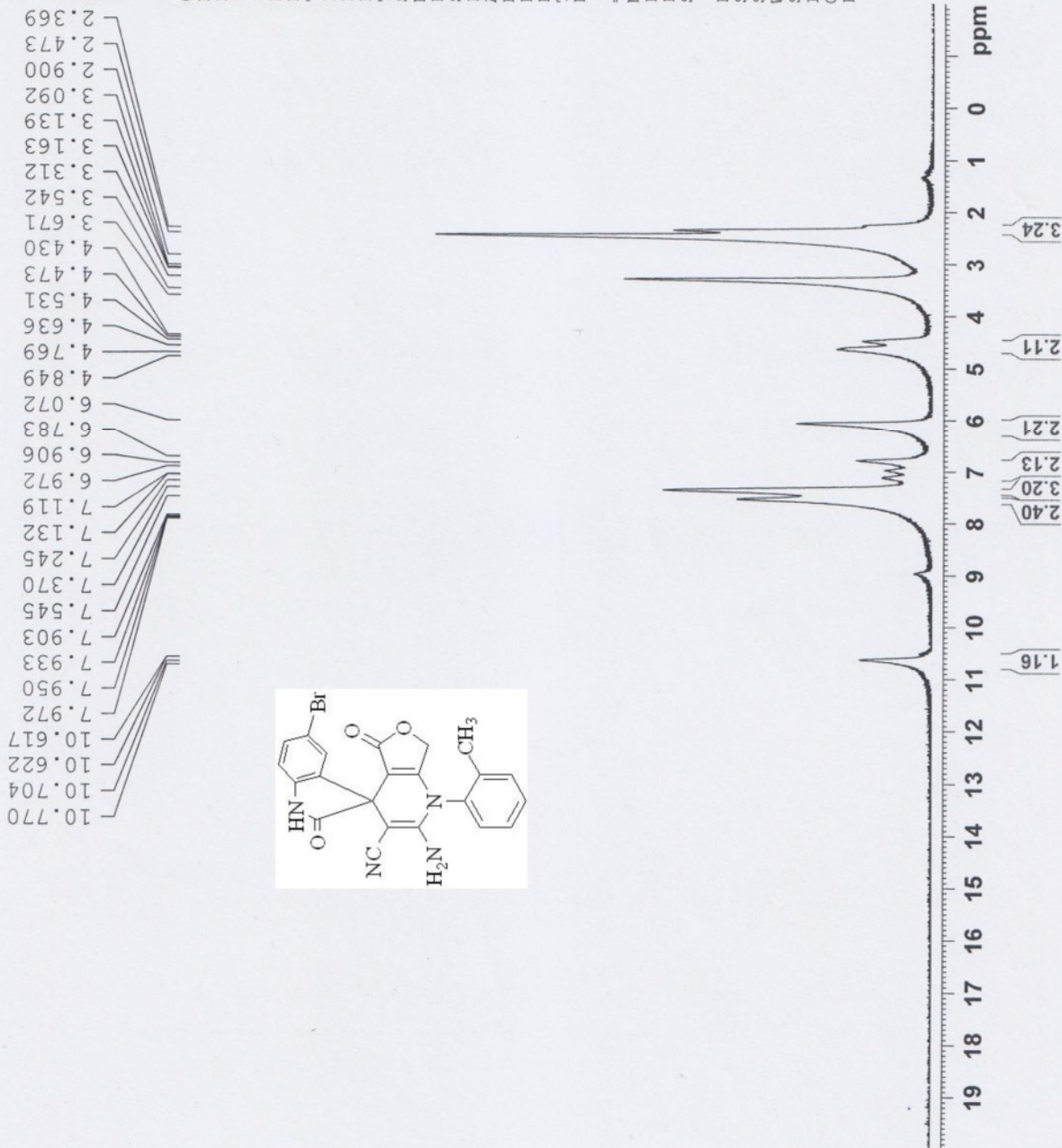
F2 - Acquisition Parameters

Date_ 20130312
Time_ 13.51
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 11574.074 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1149.4
DW 43.200 usec
DE 6.00 usec
TE 300.0 K
D1 5.00000000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





Current Data Parameters
NAME Carbon13
EXPNO 2121
PROCNO 1

F2 - Acquisition Parameters

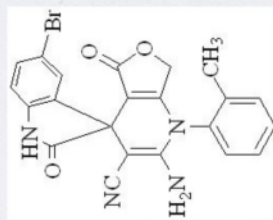
Date 20130312
Time 15.30
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1000
DS 4
SWH 40404.039 Hz
FIDRES 0.616517 Hz
AQ 0.8110580 sec
RG 32768
DW 12.375 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

==== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6204965 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.50 dB
PL13 18.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

21.30
39.58
39.79
39.99
40.19
40.40
40.40
48.54
59.34
66.29
98.29
111.90
114.32
119.42
128.27
128.94
128.94
129.26
130.08
131.23
131.68
131.68
132.15
136.60
140.41
141.18
141.18
153.01
160.08
170.11
177.76



ppm

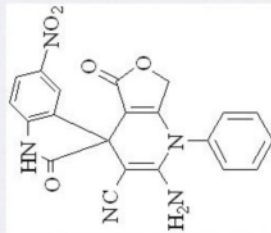
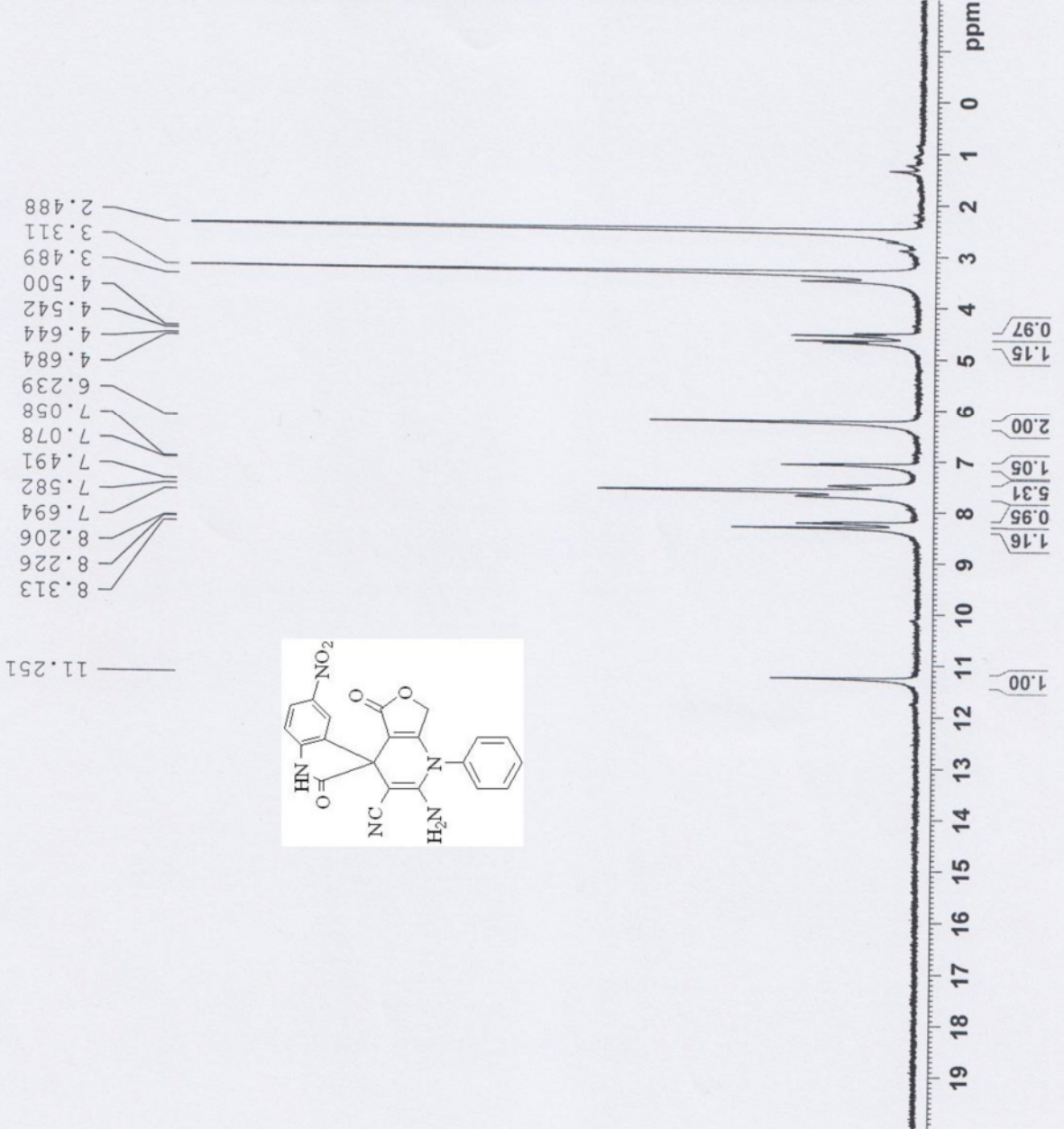


Current Data Parameters
NAME 3H
EXPNO 601
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130316
Time_ 14.12
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 11574.074 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1290.2
DW 43.200 usec
DE 6.00 usec
TE 300.0 K
D1 5.00000000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





Current Data Parameters
NAME Carbon13
EXPNO 2132
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130317
Time_ 9.26
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1000
DS 4
SWH 40404.039 Hz
FIDRES 0.616517 Hz
AQ 0.8110580 sec
RG 32768
DM 12.375 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

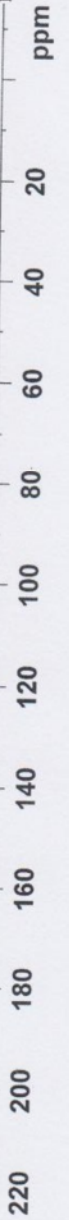
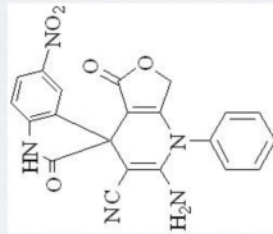
==== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SF01 100.6204965 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.50 dB
PL13 18.50 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

178.71
170.13
160.40
153.24
148.43
143.31
135.04
134.23
130.75
129.53
129.22
126.86
121.28
119.22
110.26
97.77

66.52
58.63
48.58
40.64
40.43
40.22
40.02
39.81
39.60
39.39





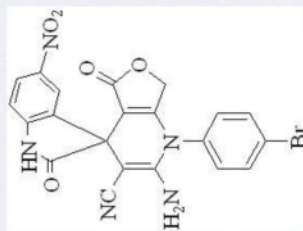
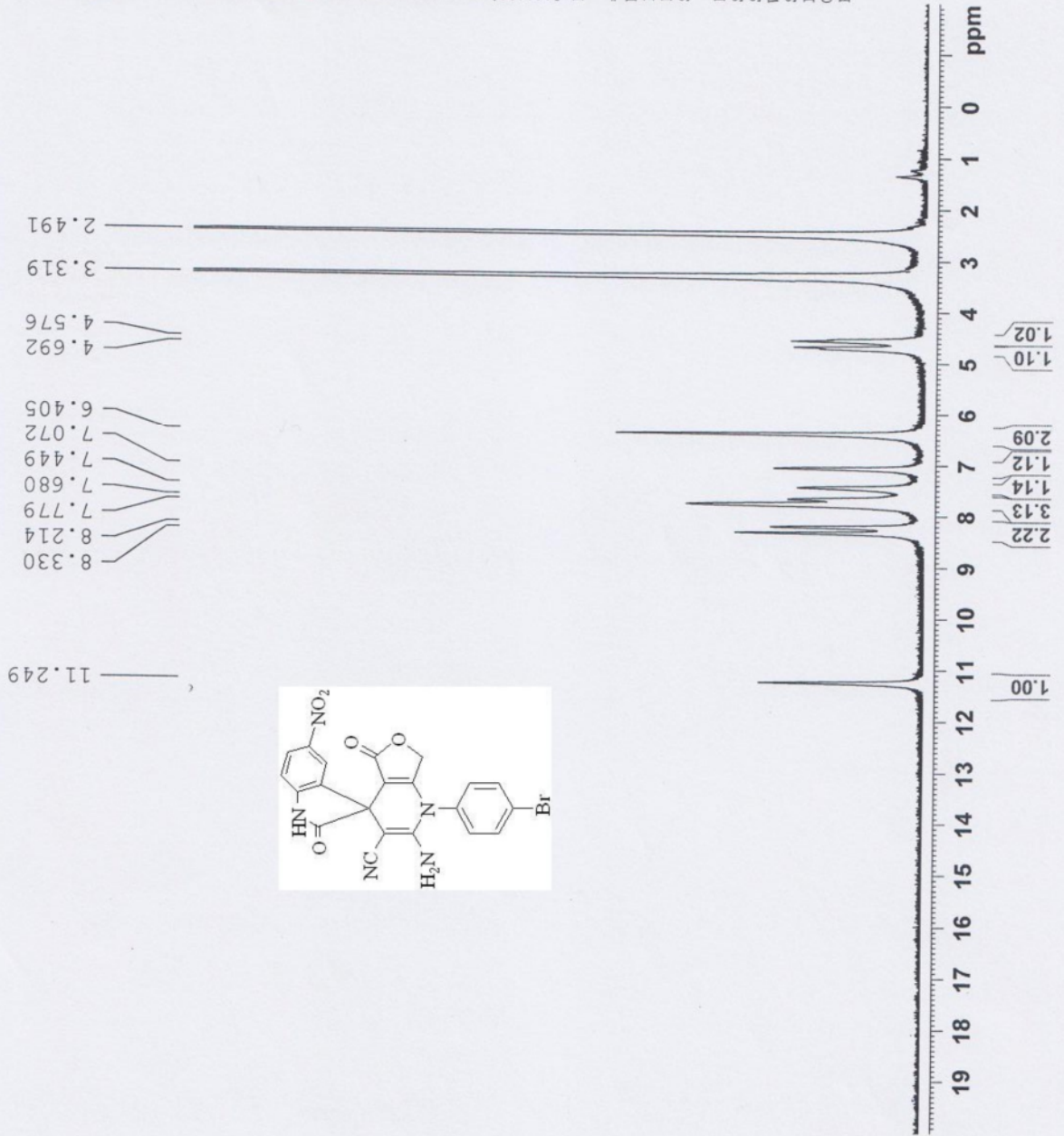
Current Data Parameters
NAME 3H
EXPNO 603
PROCNO 1

F2 - Acquisition Parameters

Date 20130316
Time 14.25
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 11574.074 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1149.4
DW 43.200 usec
DE 6.00 usec
TE 300.0 K
D1 5.00000000 sec

===== CHANNEL f1 =====
NUC1 LH
P1 9.00 usec
PL1 -6.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





Current Data Parameters
NAME Carbon13
EXPNO 2133
PROCNO 1

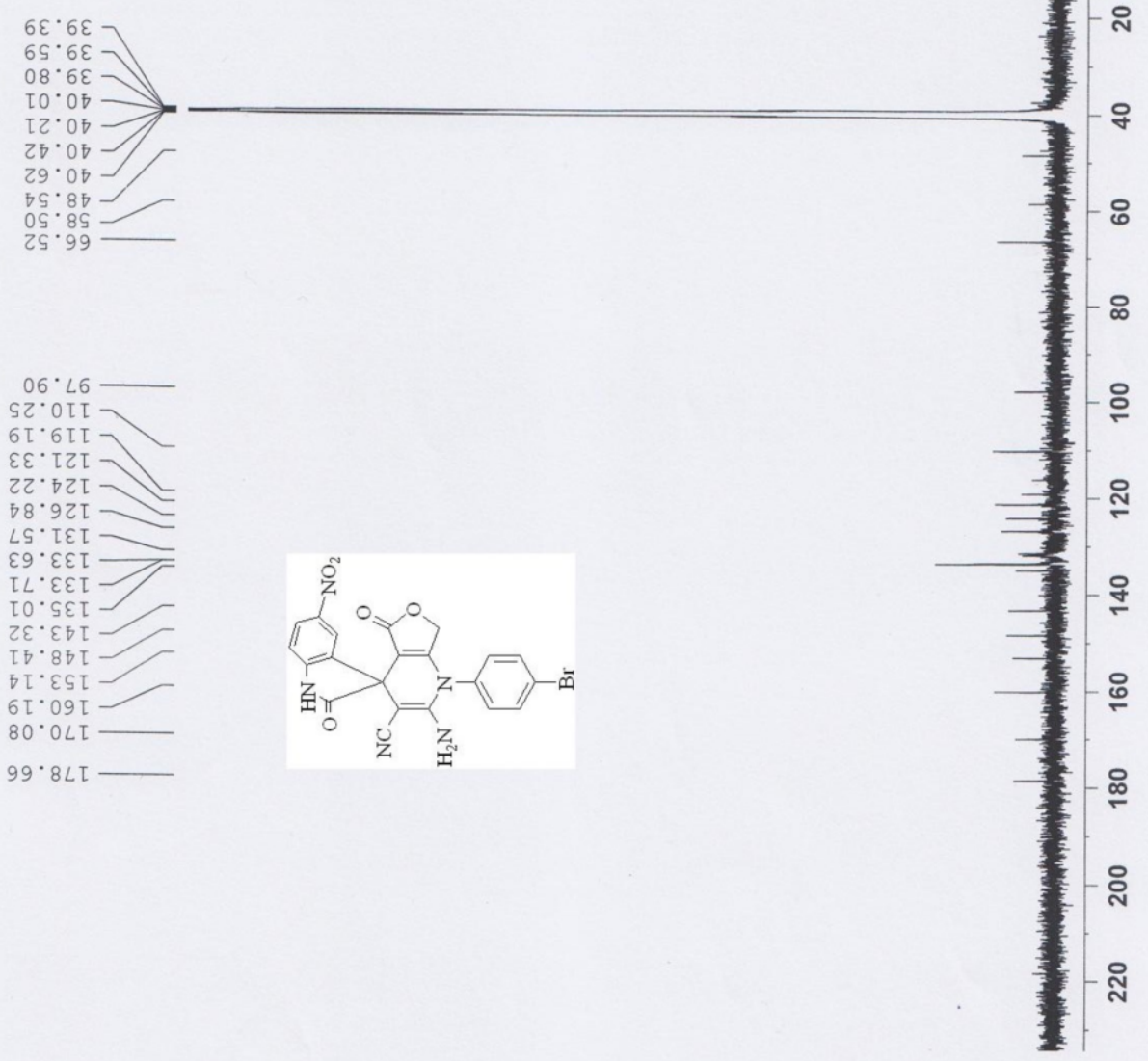
F2 - Acquisition Parameters

Date_ 20130317
Time_ 10.17
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1000
DS 4
SWH 40404.039 Hz
FIDRES 0.616517 Hz
AQ 0.8110580 sec
RG 32768
DW 12.375 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

==== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6204965 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.50 dB
PL13 18.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





Current Data Parameters
NAME 3H
EXPNO 604
PROCNO 1

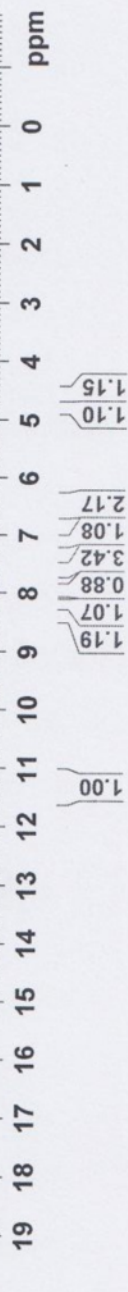
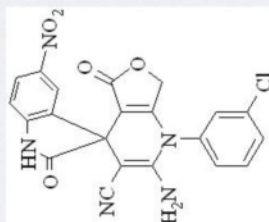
F2 - Acquisition Parameters

Date_ 20130316
Time_ 14.30
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 11574.074 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1149.4
DW 43.200 usec
DE 6.00 usec
TE 300.0 K
D1 5.00000000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300045 MHz
WDW EM
SSB 0
LB 0
GB 0
PC 1.00

11.247
8.375
8.213
7.926
7.628
7.488
7.071
6.420
4.740
4.566
3.313
2.880
2.716
2.657
2.489





Current Data Parameters
NAME Carbon13
EXPNO 2149
PROCNO 1

F2 - Acquisition Parameters

Date_ 20130424
Time_ 15.10
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2000
DS 4
SWH 40404.039 Hz
FIDRES 0.616517 Hz
AQ 0.8110580 sec
RG 32768
DW 12.375 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

==== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6204965 MHz

==== CHANNEL f2 =====
CFDPFG2 waitz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.50 dB
PL13 18.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
FC 1.40

178.70
170.14
160.15
153.13
148.37
143.33
135.50
135.02
134.59
132.14
130.96
130.01
129.63
128.59
128.25
126.90
121.47
119.28
110.26
97.86

