

Cu(OTf)₂ catalyzed three component strategy for the synthesis of thienopyridine containing spirooxindoles and their cytotoxic evaluation

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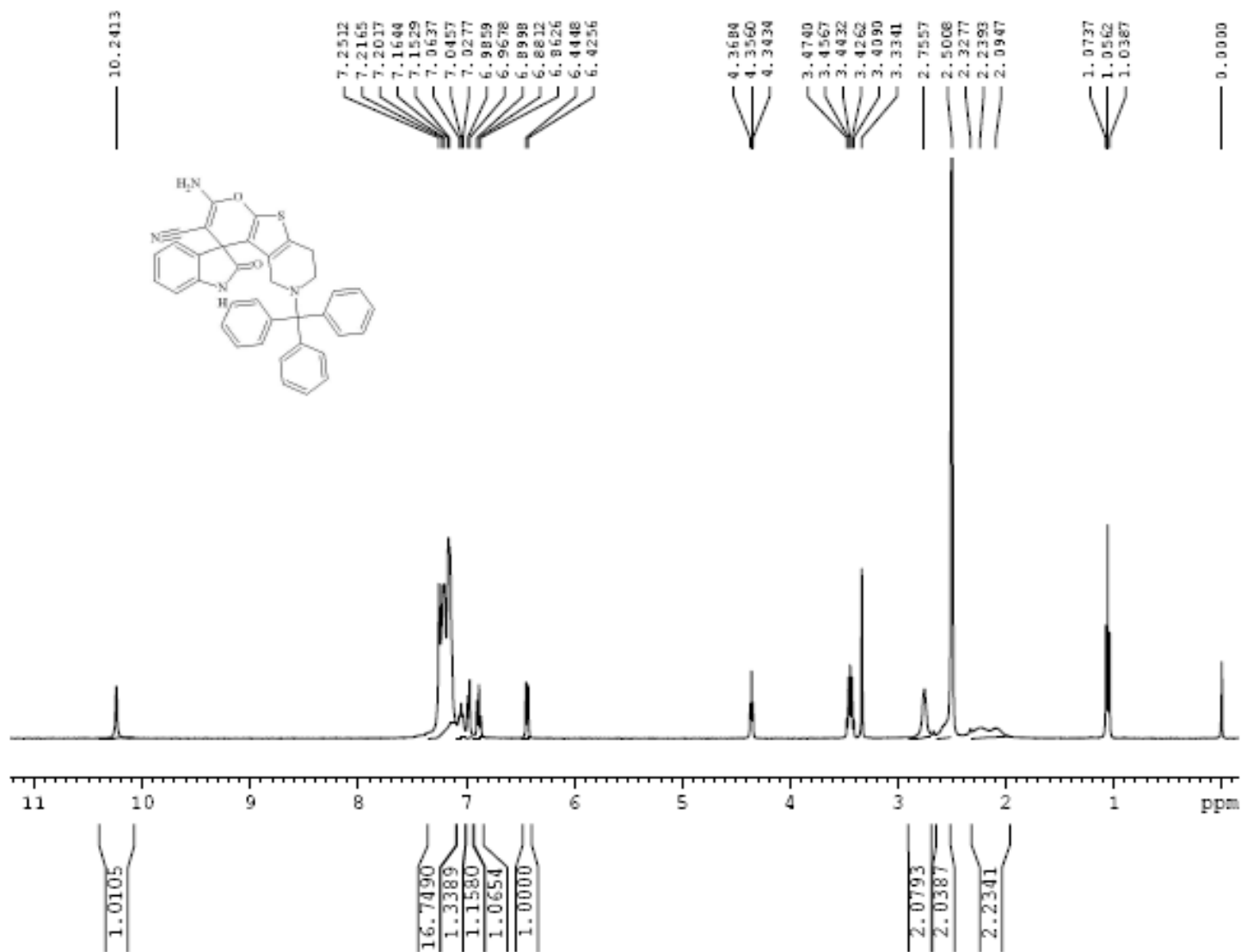
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IR, ¹H NMR, ¹³C NMR and mass spectra of selected compounds



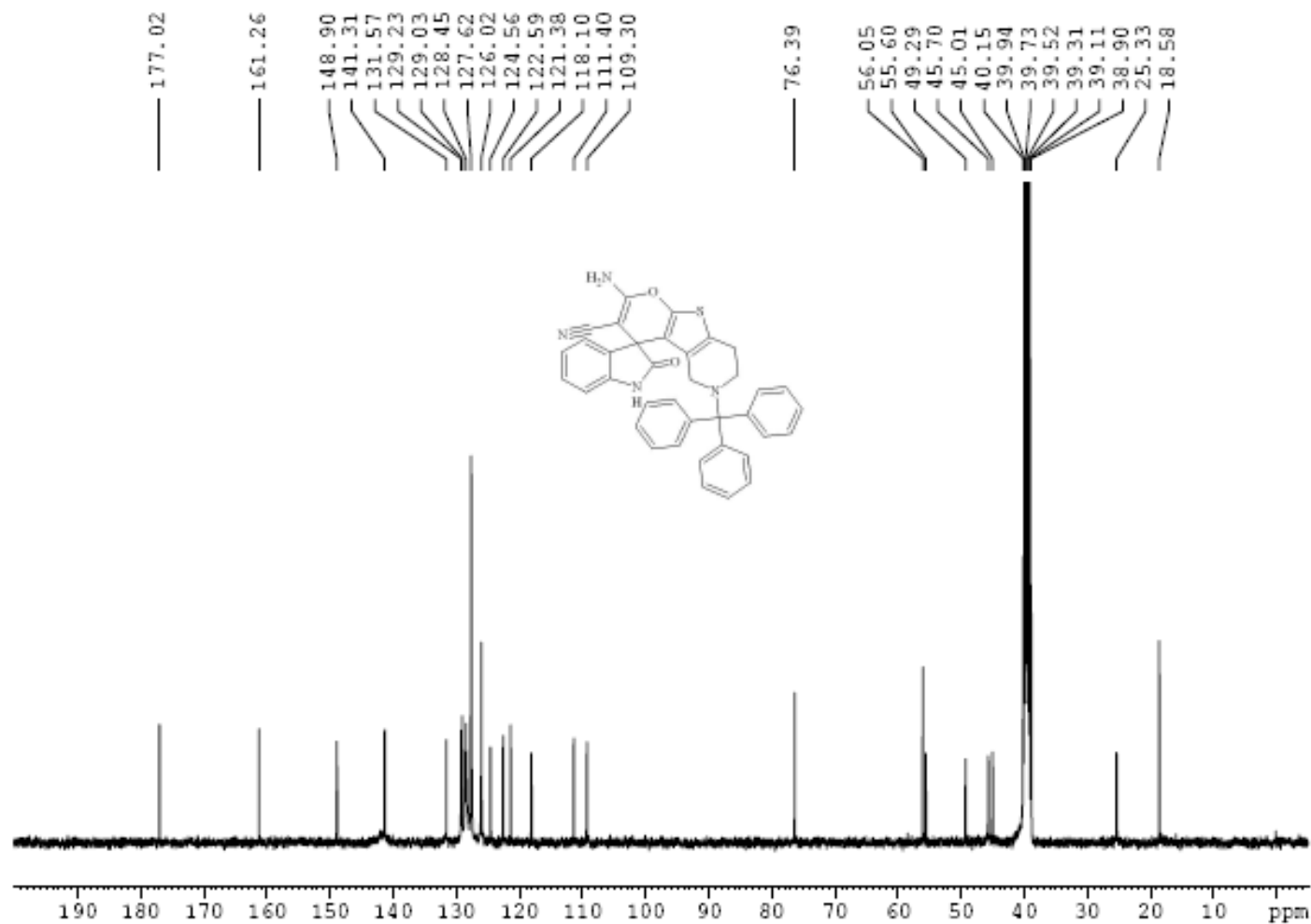
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EXPNO 58
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121203
Time 12:38
INSTRUM spect
PROBHD 5 mm MSL500
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 30
DS 0
SWH 7183.908 Hz
FIDRES 0.210235 Hz
AQ 2.2807028 sec
RG 71.8
DWM 69.600 msec
DE 6.50 msec
TE 295.1 K
D1 1.0000000 sec
TD0 1

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NUC1 1H
P1 7.05 msec
PL1 3.00 dB
SFO1 400.1332010 MHz

F2 - Processing parameters
SI 16384
SF 400.1300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹H NMR spectrum of 5a



Current Data Parameters
NAME DECI2
EXPNO 59
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121208
Time 13.11
INSTRUM spect
PROBHD 5 mm Multinuc
PULPROG zgpg
TD 32000
SOLVENT DMSO
NS 877
DS 0
SWH 25125.629 Hz
FIDRES 0.785176 Hz
AQ 0.6368500 sec
RG 18390.4
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DE 6.50 usec
TE 295.5 K
D1 5.00000000 sec
d11 0.03000000 sec
TD0 1

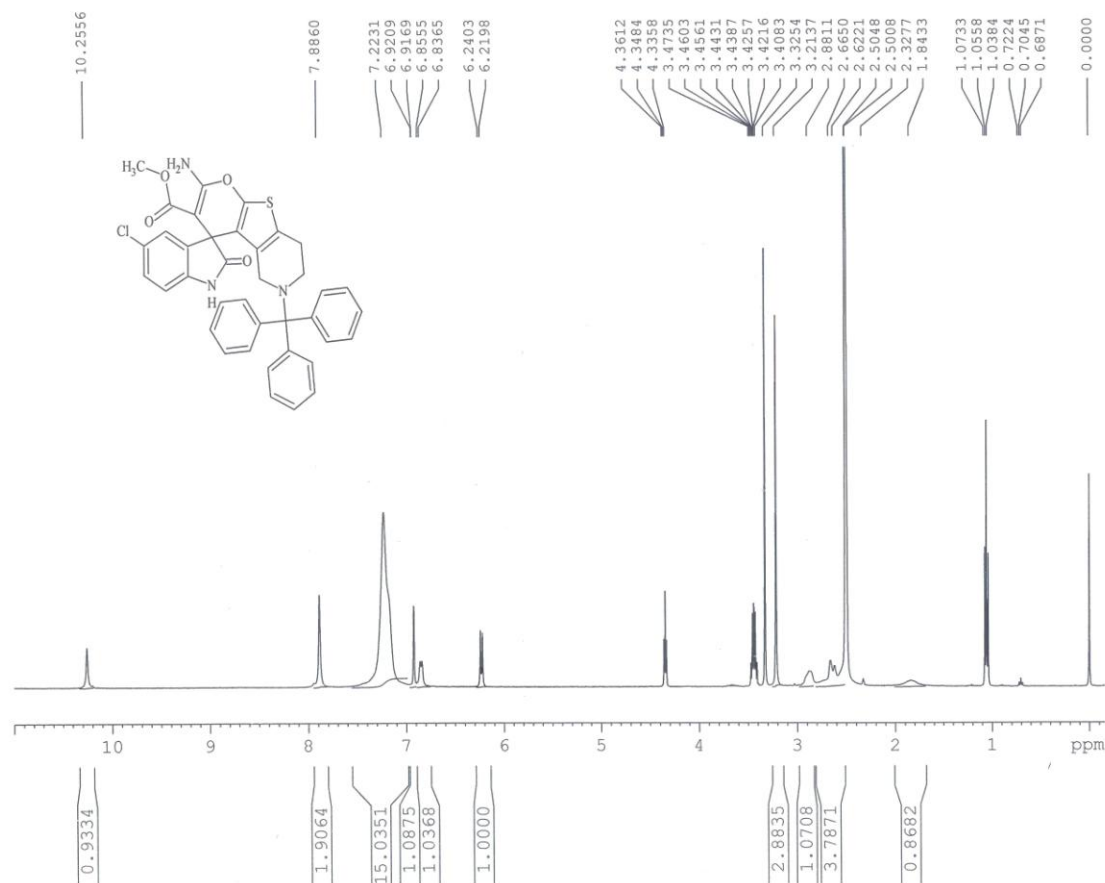
CHANNEL f1
NUC1 13C
P1 11.50 usec
PL1 2.00 dB
SFO1 100.6257964 MHz

CHANNEL f2
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NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.19 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
S1 32768
SF 100.6128160 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 3.00

¹³C NMR spectrum of 5a

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
Ph.D. KP/S-II/03/5-ClISATRITHIMCA REF:NMR/1580/64/24



Current Data Parameters
NAME JAN13
EXPNO 184
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130107
Time 13.19
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PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 71.8
DW 69.600 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 7.05 usec
PL1 3.00 dB
SFO1 400.1332010 MHz

F2 - Processing parameters
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SF 400.1300046 MHz
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LB 0.30 Hz
GB 0
PC 1.00

¹H NMR spectrum of 5c



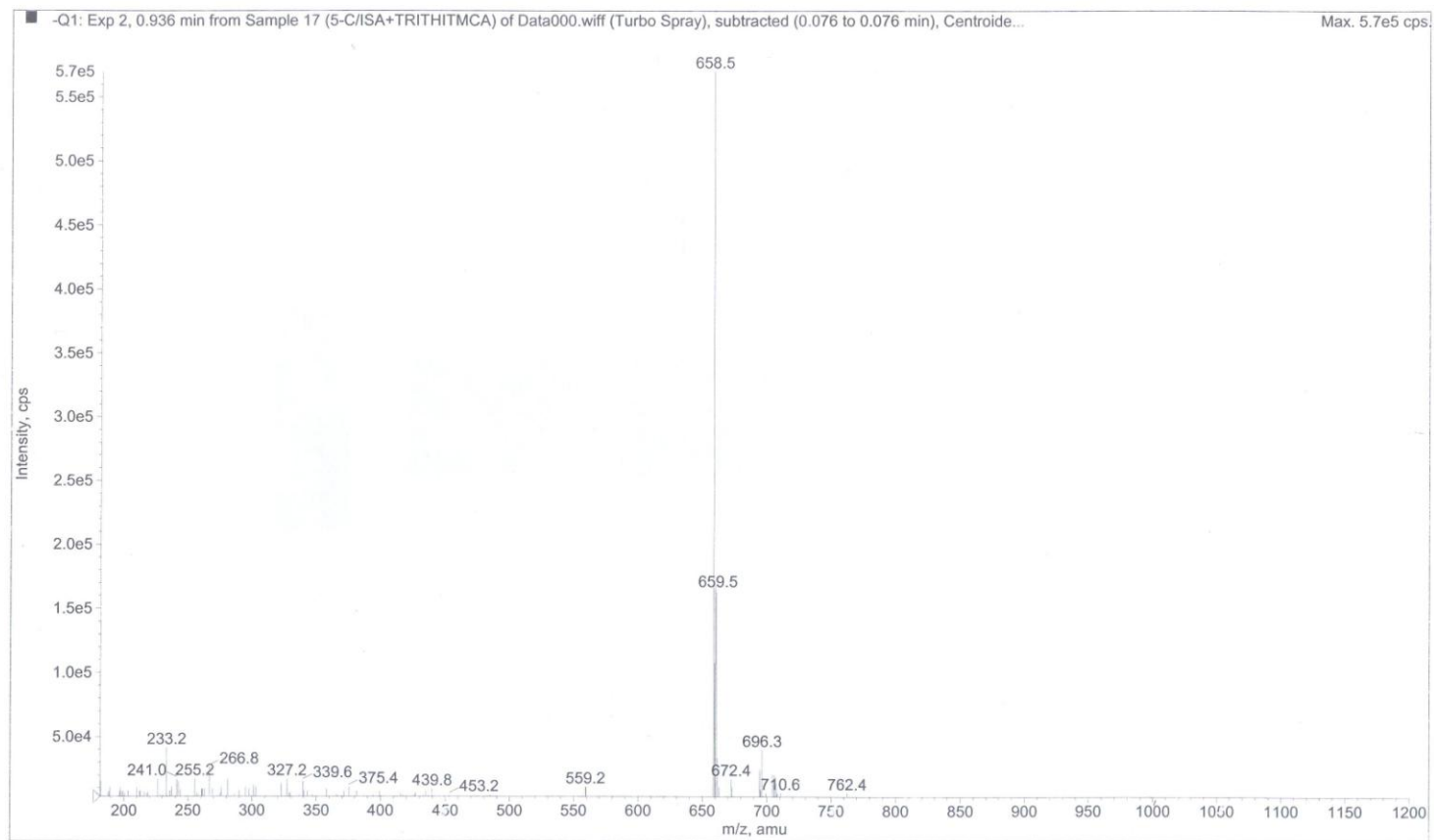
Acq. Date: Friday, May 24, 2013

ORCHID CHEMICALS AND PHARMACEUTICALS LTD., Printing Date: May 24, 2013

Acq. Time: 11:09

Sample Name: 5-C/ISA+TRITHITMCA

Sample ID:

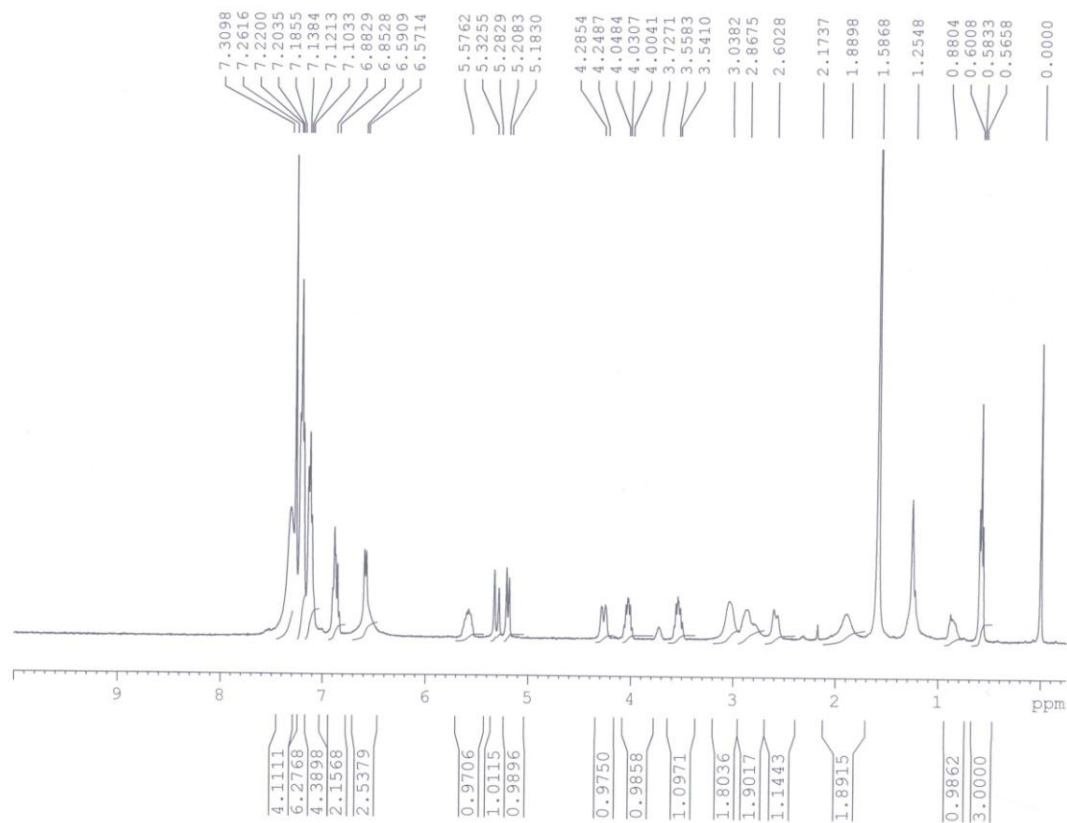


Project: ORCHID

REF PAGE: HKA/1577/

Mass spectrum of 5c

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
N-Allylisaecatriti REF:NMR/1610/100/11



Current Data Parameters
NAME jul13
EXPNO 422
PROCNO 1

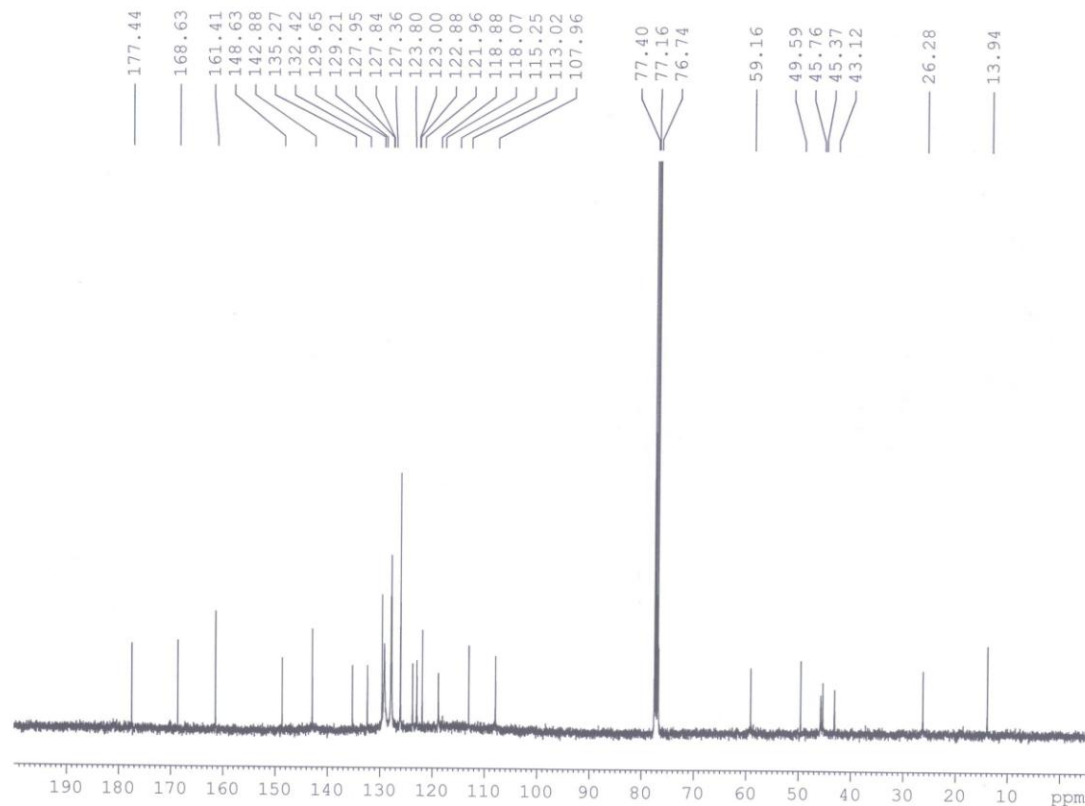
F2 - Acquisition Parameters
Date_ 20130731
Time 12.24
INSTRUM spect
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 57
DW 69.600 usec
DE 6.00 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
PI 9.00 usec
PL1 3.00 dB
SFO1 400.1332010 MHz

F2 - Processing parameters
SI 32768
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 5.00

¹H NMR spectrum of 5i

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
n-allysisaecatriti-S REF:NMR/1610/99/23



Current Data Parameters
NAME jul13
EXPNO 411
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130730
Time 19.53
INSTRUM spect
PROBHD 5 mm SEI 1H-BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3072
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1

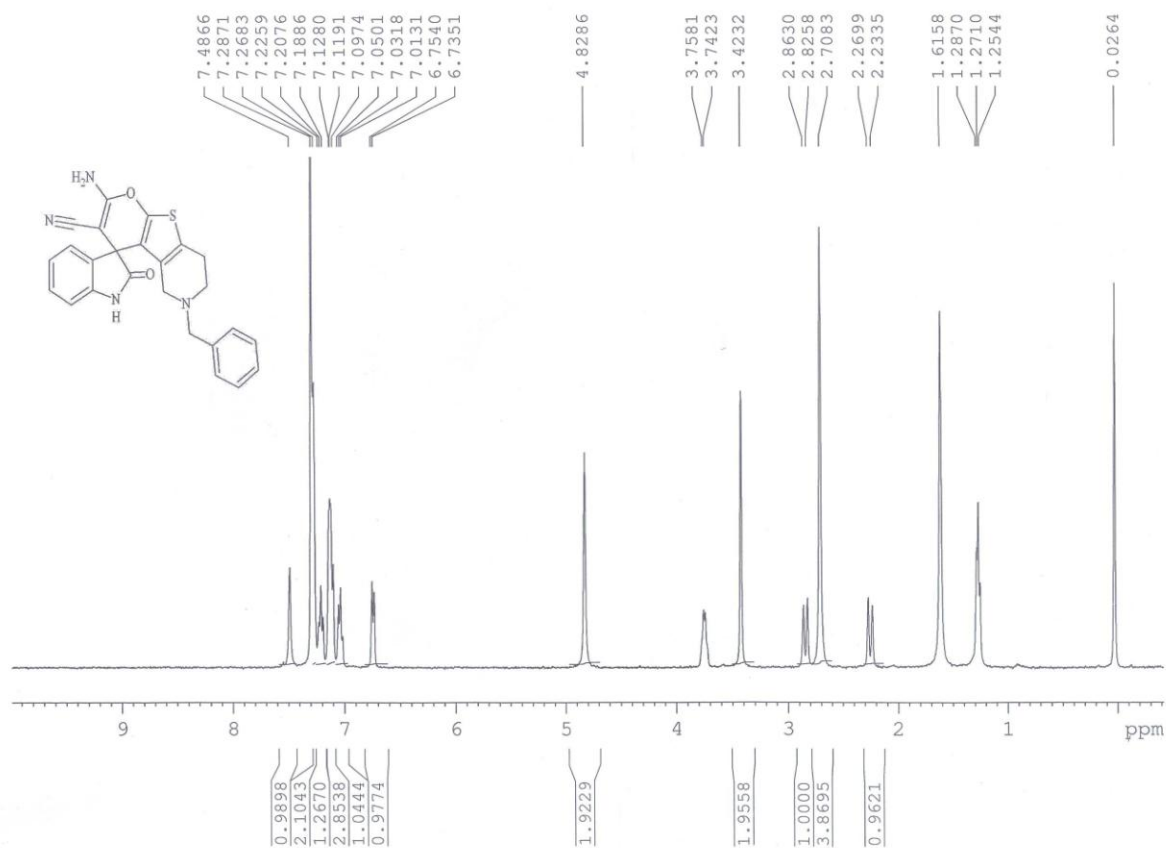
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NUC1 13C
P1 12.00 usec
PL1 -3.00 dB
SFO1 100.6228300 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 16.79 dB
SFO2 400.1316000 MHz

F2 - Processing parameters
SI 32768
SF 100.6127566 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

¹³C NMR spectrum of 5i

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
Ph.D ISATIN+MN+BENZ-12 REF:NMR/1610/39/14



Current Data Parameters
NAME may13
EXPNO 113
PROCNO 1

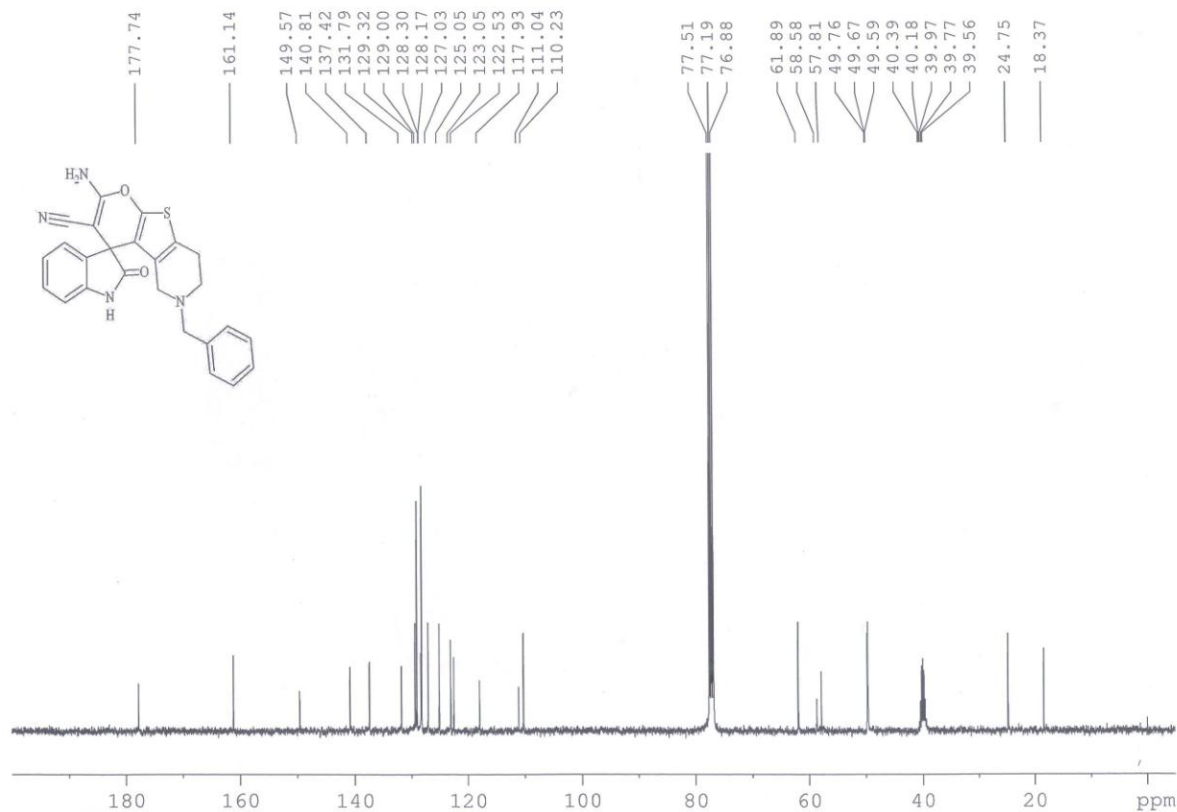
F2 - Acquisition Parameters
Date_ 20130509
Time 18.31
INSTRUM spect
PROBHD 5 mm Multinuc
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 57
DW 69.600 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 :
NUC1 1H
P1 7.05 usec
PL1 3.00 dB
SFO1 400.1332010 MI

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

¹H NMR spectrum of 5j

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
Ph.D ISATIN+MN+BENZ-12 REF:NMR/1610/39/13



Current Data Parameters
NAME may13
EXPNO 112
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130509
Time 17.22
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgdc
TD 32000
SOLVENT CDCl3
NS 1262
DS 0
SWH 25125.629 Hz
FIDRES 0.785176 Hz
AQ 0.6368500 sec
RG 18390.4
DW 19.900 usec
DE 6.50 usec
TE 295.8 K
D1 5.00000000 sec
d11 0.03000000 sec
TD0 1

===== CHANNEL f1 ==
NUC1 13C
P1 14.00 usec
PL1 2.00 dB
SFO1 100.6237964 MHz

===== CHANNEL f2 ==
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.19 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6132489 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 3.00

¹³C NMR spectrum of 5j

Acq. Date: Friday, May 24, 2013

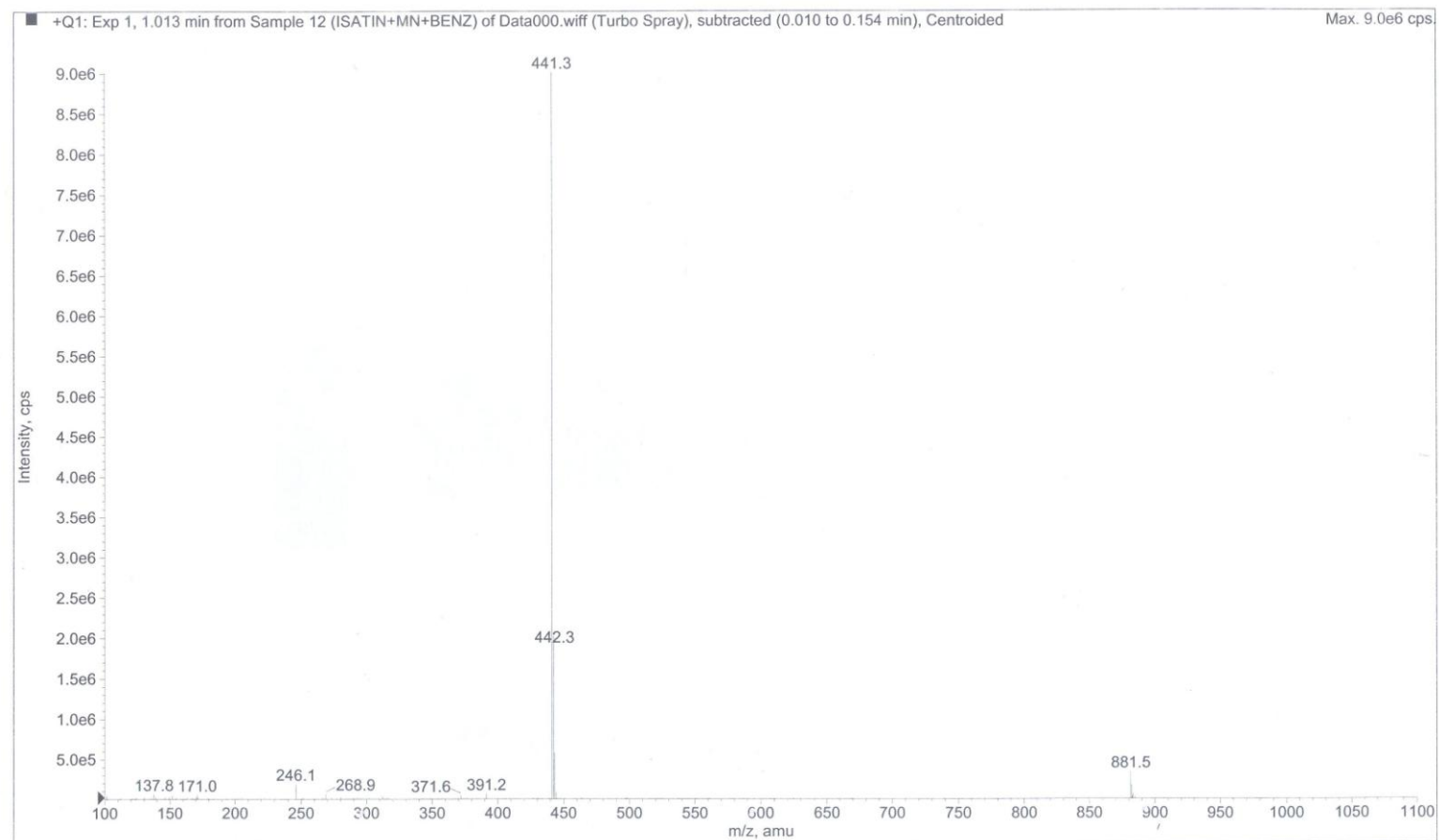
ORCHID CHEMICALS AND PHARMACEUTICALS LTD.,

Printing Date: May 24, 2013

Acq. Time: 10:42

Sample Name: ISATIN+MN+BENZ

Sample ID:

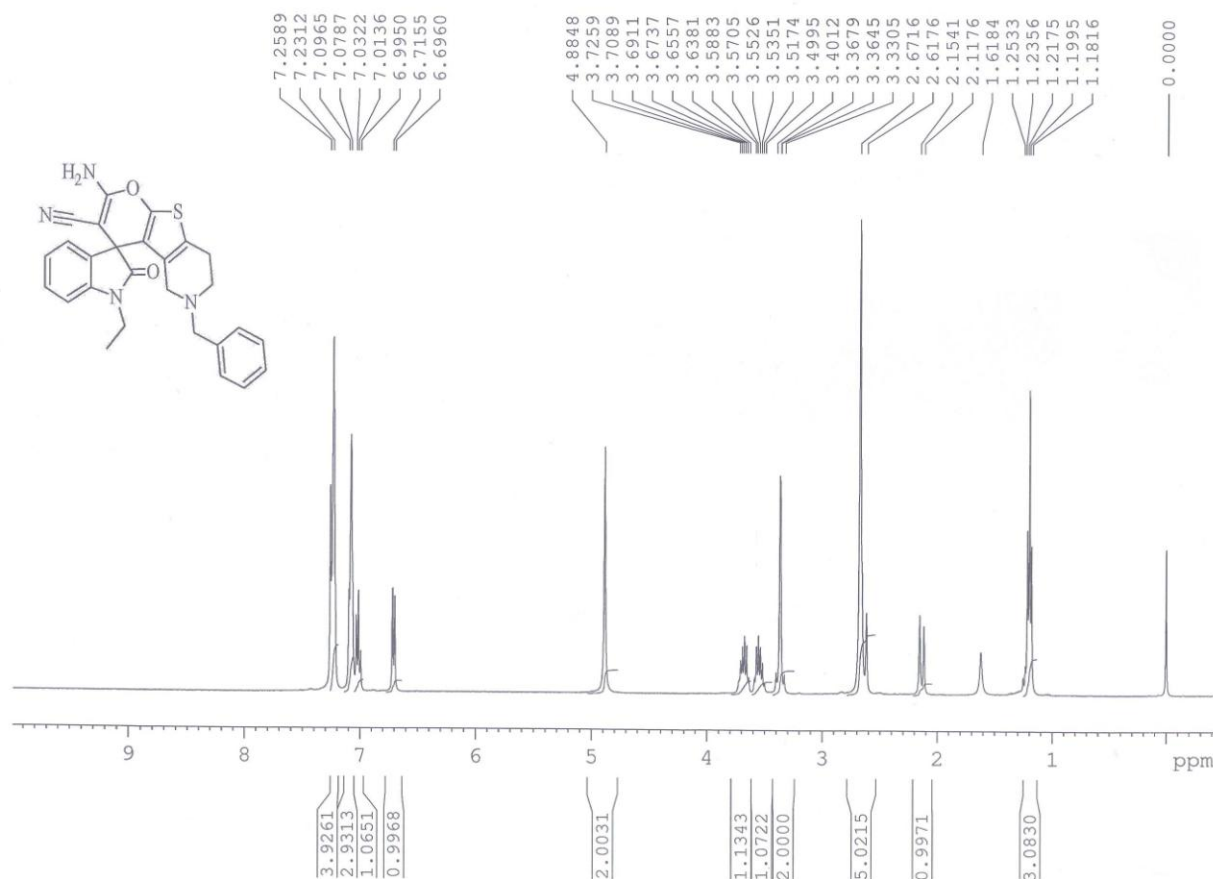


Project: ORCHID

REF PAGE: HKA/1577/

Mass spectrum of 5j

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
N-Ethyl ISA+BETHI+MN REF:NMR/1610/32/10



Current Data Parameters
NAME may13
EXPNO 10
PROCNO 1

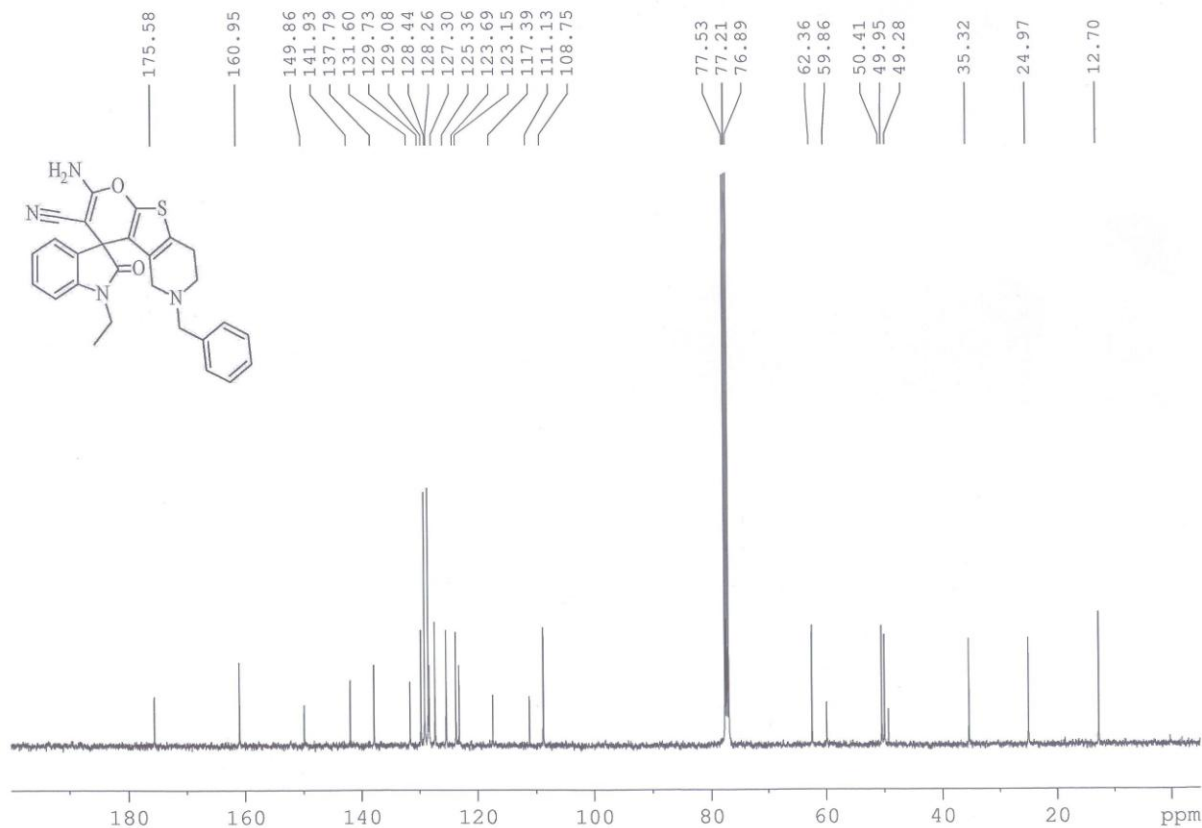
F2 - Acquisition Parameters
Date_ 20130502
Time 16.38
INSTRUM spect
PROBHD 5 mm Multinuc
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 406.4
DW 69.600 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 :
NUC1 1H
P1 7.05 usec
PL1 3.00 dB
SFO1 400.1332010 MI

F2 - Processing parameters
SI 16384
SF 400.1300116 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

¹H NMR spectrum of 5k

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
N-Ethyl ISA+BETHI+MN REF:NMR/1610/32/11



Current Data Parameters
NAME may13
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130502
Time 17.09
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgdc
TD 32000
SOLVENT CDCl3
NS 1202
DS 0
SWH 25125.629 Hz
FIDRES 0.785176 Hz
AQ 0.6368500 sec
RG 18390.4
DW 19.900 usec
DE 6.50 usec
TE 297.6 K
D1 5.00000000 sec
d11 0.03000000 sec
TD0 1

===== CHANNEL f1 ==
NUC1 13C
P1 11.50 usec
PL1 2.00 dB
SFO1 100.6237964 MHz

===== CHANNEL f2 ==
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.19 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
S1 32768
SF 100.6127537 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 3.00

¹³C NMR spectrum of 5k

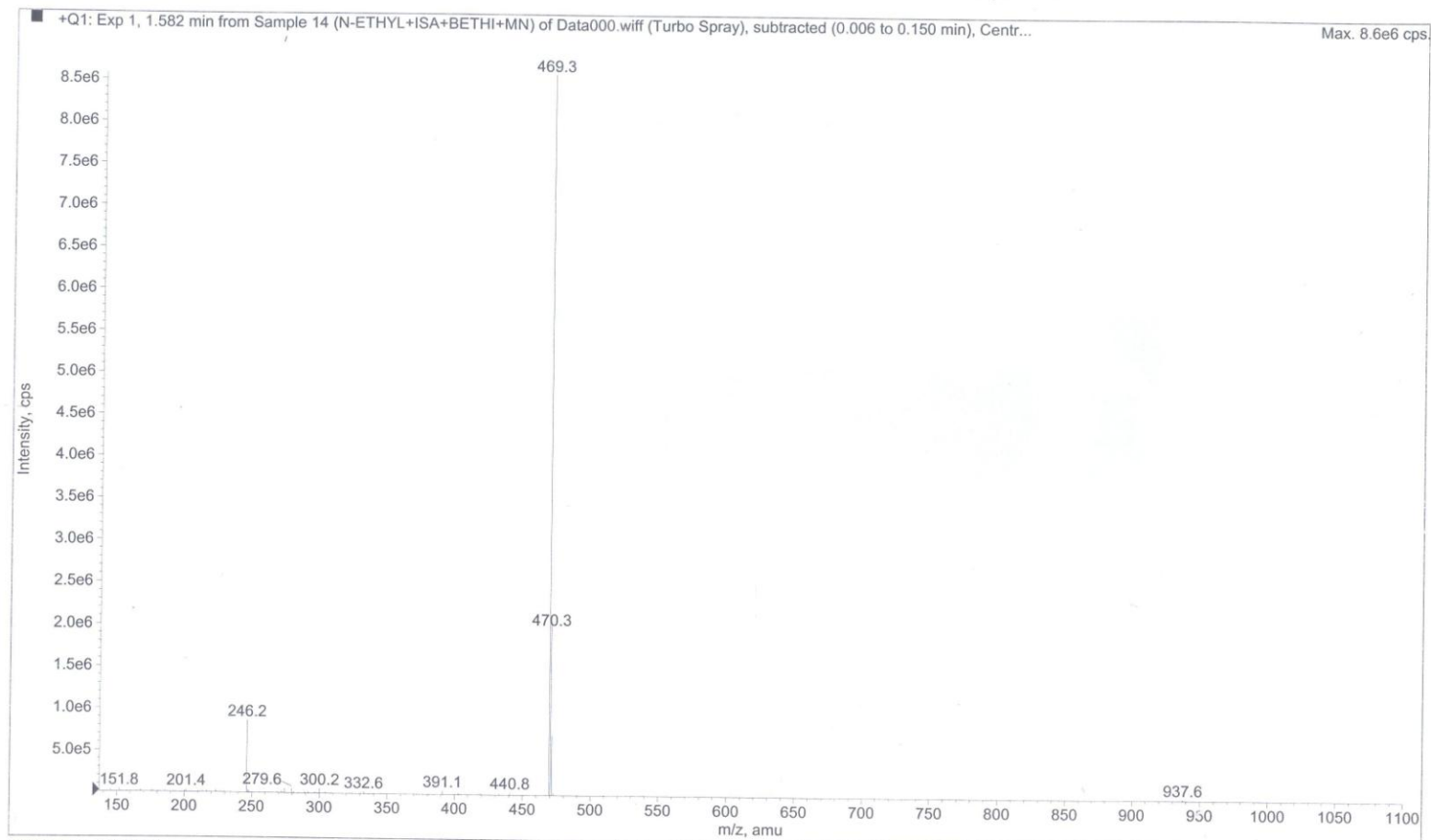
Acq. Date: Friday, May 24, 2013

ORCHID CHEMICALS AND PHARMACEUTICALS LTD., Printing Date: May 24, 2013

Acq. Time: 10:48

Sample Name: N-ETHYL+ISA+BETHI+MN

Sample ID:



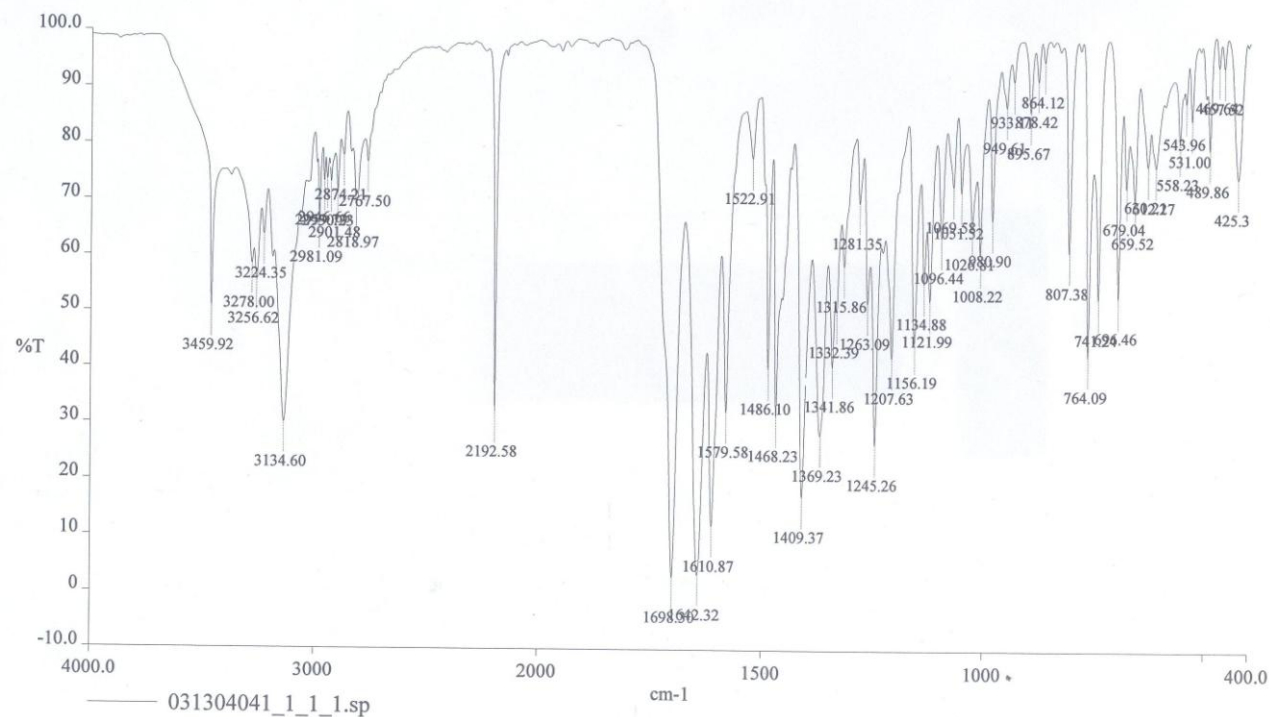
Project: ORCHID

REF PAGE: HKA/1577/

Mass spectrum of 5k

ORCHID CHEMICALS & PHARMACEUTICALS LIMITED

R&D CENTRE, CHENNAI



Spectrum Pathname: D:\DDRIVE\pel_data\spectra\spectra\2013\APR\031304041_1_1_1.sp

Date Created: Tuesday, April 23, 2013 11:53

Description: N-ETHYL ISA BETHI+MN

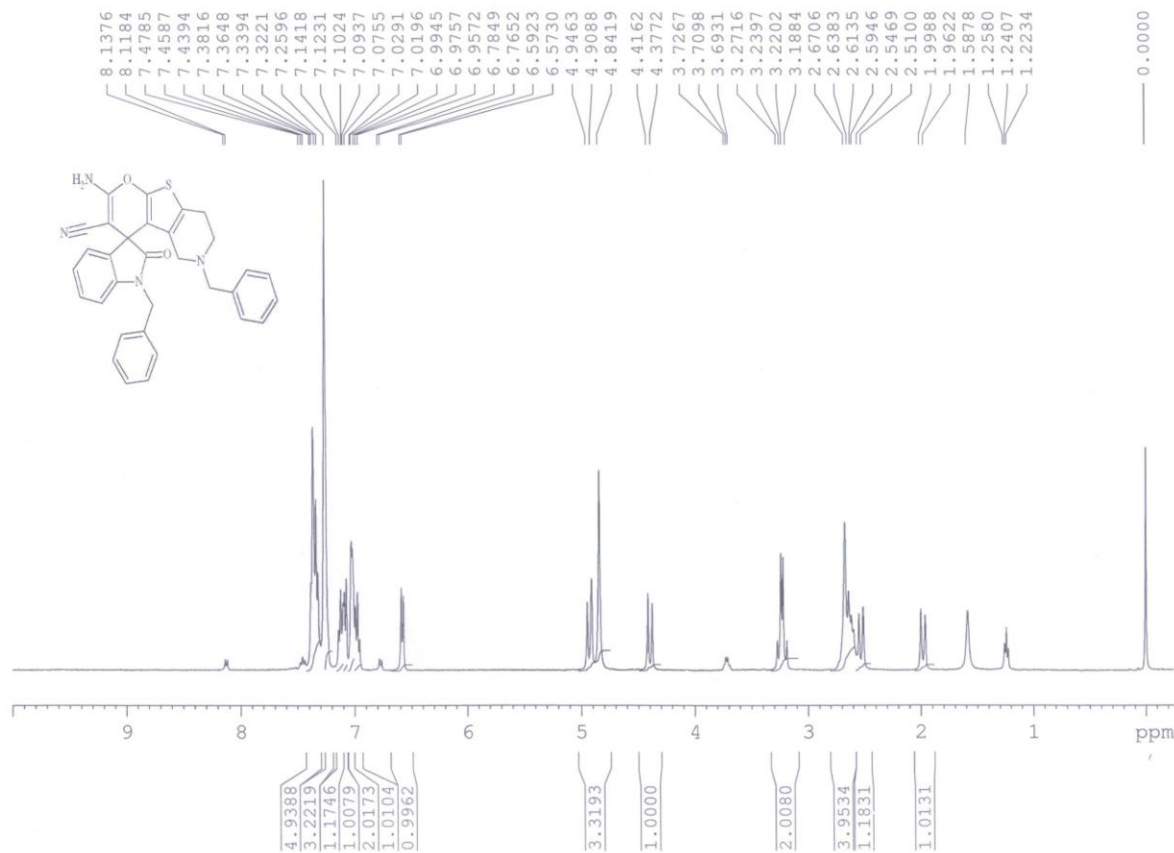
Analyst: *SV*

Technique: KBr/Neat/Nujol

Reference: *SD/1558/107/15*

IR spectrum of 5k

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
N-Benzyl ISATIN+MN+BENZTHIENE REF:NMR/1610/33/11



Current Data Parameters
NAME May13
EXPNO 31
PROCNO 1

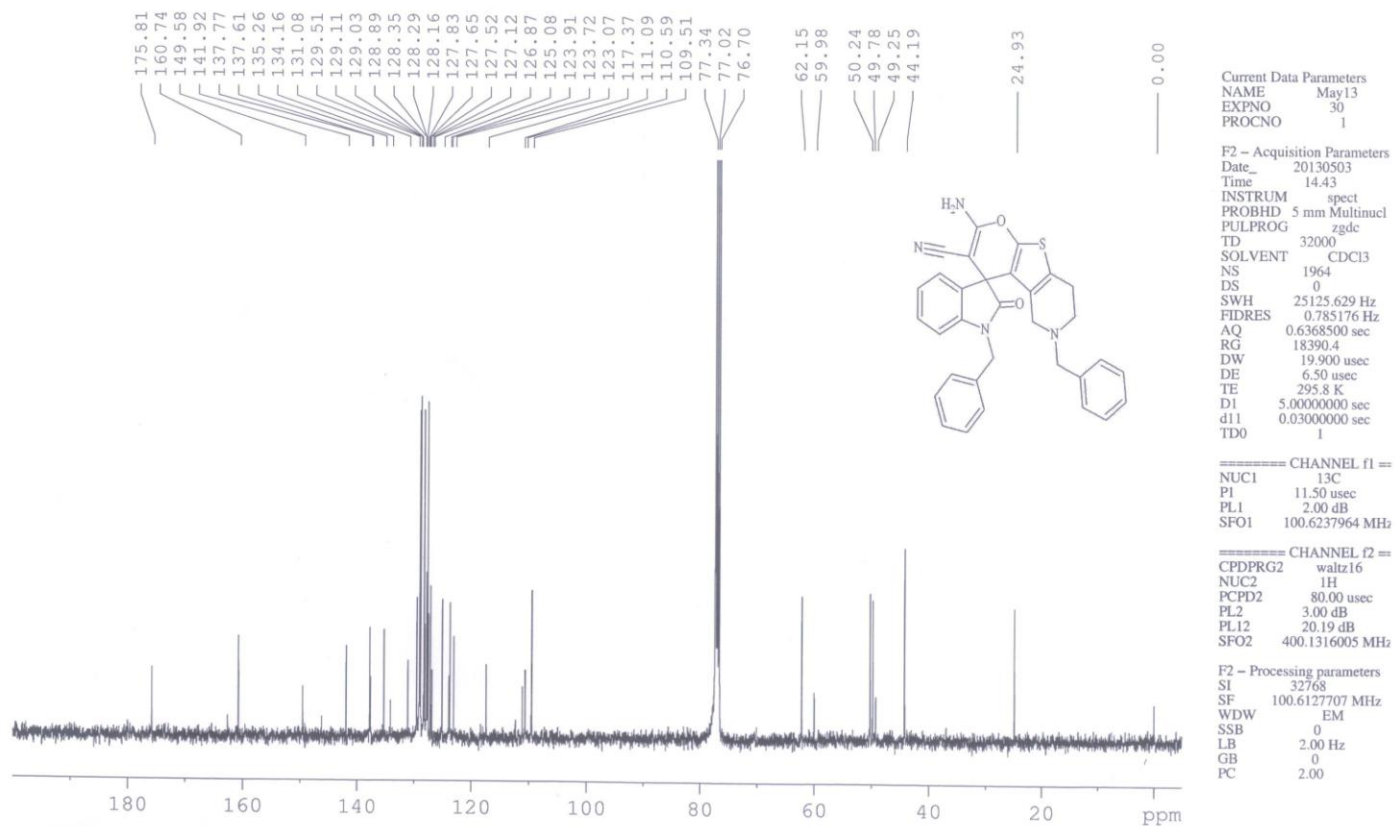
F2 - Acquisition Parameter
Date_ 20130503
Time 17.45
INSTRUM spect
PROBHD 5 mm Multinuc
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 57
DW 69.600 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 :
NUC1 1H
P1 7.05 usec
PL1 3.00 dB
SFO1 400.1332010 MI

F2 - Processing parameters
SI 16384
SF 400.1300113 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

¹H NMR spectrum of 5l

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
N-Benzyl ISA+BENZTHI+MN REF:NMR/1610/32/11



¹³C NMR spectrum of 51

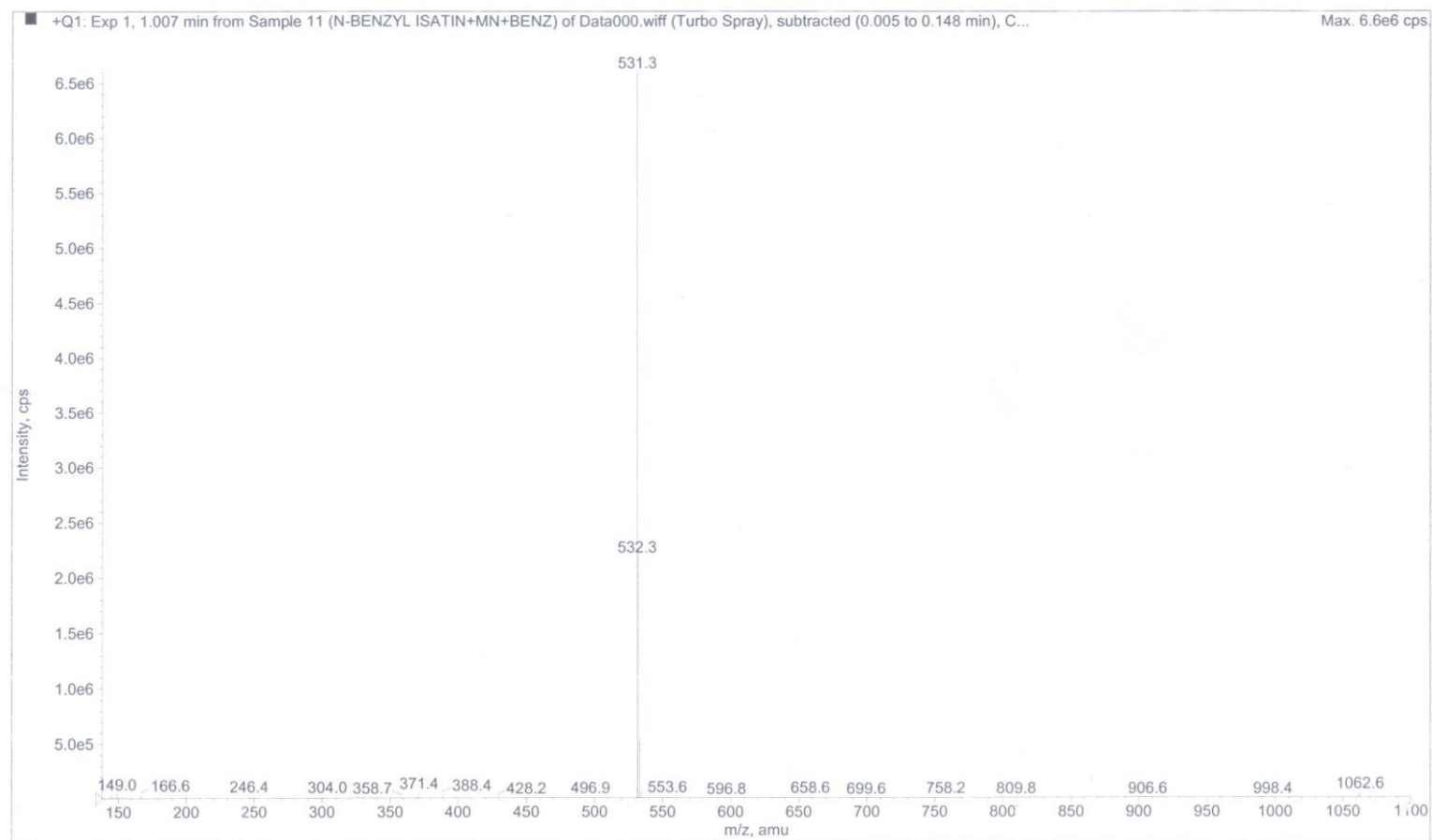
Acq. Date: Friday, May 24, 2013

ORCHID CHEMICALS AND PHARMACEUTICALS LTD., Printing Date: May 24, 2013

Acq. Time: 10:39

Sample Name: N-BENZYL ISATIN+MN+BENZ

Sample ID:



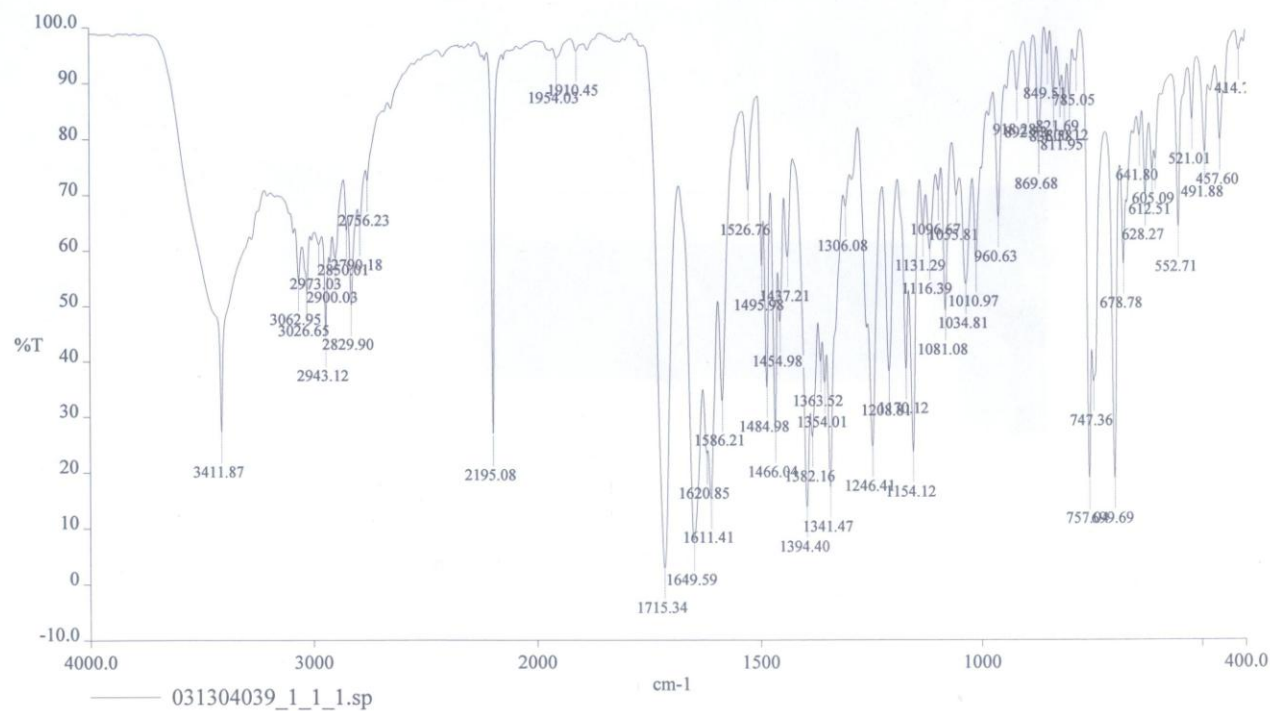
Project: ORCHID

REF PAGE: HKA/1577/

Mass spectrum of 5l

ORCHID CHEMICALS & PHARMACEUTICALS LIMITED

R&D CENTRE, CHENNAI



031304039_1_1_1.sp

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Date Created: Tuesday, April 23, 2013 11:39

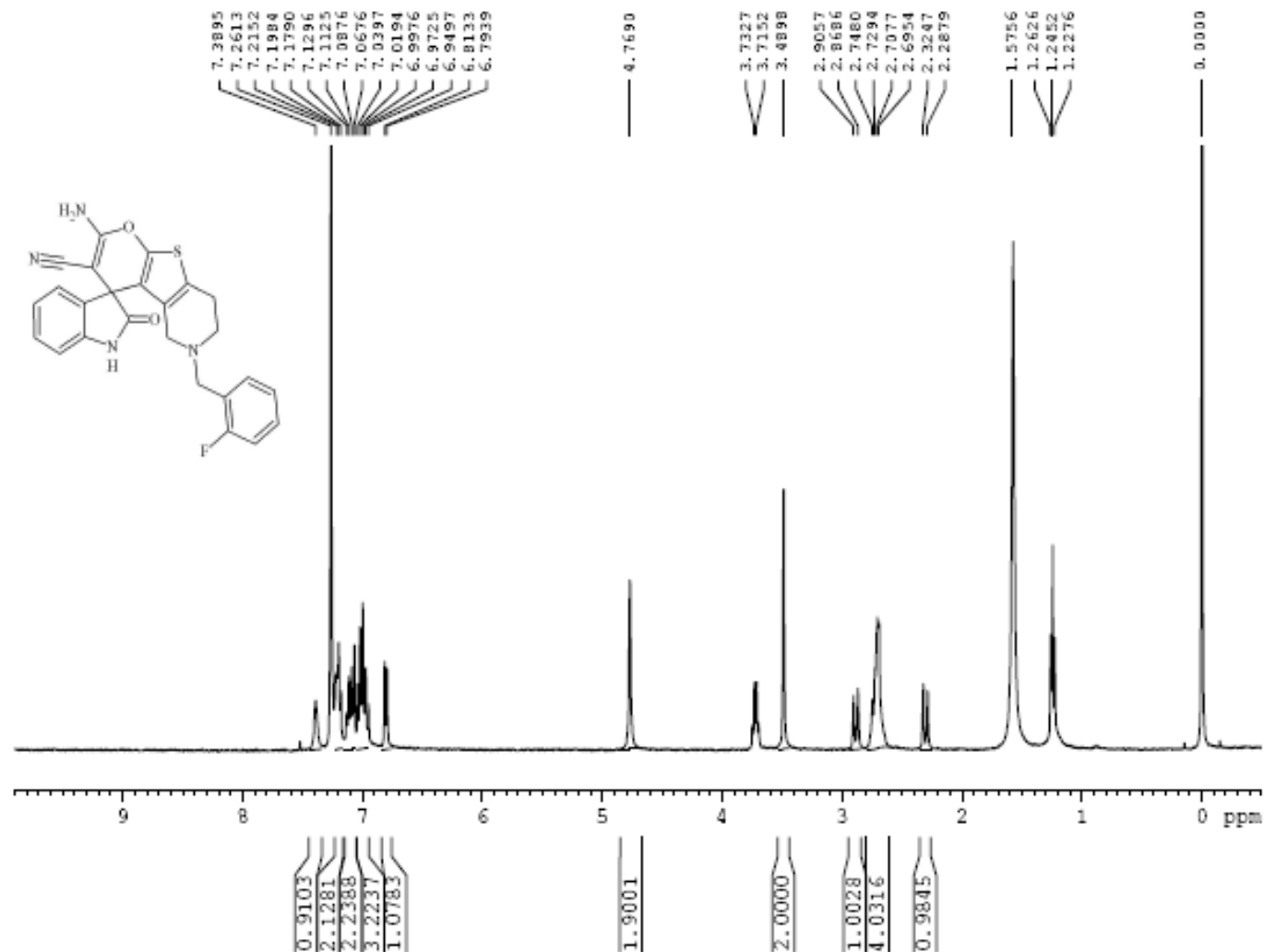
Description: N-BENZYL ISATIN+MN+BENZ-II

Analyst: *23/4/13*

Technique: KBr/Neat/Nujol

Reference: *20/1555/107/13*

IR spectrum of 5l



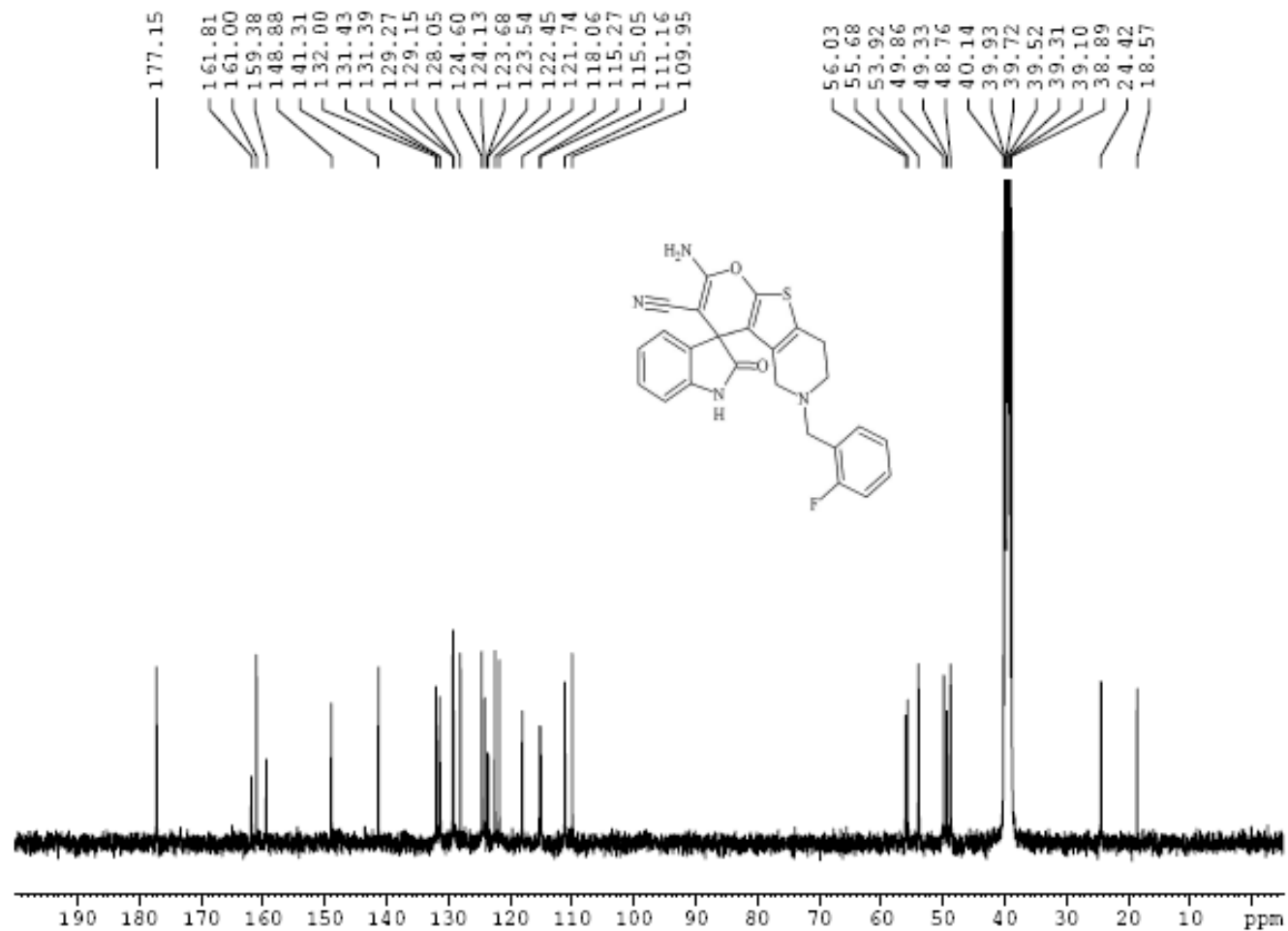
Current Data Parameters
NAME FEB13
EXPNO 488
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130220
Time 18.47
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PROBHD 5 mm Mxlineci
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 80.6
DW 69.600 usec
DE 6.50 usec
TE 296.2 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 7.05 usec
PL1 3.00 dB
SFO1 400.133010 MHz

F2 - Processing parameters
SI 16384
SF 400.1300105 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 3.00

¹H NMR spectrum of 50



Current Data Parameters
NAME FEB13
EXPNO 386
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130227
Time 12.45
INSTRUM spect
PROBHD 5 mm Multinuc
PULPROG zgpg30
TD 32000
SOLVENT DMSO
NS 687
DS 0
SWH 25125.629 Hz
FIDRES 0.785176 Hz
AQ 0.6368500 sec
RG 23170.5
DW 19.900 usec
DE 6.50 usec
TE 295.8 K
D1 5.00000000 sec
d11 0.03000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 13C
P1 11.50 usec
PL1 2.00 dB
SFO1 100.6237964 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.19 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
S1 32768
SF 100.6128166 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

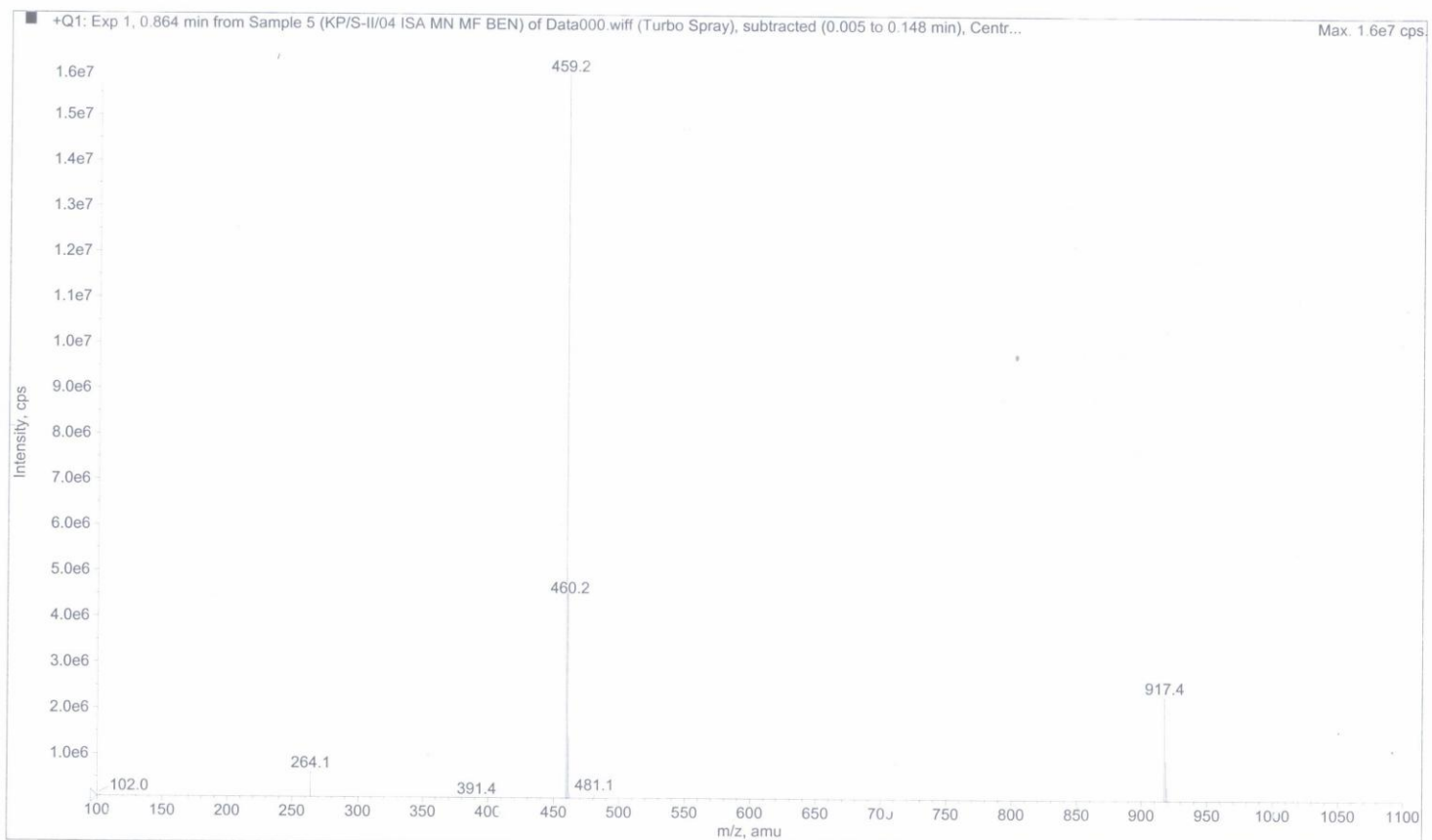
¹³C NMR spectrum of 50

Acq. Date: Friday, May 24, 2013

ORCHID CHEMICALS AND PHARMACEUTICALS LTD., Printing Date: May 24, 2013

Acq. Time: 10:21

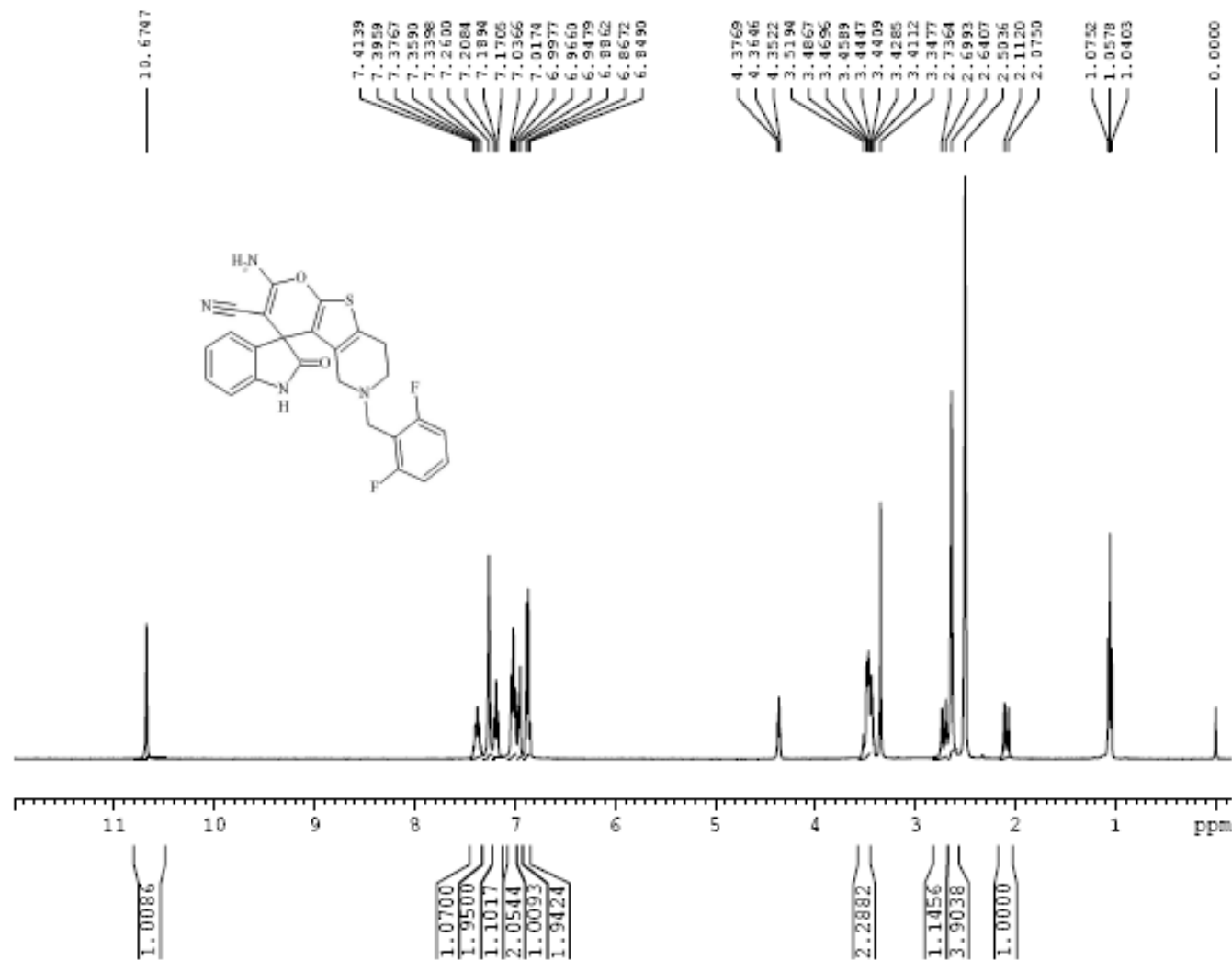
Sample Name: KP/S-II/04 ISA MN MF BEN Sample ID:



Project: ORCHID

REF PAGE: HKA/1577/

Mass spectrum of 5o



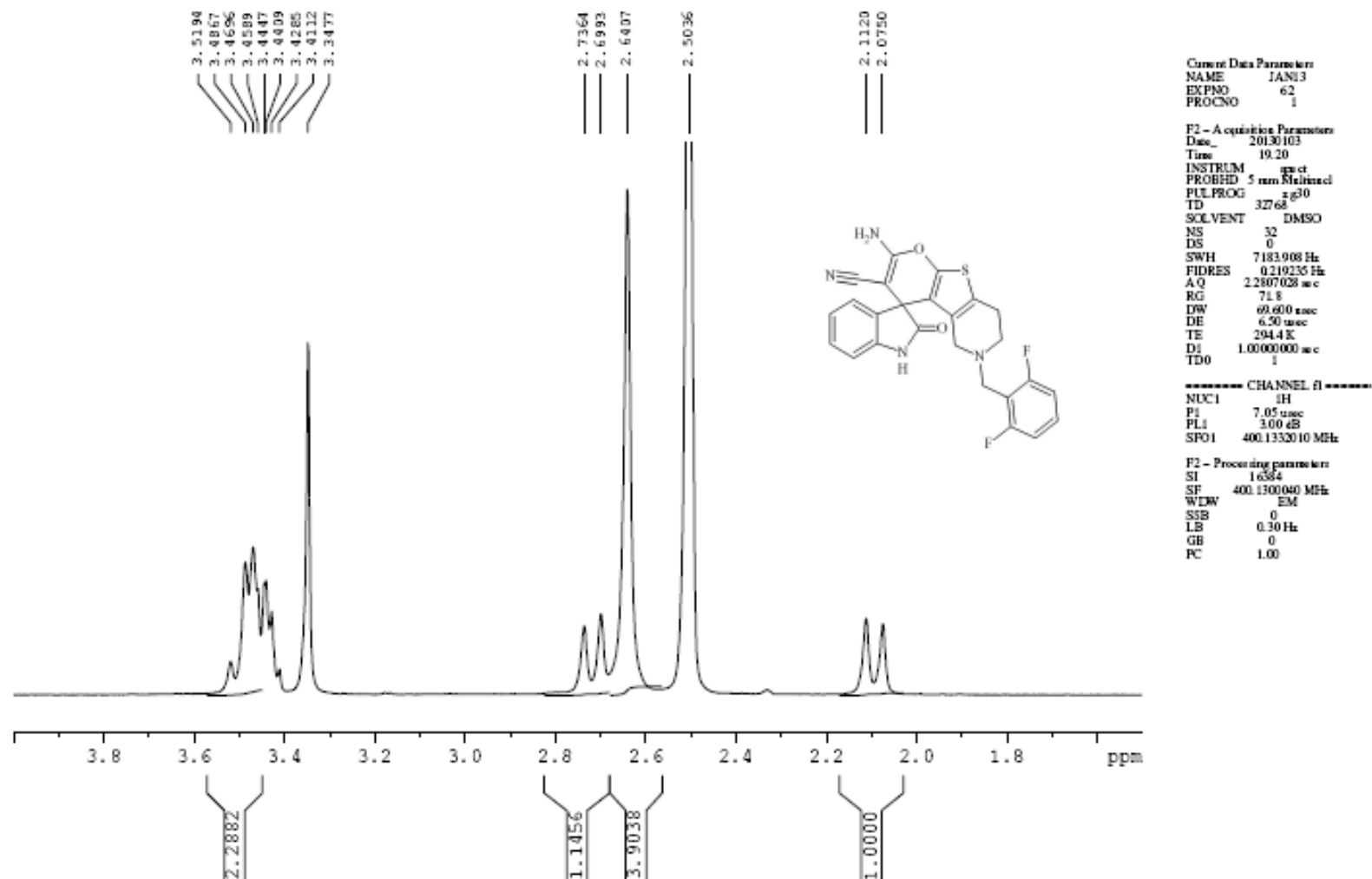
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NAME JAN13
EXPNO 62
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130103
Time 19.20
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PROBHD 5 mm Mxlinecl
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 71.8
DWB 69.600 msec
DE 6.30 msec
TE 294.4 K
D1 1.00000000 sec
TD0 1

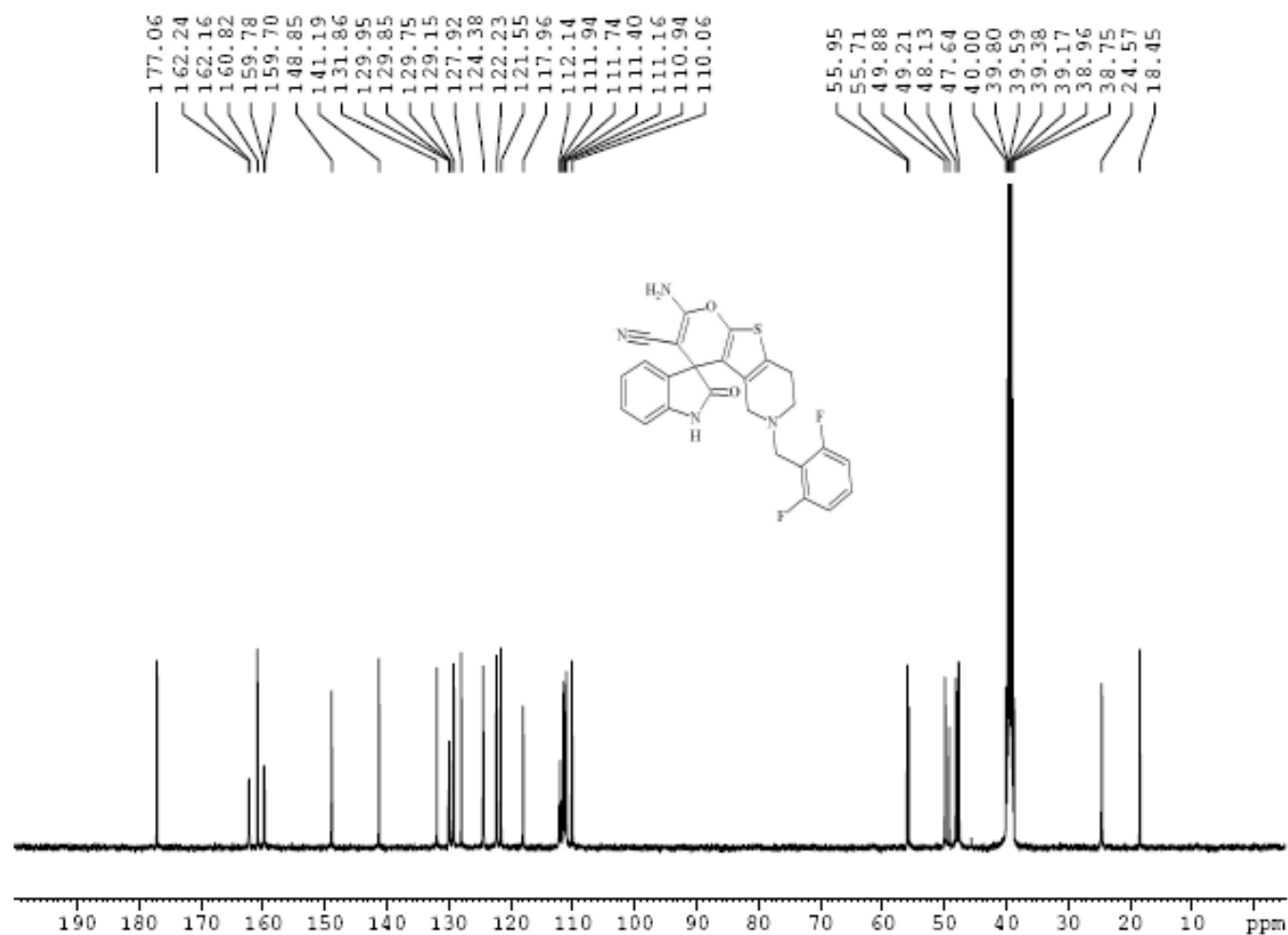
----- CHANNEL f1 -----
NUC1 1H
P1 7.05 msec
PL1 3.00 dB
SFO1 400.1332010 MHz

F2 - Processing parameters
SI 16384
SF 400.1300040 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹H NMR spectrum of 5t



Expanded ^1H NMR spectrum of 5t



Current Data Parameters
NAME JAN13
EXPNO 61
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130103
Time 18.28
INSTRUM spect
PROBHD 5 mm Multispec
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 670
DS 0
SWH 25125.629 Hz
FIDRES 0.785176 Hz
AQ 0.6368500 sec
RG 18390.4
DW 19.900 usec
DE 6.50 usec
TE 295.0 K
D1 5.00000000 sec
d11 0.03000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 13C
P1 11.50 usec
PL1 2.00 dB
SFO1 100.6237964 MHz

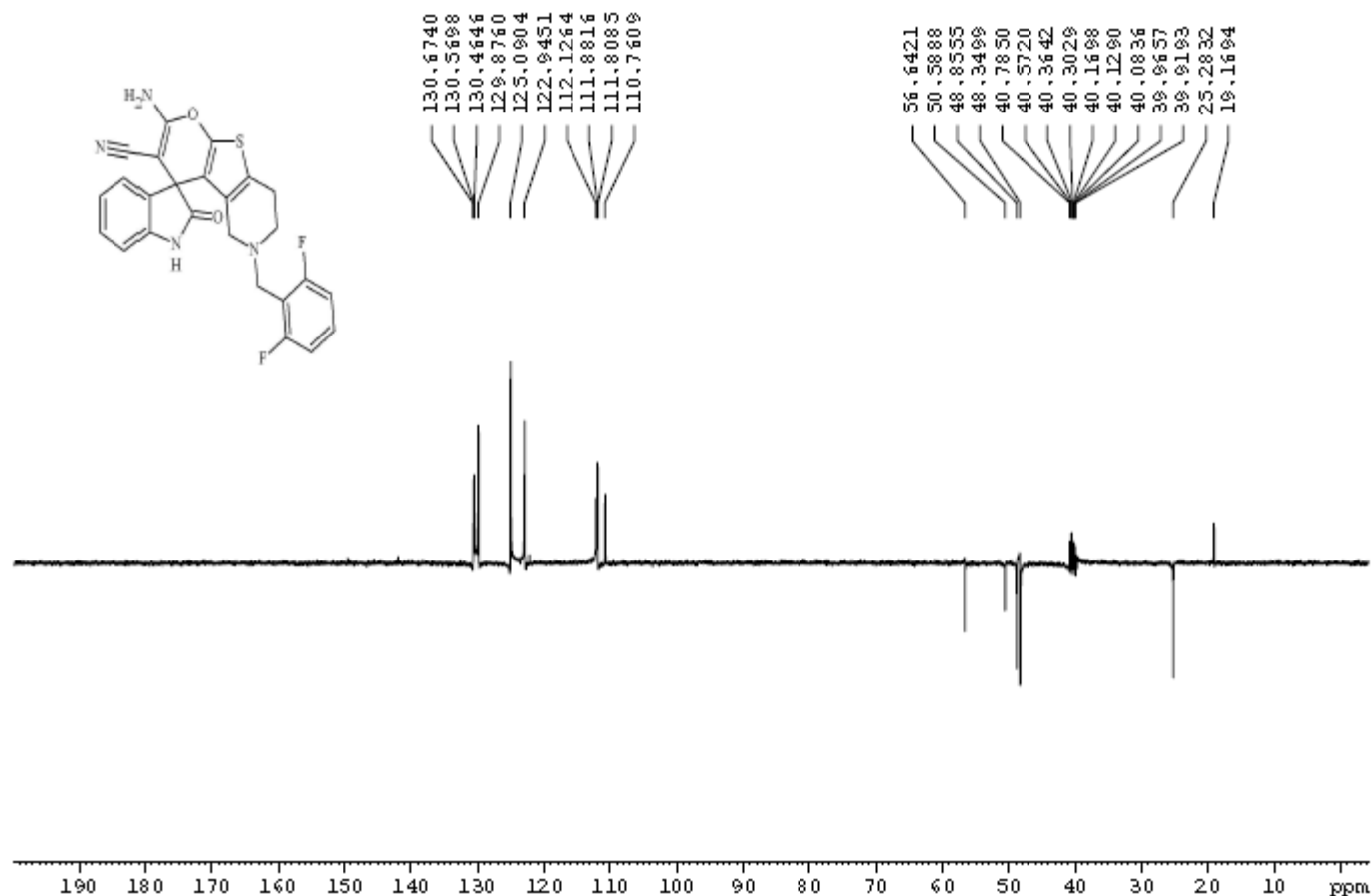
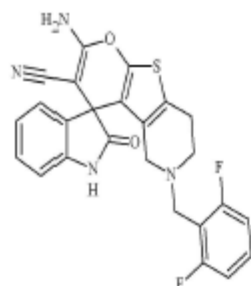
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CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.19 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
S1 32768
SF 100.6128272 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

^{13}C NMR spectrum of 5t

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
ISA+DIFBENTHI+MN KP/S-II/02

REF:NMR/1610/32/17



Current Data Parameters
NAME May13
EXPNO 17
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130503
Time 0.06
INSTRUM spect
PROBHD 5 mm Multispec1
PULPROG zgpg30
TD 32768
SOLVENT H2O
NS 3072
DS 8
SWH 24154.530 Hz
FIDRES 0.737190 Hz
AQ 0.6783476 sec
RG 32768
SI 20.700 usec
DE 6.50 usec
TE 236.5 K
QNP2 130.0000000
D1 3.00000000 sec
d2 0.00357143 sec
d12 0.00002000 sec
DELTA 0.00001964 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 11.50 usec
P2 23.00 usec
PL1 2.00 dB
PL2 100.6227303 dB
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P3 11.05 usec
P4 22.10 usec
PCPD2 30.00 usec
PL3 3.00 dB
PL4 20.13 dB
PL5 900.1320791 dB

F2 - Processing parameters
SI 32768
SF 100.6127556 MHz
RG 0
SWH 0
LE 2.00 Hz
GB 0
PC 1.00

DEPT-135 ^{13}C NMR of compound 5t

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
ISA+DIFBENTHI+MN KP/S-II/02 REF:NMR/1610/32/18



Current Data Parameters:
NAME may11
EXPNO 18
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130501
Time 0.01
INSTRUM spect
PROBHD 5 mm hmltinal
PULPROG cosygpgp
TD 2048
SOLVENT DMSO
NS 2
DS 14
SWH 5995.204
FIDRES 2.927344
AQ 0.1708532
RG 181
DQ 83.400
DE 6.50
TE 296.2
AQ 0.00000300
D1 2.00000000
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IN0 0.00016680

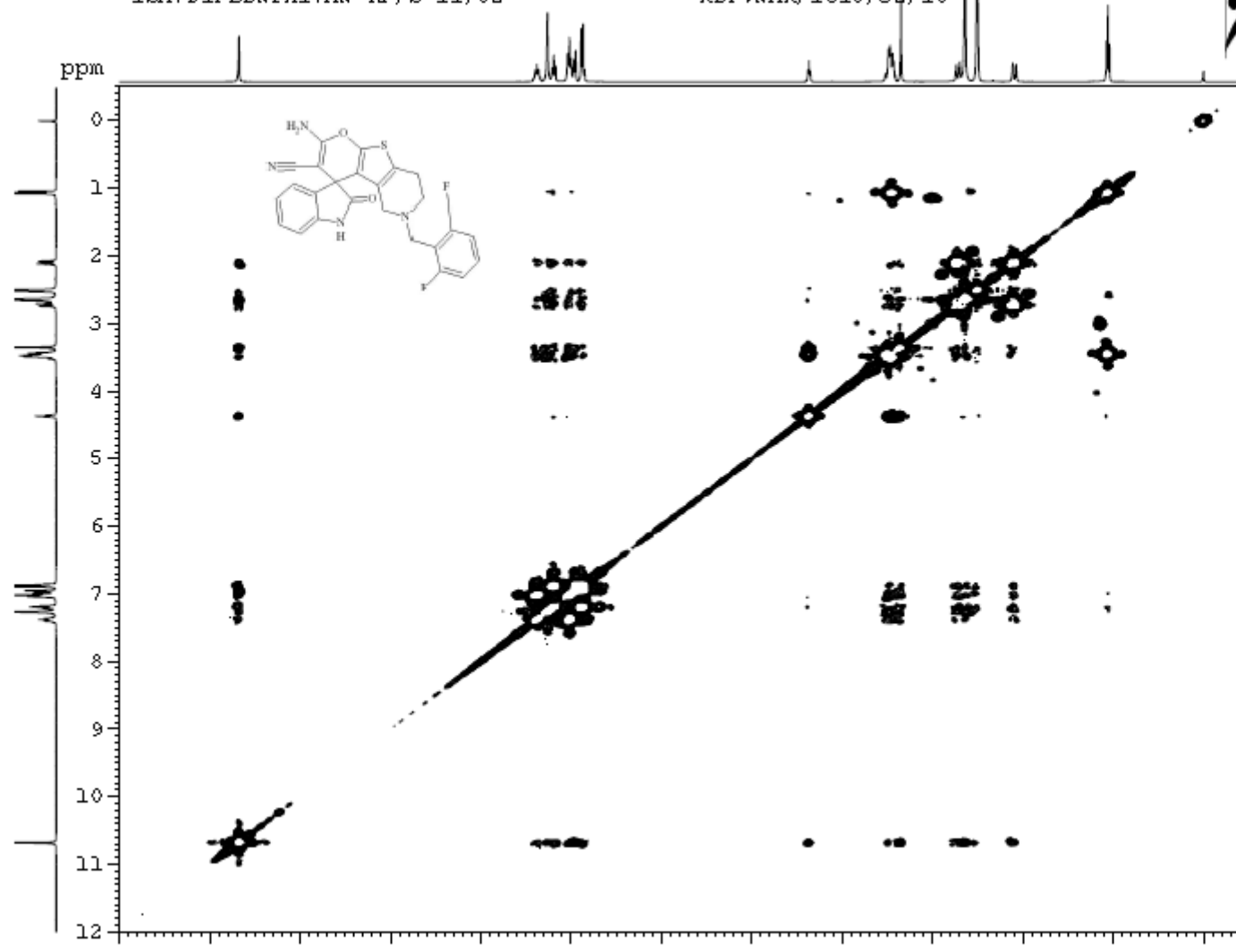
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P1 11.05
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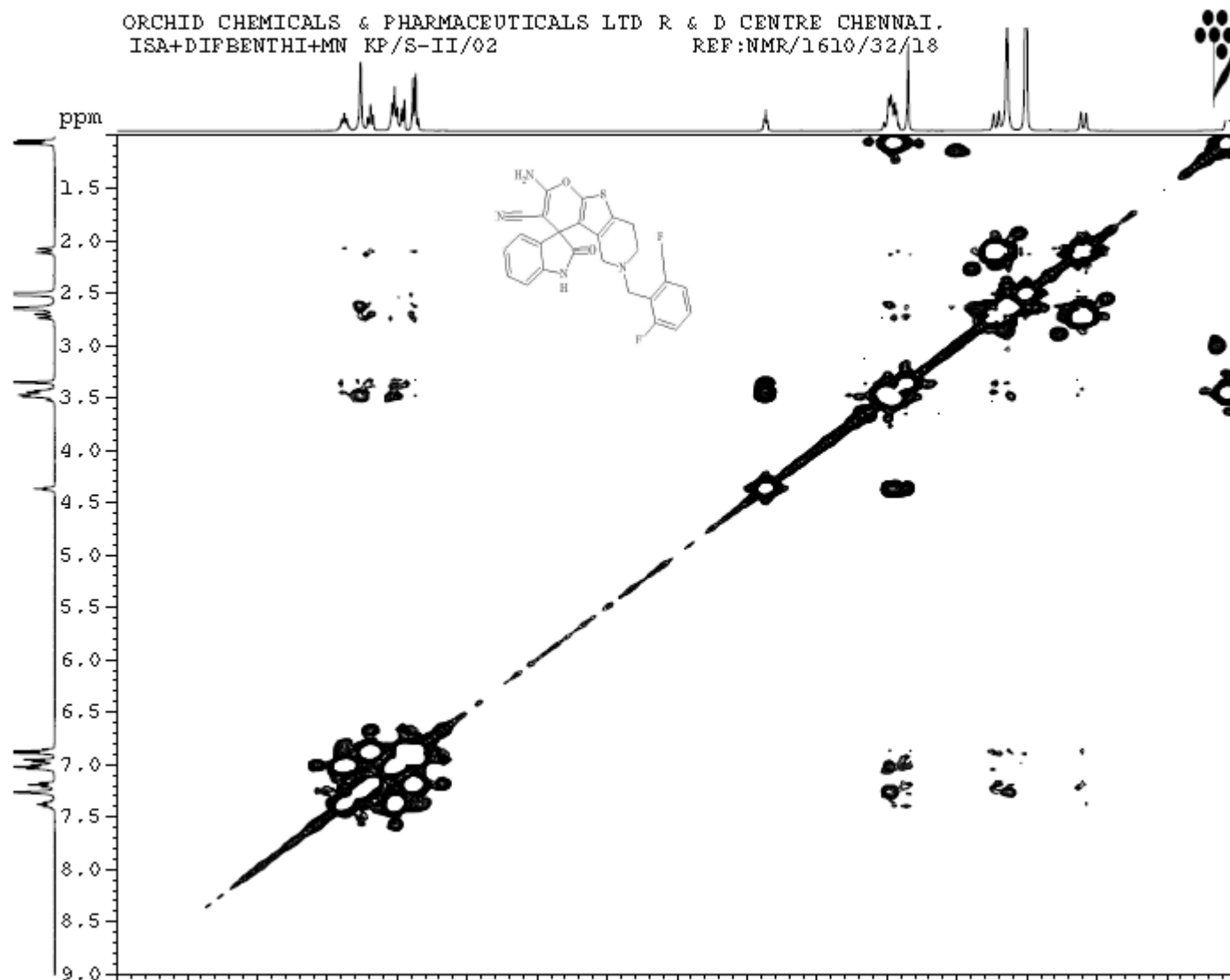
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SSB ()
LB 0.00
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PC 0.60

F1 - Processing parameters
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MC2 QF
SF 400.1300000
WDW QSINE
SSB ()
LB 0.00



COSY spectrum of compound 5t

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
ISA+DIFBENTHI+MN KP/S-II/02 REF:NMR/1610/32/18



Current Data Parameters:
NAME may11
EXPNO 18
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130501
Time 0.07
INSTRUM spect
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PULPROG cosygag
TD 2048
SOLVENT DMS
NS 2
DS 14
SWH 5995.204
FIDRES 2.927344
AQ 0.1708532
RG 181
AW 83.400
DE 6.50
TE 296.5
AQ 0.00000300
D1 2.00000000
d13 0.00000400
D16 0.00020000
IN0 0.00016680

===== CHANNEL f1 =====
NUC1 13
P0 11.00
P1 11.00
P11 3.00
SF01 400.1328000

===== GRADIENT CHANNEL =====
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CP21 20.00
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F1 - Acquisition parameters
MD0 1
TD 1024
SF01 400.1328
FIDRES 5.854691
SW 14.982
FNAME Q2

F2 - Processing parameters
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PC 0.60

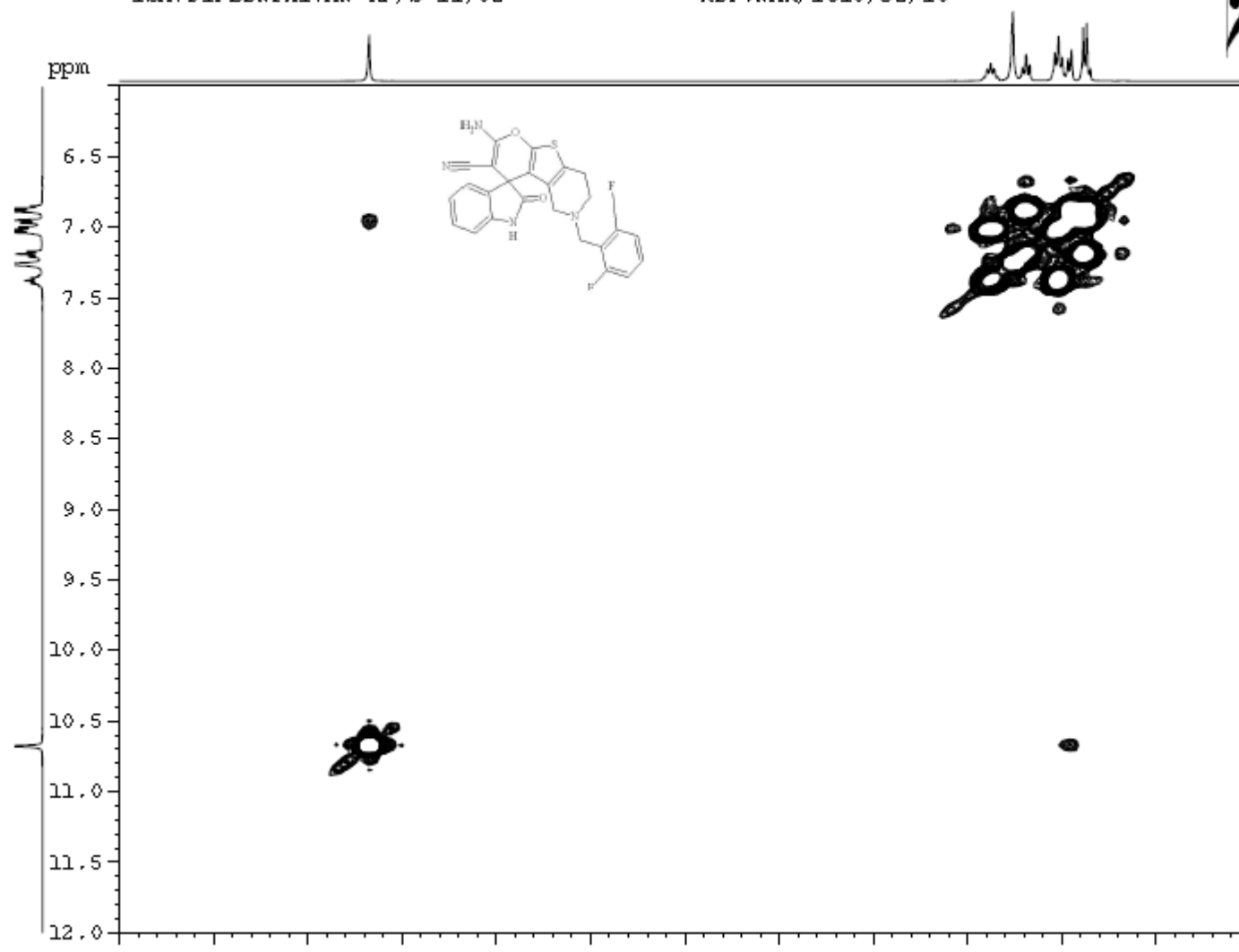
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LB 0.00

Expanded COSY spectrum of compound 5t

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
ISA+DIFBENTHI+MN KP/S-II/02 REF:NMR/1610/32/18



Current Data Parameters:
NAME may13
EXPNO 18
PROCNO 1



F2 - Acquisition Parameters
Date_ 20130501
Time 0.01
INSTRUM spect
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PULPROG cosygag
ID 2048
SOLVENT DMSO
NS 2
DS 14
SWH 5995.204
FIDRES 2.927344
AQ 0.1708532
RG 181
RW 83.400
DE 6.50
TE 294.2
D0 0.00000300
D1 2.00000000
d13 0.00000400
D16 0.00020000
IN0 0.00016680

===== CHANNEL f1 =====
NUC1 13
P0 11.05
P1 11.05
PL1 3.00
SFO1 400.1328000

===== GRADIENT CHANNEL =====
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CP21 20.00
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F1 - Acquisition parameters
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SW 14.983
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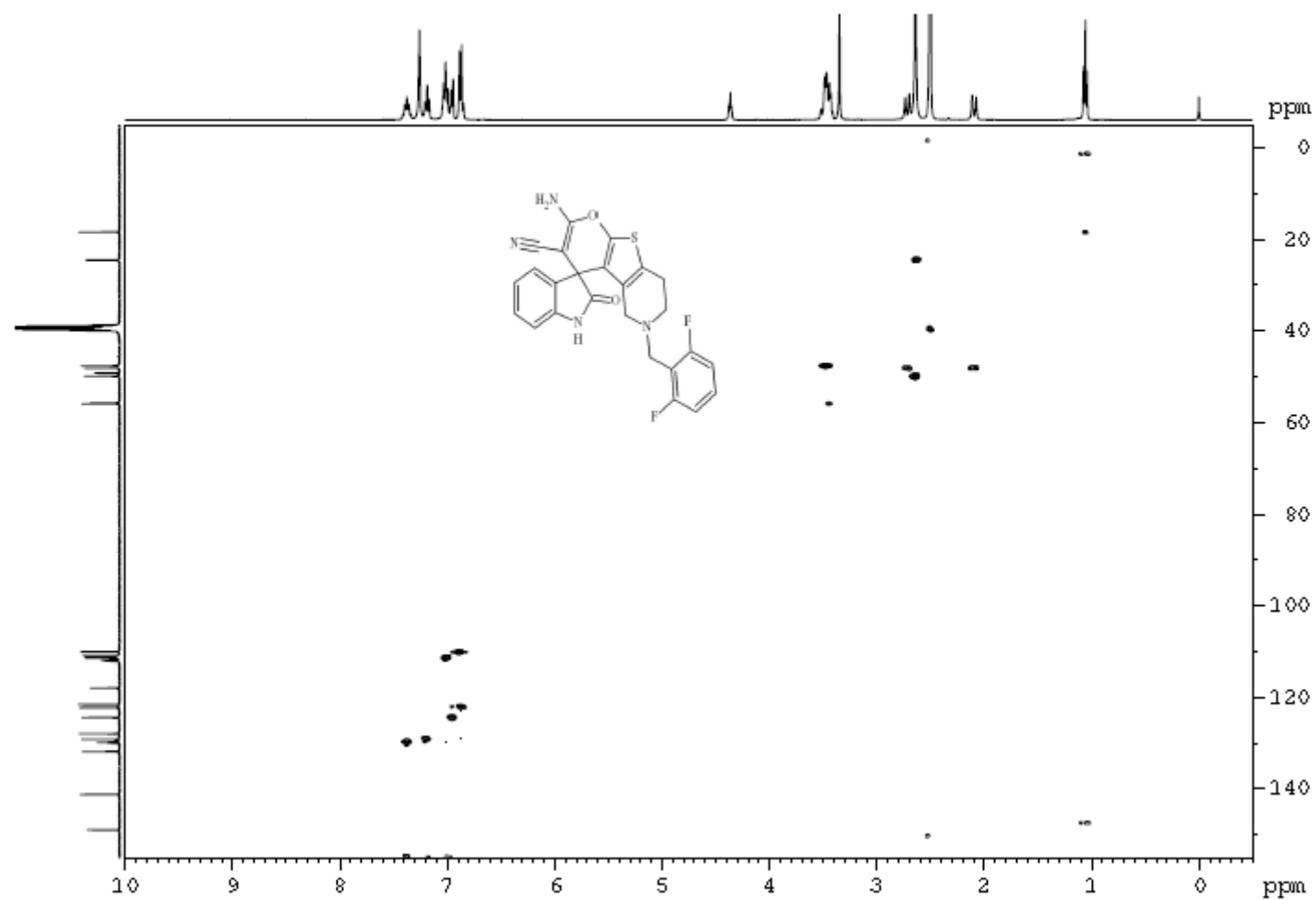
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GB 0
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F1 - Processing parameters
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SF 400.1300000
WDW QSINE
SSB 0
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Expanded COSY spectrum of compound 5t

10M+DIFFERENTIAL PM 05-11/06

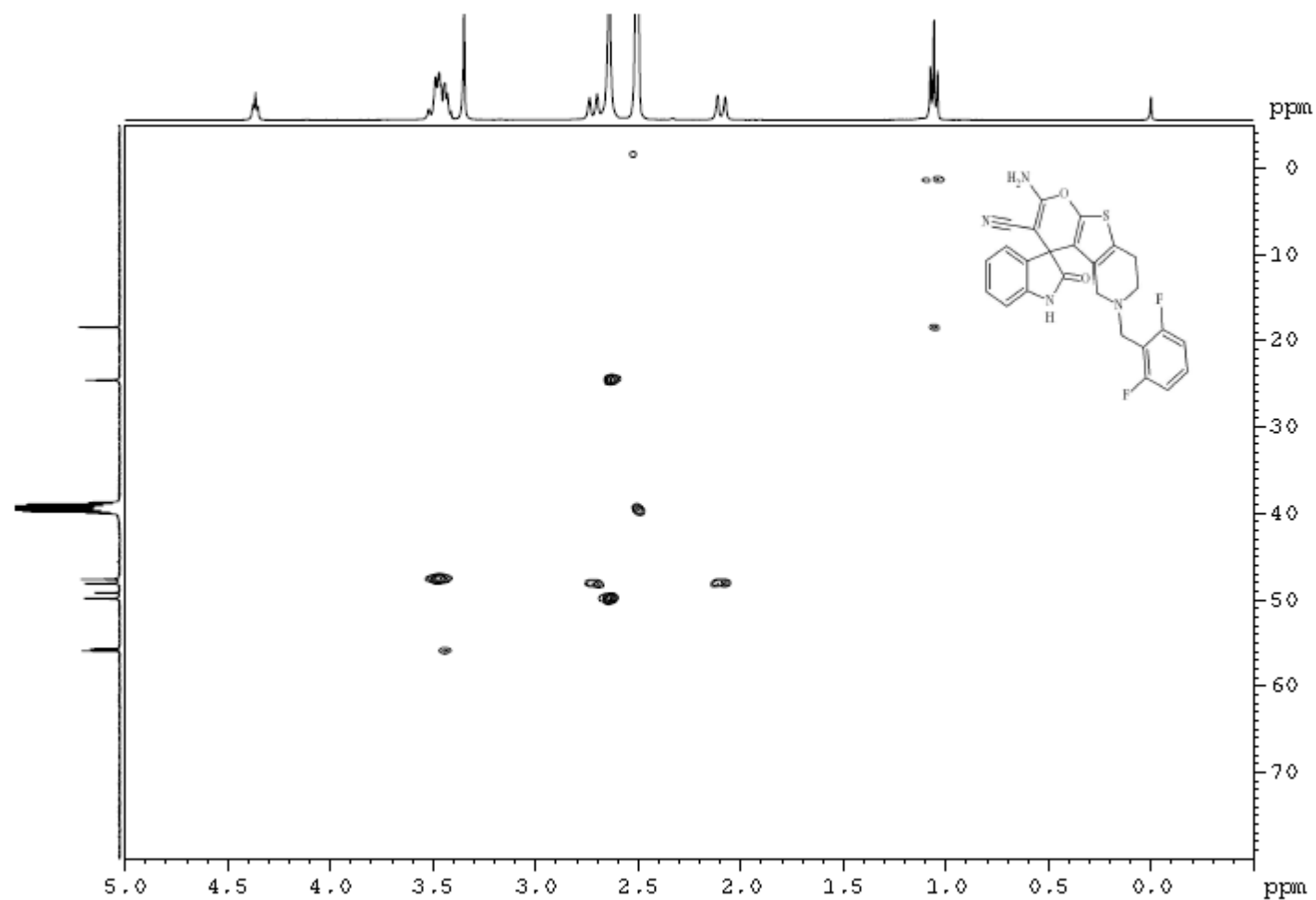
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HSQC spectrum of compound 5t

10M+DIFFERENTIAL F1 / 0-11/02

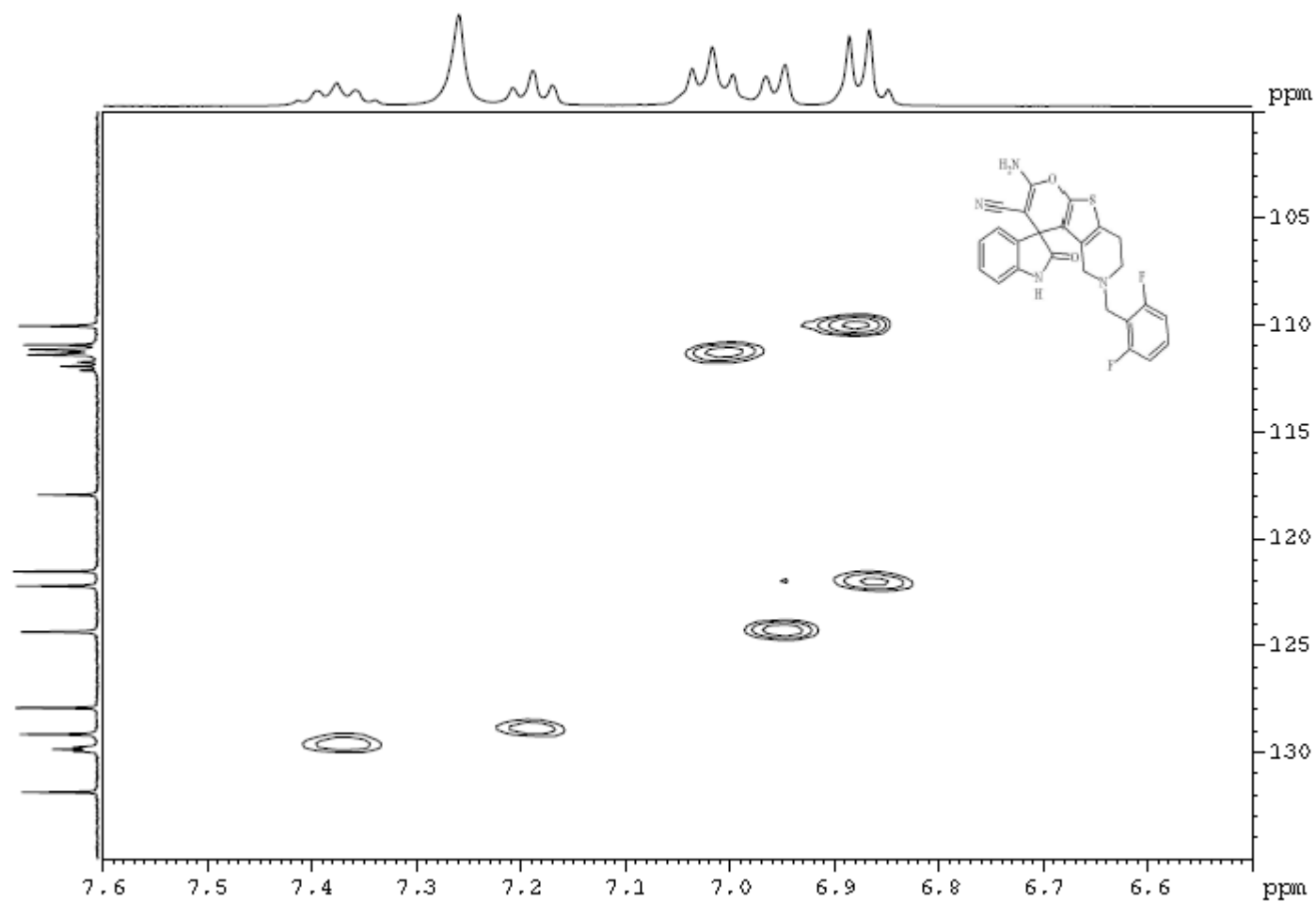
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Expanded HSQC spectrum of compound 5t

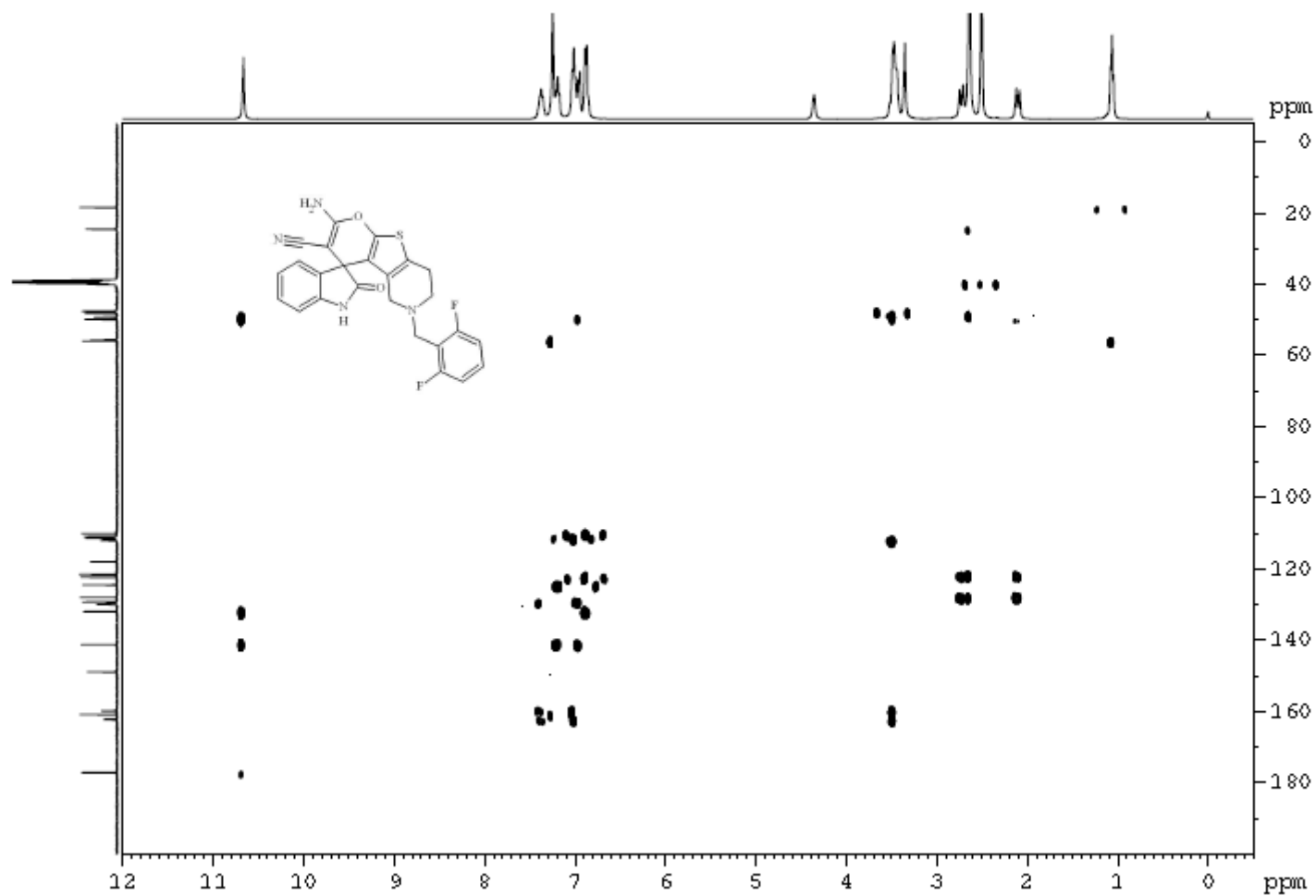
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REF : WIPK / 1010/06 / 19



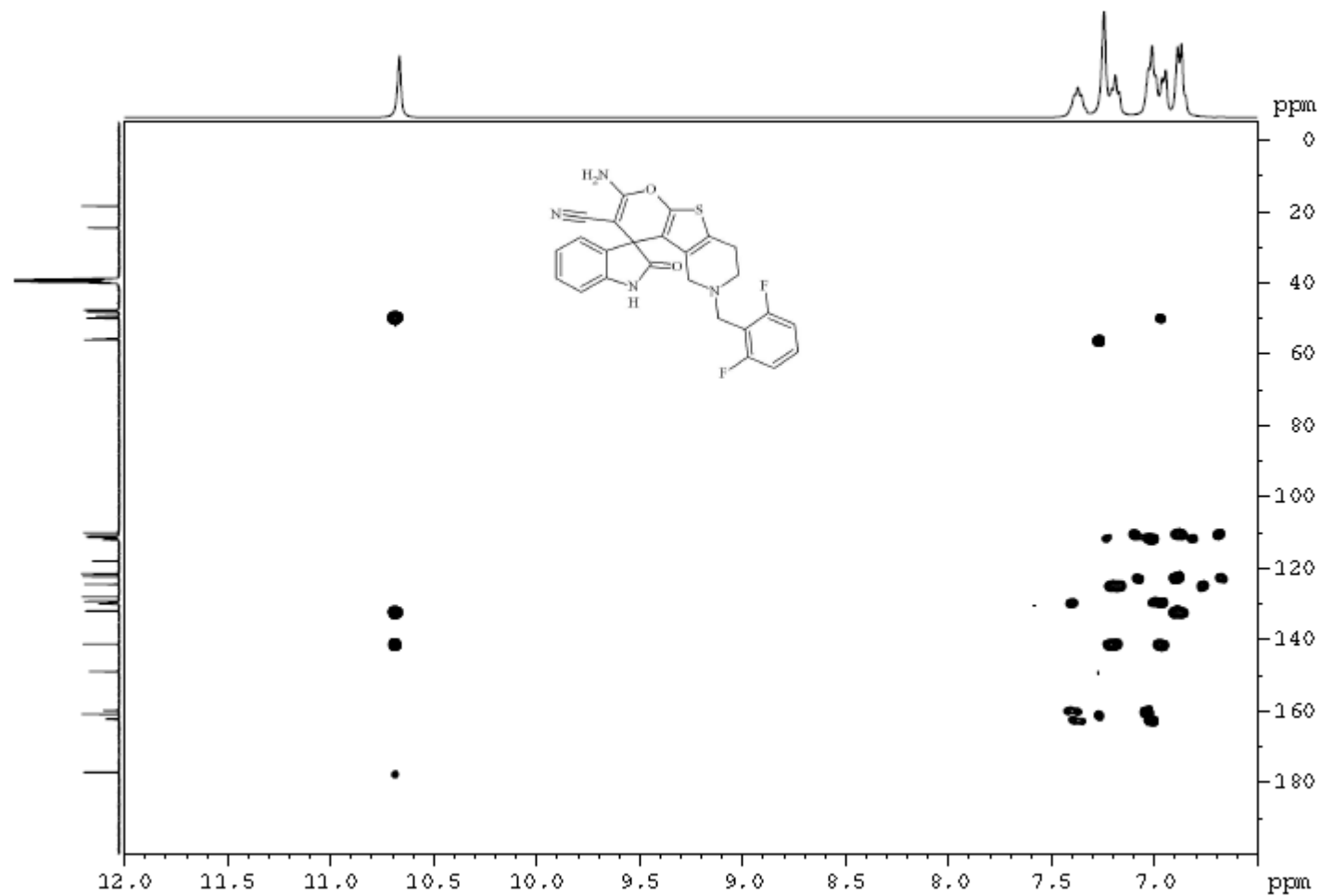
Expanded HSQC spectrum of compound 5t

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
ISA+DIFBENTHI+MN KP/S-II/02 REF:NMR/1610/32/20



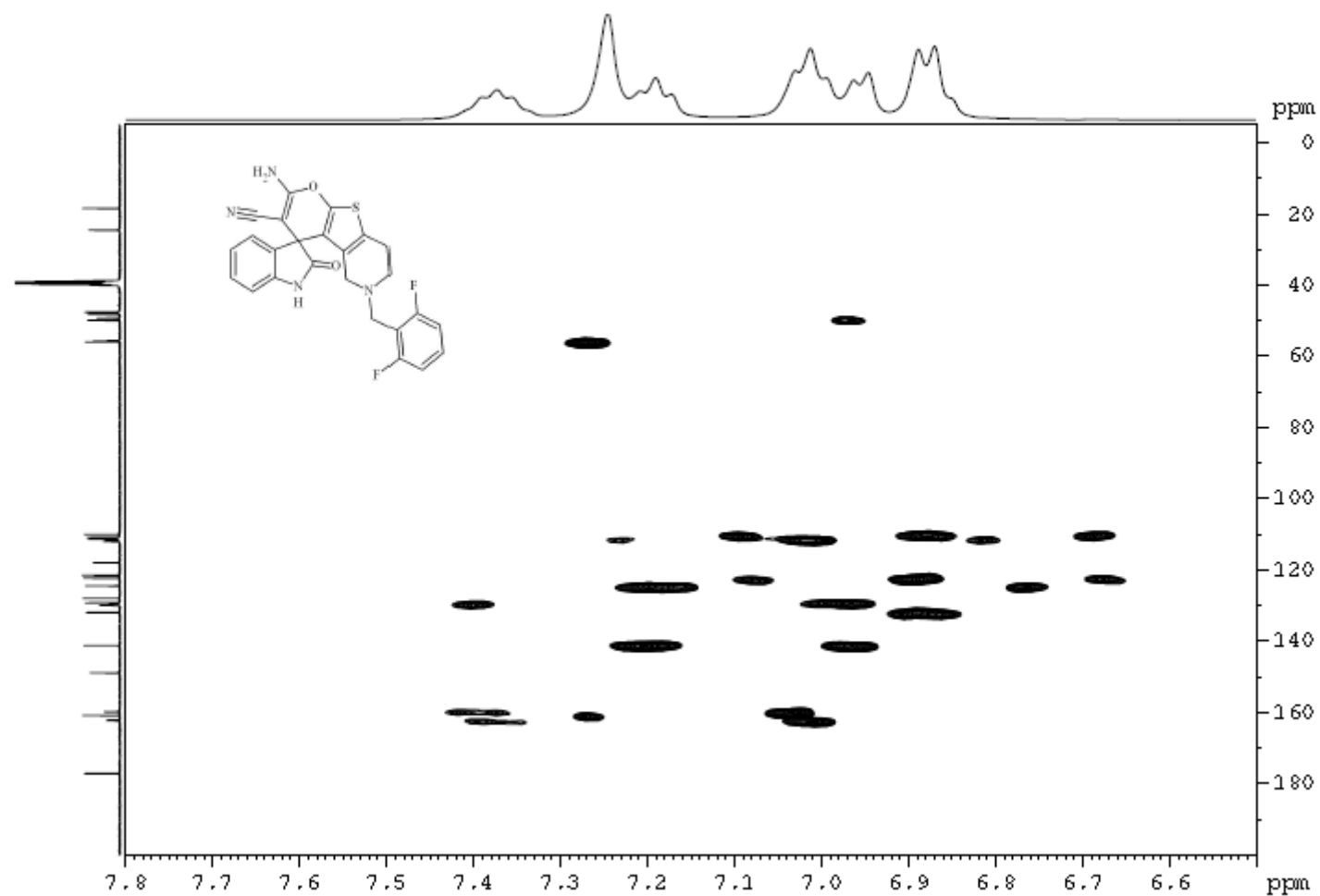
HMBC spectrum of compound 5t

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
ISA+DIFBENTHI+MN KP/S-II/02 REF:NMR/1610/32/20



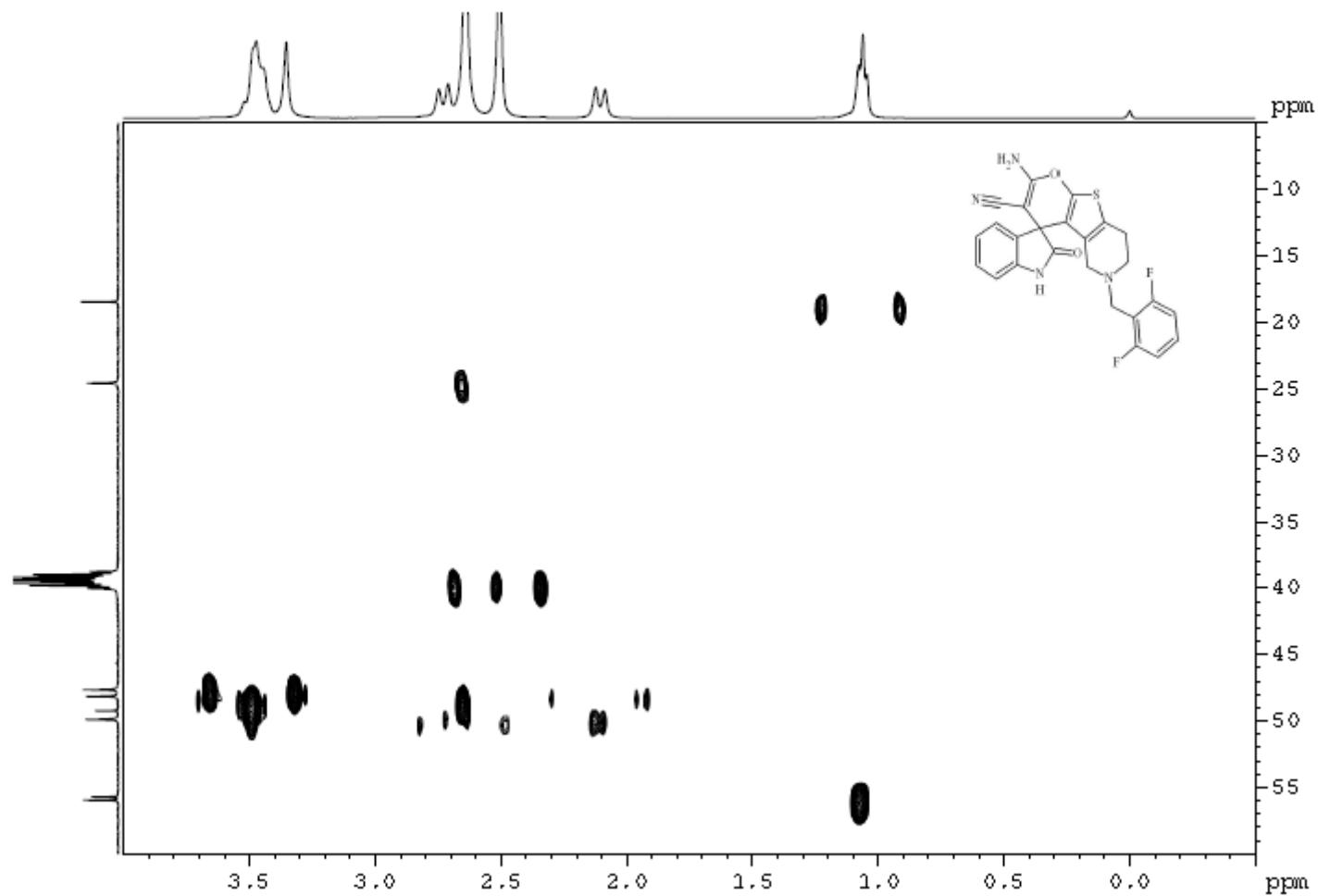
Expanded HMBC spectrum of compound 5t

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI,
ISA+DIFBENTHI+MN KP/S-II/02 REF:NMR/1610/32/20



Expanded HMBC spectrum of compound 5t

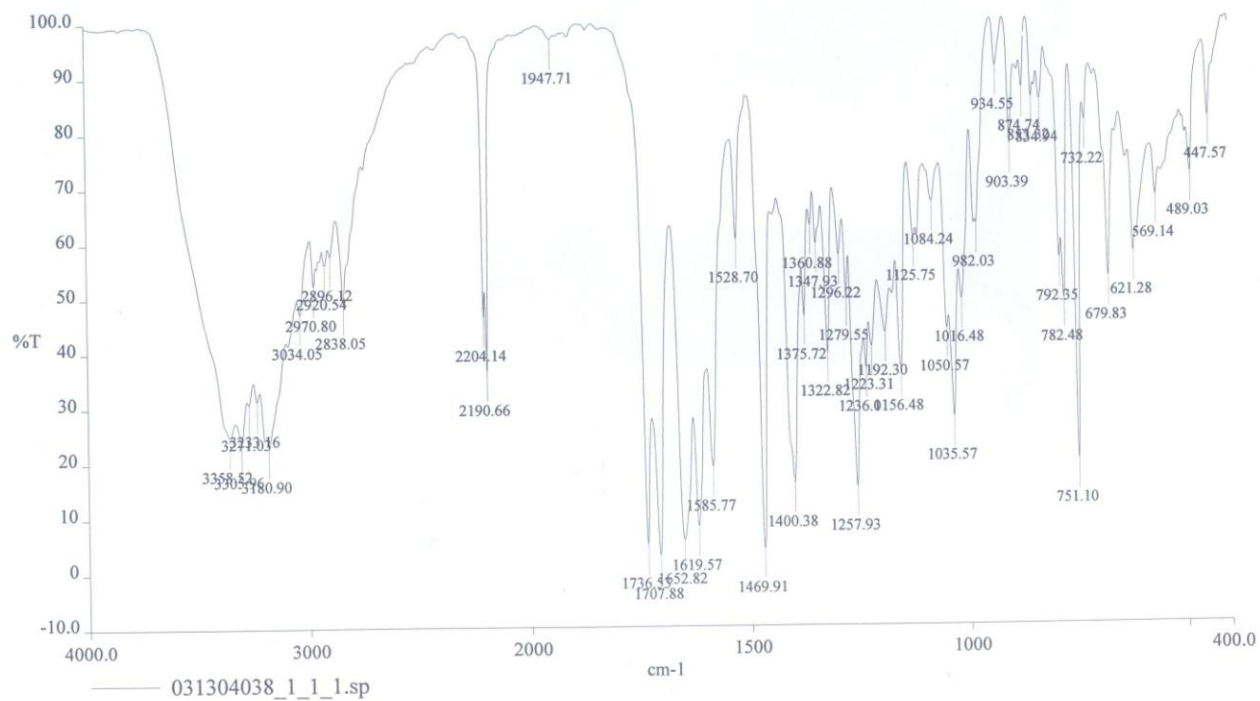
ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
ISA+DIFBENTHI+MN KP/S-11/02 REF:NMR/1610/32/20



Expanded HMBC spectrum of compound 5t

ORCHID CHEMICALS & PHARMACEUTICALS LIMITED

R&D CENTRE, CHENNAI



Spectrum Pathname: D:\DDRIVE\pel_data\spectra\spectra\2013\APR\031304038_1_1_1.sp

Date Created: Tuesday, April 23, 2013 11:33

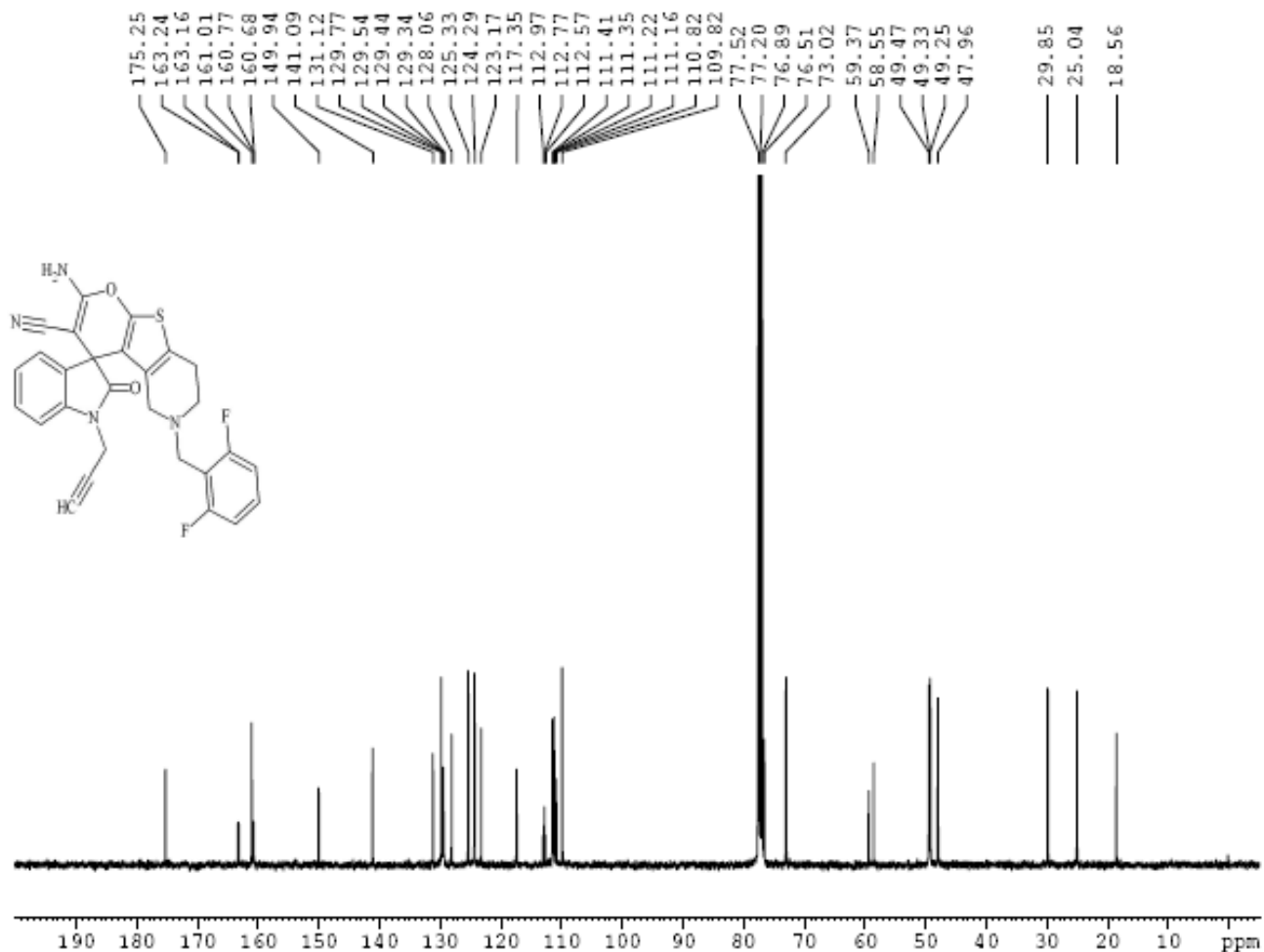
Description: ISA+DIFBENTHI+MN #KP/S-II/02

Analyst: *25/04/13*

Technique: KBr/Neat/Nujol

Reference: *2011559/107/12*

IR spectrum of 5t



Current Data Parameters
NAME FEB13
EXPNO 461
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130219
Time 15.59
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PROBHD 5 mm Multinuc
PULPROG zgpg
TD 32000
SOLVENT CDCl3
NS 1018
DS 0
SWH 25125.629 Hz
FIDRES 0.785176 Hz
AQ 0.6368500 sec
RG 23170.5
DW 19.900 usec
DE 6.50 usec
TE 297.4 K
D1 5.0000000 sec
d11 0.0300000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 ¹³C
P1 11.50 usec
PL1 2.00 dB
SFO1 100.6237964 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.19 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
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SSB 0
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GB 0
PC 3.00

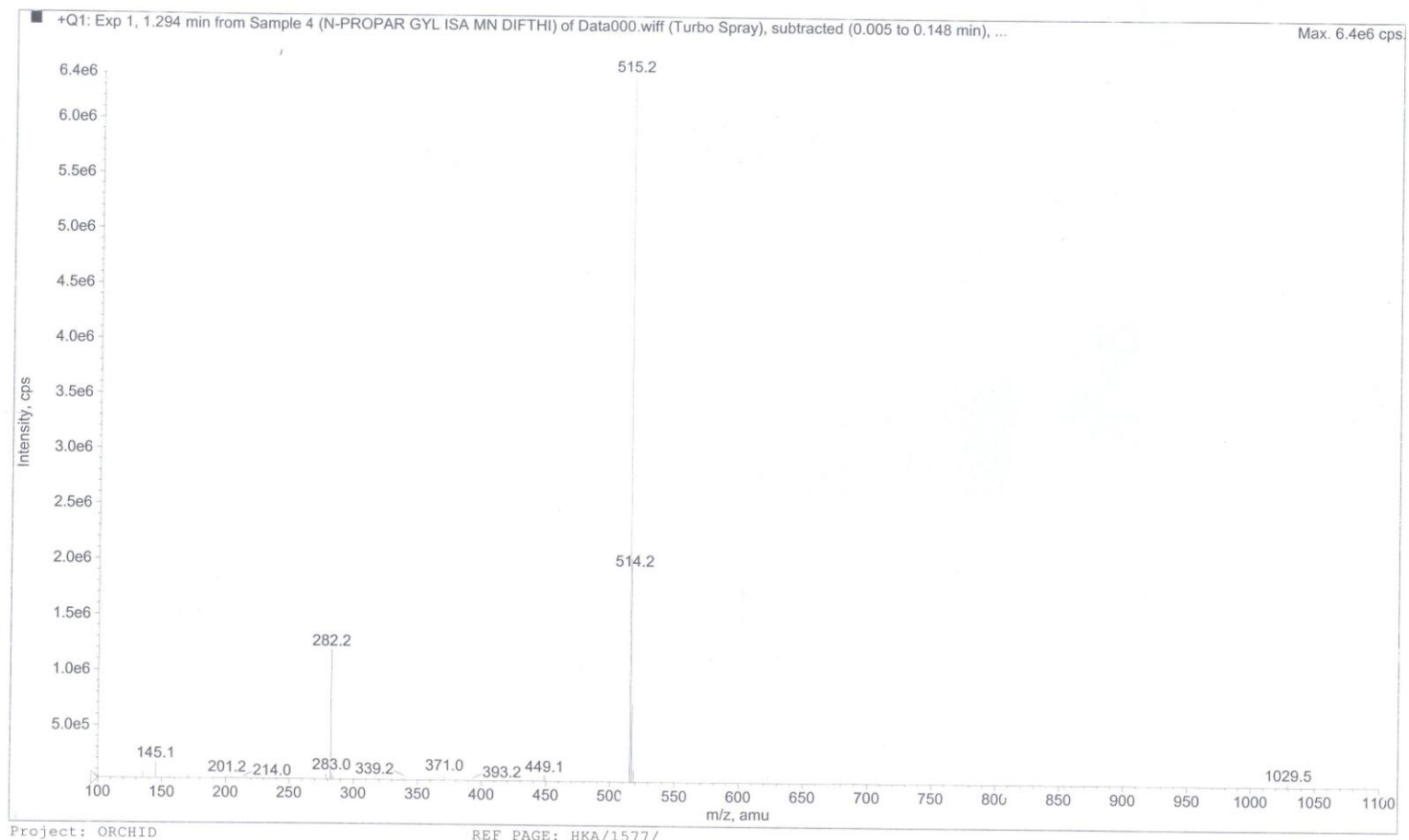
¹³C NMR spectrum of 5u

Acq. Date: Friday, May 24, 2013

ORCHID CHEMICALS AND PHARMACEUTICALS LTD., Printing Date: May 24, 2013

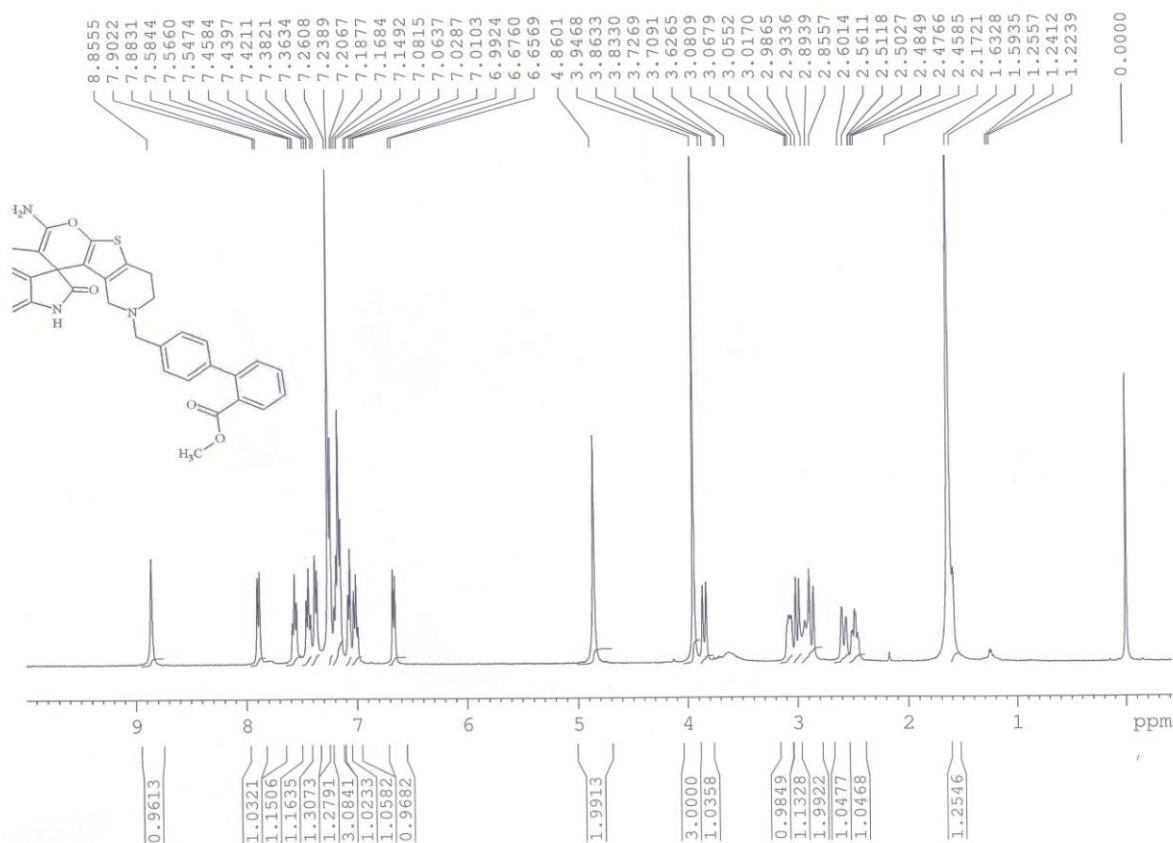
Acq. Time: 10:18

Sample Name: N-PROPAR GYL ISA MN DIFTHI Sample ID:



Mass spectrum of 5u

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
P.h.d., KP/ISA-MN-BMBTHI REF:NMR/1610/22/21



Current Data Parameters
NAME Apr13
EXPNO 477
PROCNO 1

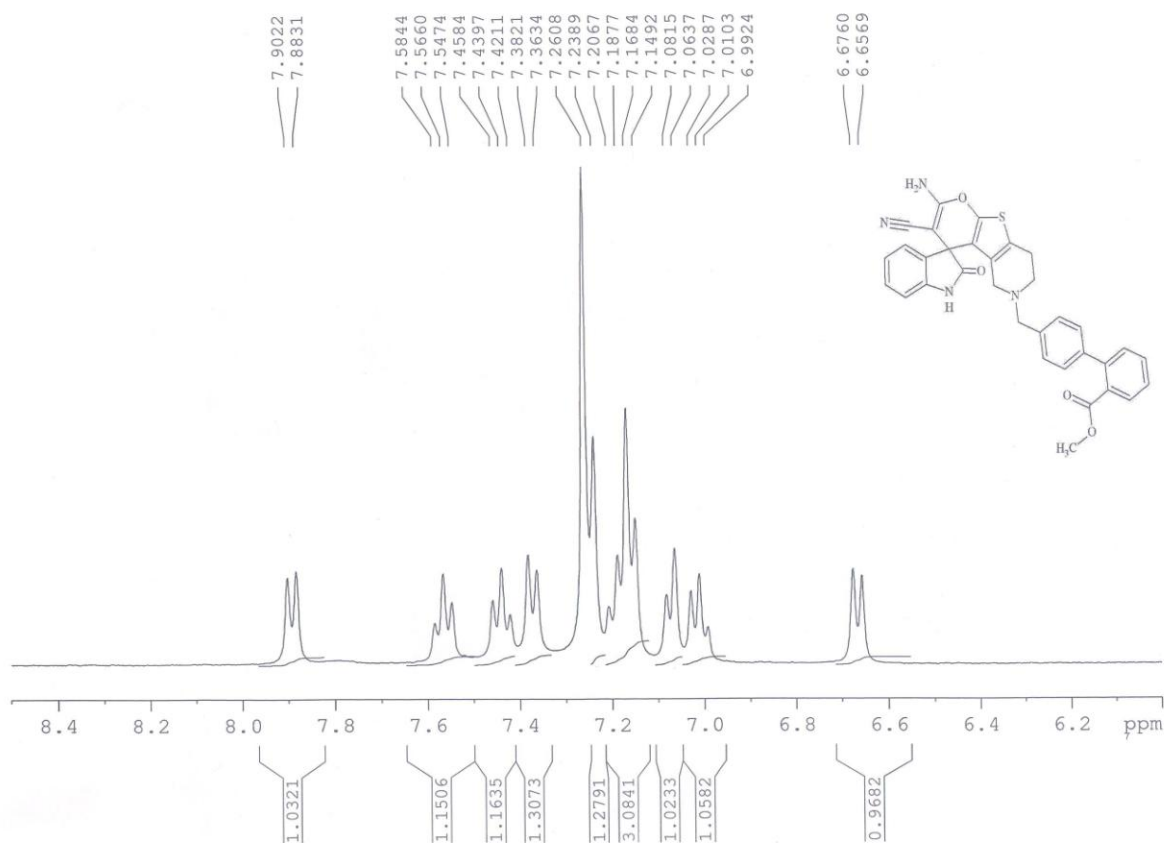
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 45.3
DW 69.600 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1

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NUC1 1H
P1 7.05 usec
PL1 3.00 dB
SFO1 400.1332010 MI

F2 - Processing parameters
SI 16384
SF 400.1300116 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

¹H NMR spectrum of 5w

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
P.h.d., KP/ISA-MN-BMBTHI REF:NMR/1610/22/21



Current Data Parameters
NAME Apr13
EXPNO 477
PROCNO 1

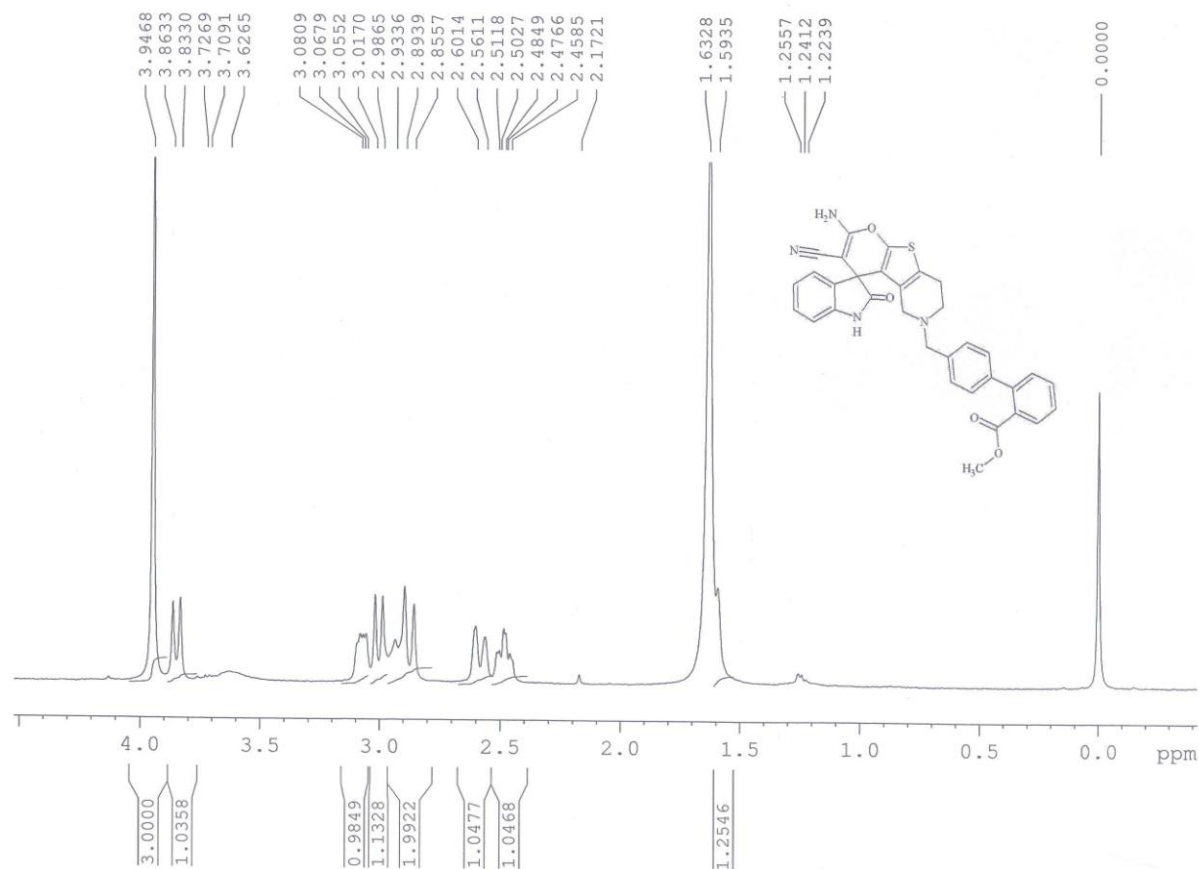
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INSTRUM spect
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 45.3
DW 69.600 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 :
NUC1 1H
P1 7.05 usec
PL1 3.00 dB
SFO1 400.1332010 MI

F2 - Processing parameters
SI 16384
SF 400.1300116 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

Expanded ¹H NMR spectrum of 5w

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
P.h.d., KP/ISA-MN-BMBTHI REF:NMR/1610/22/21



Current Data Parameters
NAME Apr13
EXPNO 477
PROCNO 1

F2 - Acquisition Parameter
Date_ 20130423
Time 20.29
INSTRUM spect
PROBHD 5 mm Multinuc
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 45.3
DW 69.600 usec
DE 6.50 usec
TE 296.6 K
D1 1.0000000 sec
TD0 1

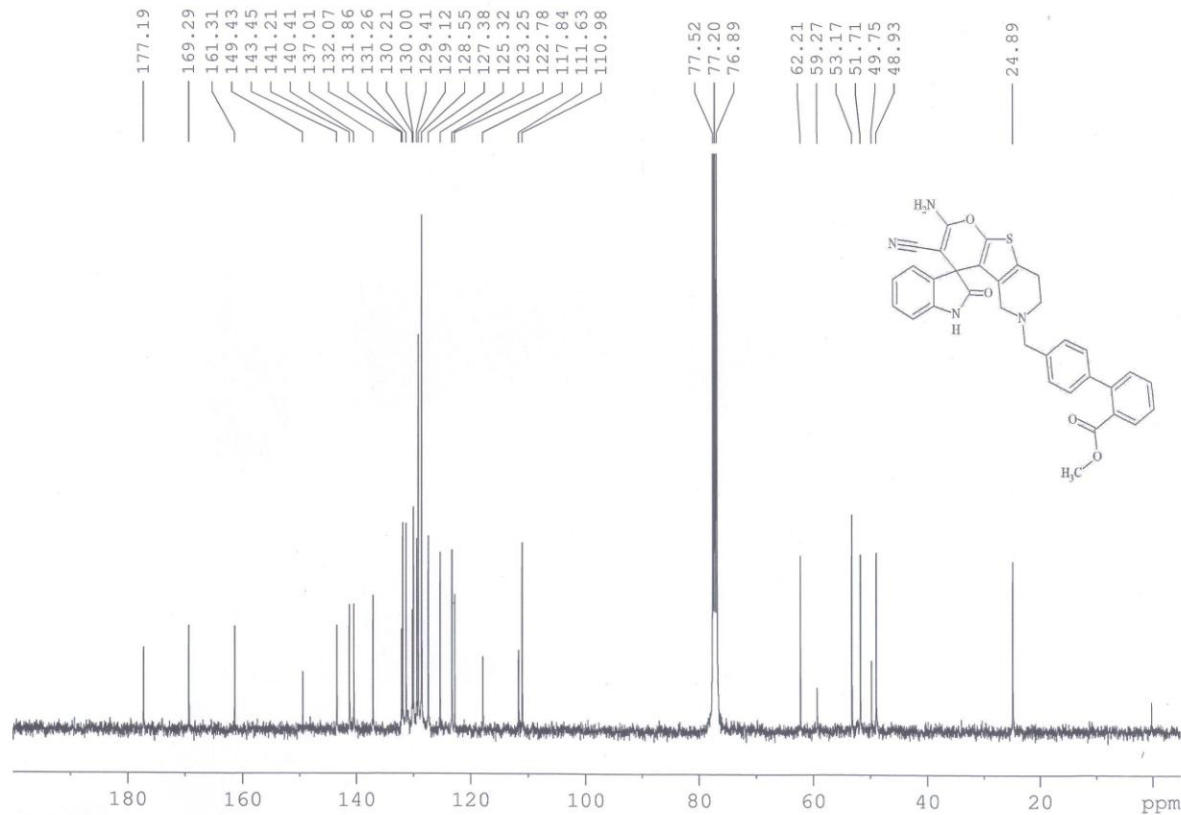
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NUC1 1H
P1 7.05 usec
PL1 3.00 dB
SFO1 400.1332010 MI

F2 - Processing parameters
SI 16384
SF 400.1300116 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

Expanded ¹H NMR spectrum of 5w

ORCHID CHEMICALS & PHARMACEUTICALS LTD R & D CENTRE CHENNAI.
P.h.d., KP/ISA-MN-BMBTHI

REF:NMR/1610/22/22



Current Data Parameters
NAME Apr13
EXPNO 478
PROCNO 1

F2 - Acquisition Parameters
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TD 32000
SOLVENT CDCl3
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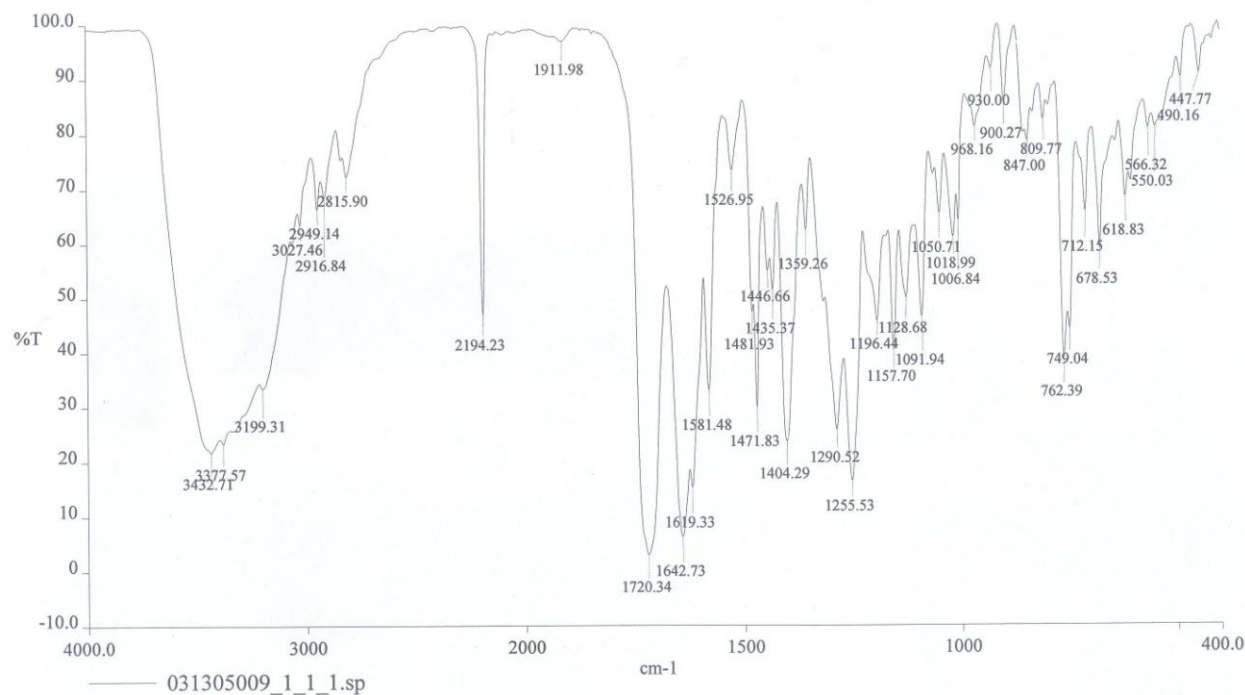
===== CHANNEL f1 ==
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P1 11.50 usec
PL1 2.00 dB
SFO1 100.6237964 MHz

===== CHANNEL f2 ==
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.19 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127545 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 3.00

¹³C NMR spectrum of 5w

ORCHID CHEMICALS & PHARMACEUTICALS LIMITED
R&D CENTRE, CHENNAI



Spectrum Pathname: D:\DDRIVE\pel_data\spectra\spectra\2013\MAY\031305009_1_1_1.sp

Date Created: Thursday, May 02, 2013 2:53

Description: ISATIN MN+BMB THIENE

Analyst: *ST*

Technique: KBr/Neat/Nujol

Reference: *IR/1558/111/9*

IR spectrum of 5w

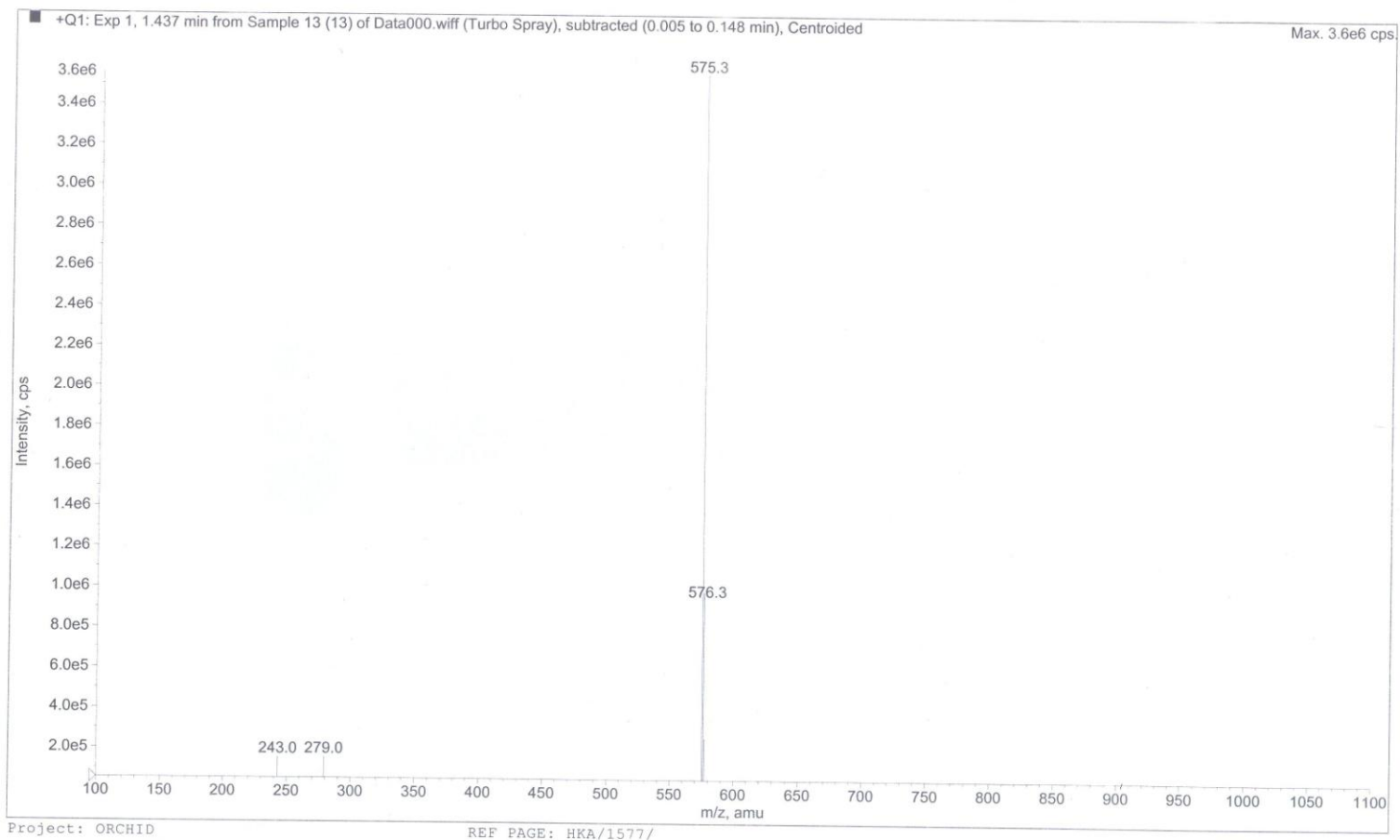
Acq. Date: Friday, May 24, 2013

ORCHID CHEMICALS AND PHARMACEUTICALS LTD., Printing Date: May 24, 2013

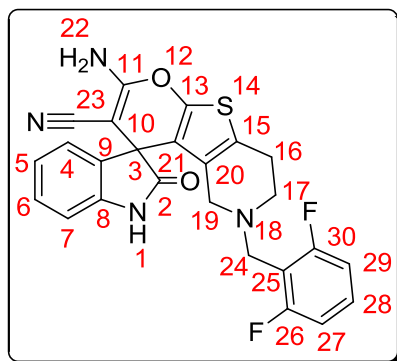
Acq. Time: 10:45

Sample Name: 13

Sample ID:



Mass spectrum of 5w



Numbering of compound **5t** for NMR assignments

^1H NMR characteristics of compound **5t** recorded in $\text{DMSO-}d_6$

Chemical Shift δ (ppm)	Multiplicity	Number of Hydrogen(s)	Coupling constant $^2J_{\text{HH}}$ & $^3J_{\text{HH}}$ (Hz)	Assignment
2.08 & 2.69	Two doublets	2	14.8	24- CH_2
2.64	Multiplet	4	-	16- & 17- CH_2
3.44	Singlet	2	-	19- CH_2
6.85-6.89	Multiplet	2	-	5- & 7- CH
6.95	Doublet	1	7.2	4- CH
6.99	Triplet	2	7.2	27- & 29- CH
7.17	Triplet	1	7.6	6- CH
7.26	Broad signal (D_2O exchangeable)	2	-	22- NH_2
7.34-7.41	Multiplet	1	-	28- CH
10.67	Broad signal (D_2O exchangeable)	1	-	1- NH

Chemical shift at δ 2.50 ppm and at δ 3.35 ppm are due to $\text{DMSO-}d_6$ and solvent water respectively.
Additional signals at δ 1.04, δ 3.41 and δ 4.35 ppm are due to ethanol used for recrystallization. The numbering is given for NMR assignments only.

¹³C NMR characteristics of compound **5t** recorded in DMSO-*d*₆

Group/Carbon	Chemical shift δ (ppm)
C-17 & C-19	24.6 & 49.9
C-24	47.6
C-16	48.1
C-3	49.2
C-10	55.7
C-7	110.1
C-21	110.9
*C-27 & C-29	111.2 & 111.4
*C-25	111.7, 111.9 & 112.1
C-23	117.9
C-15	121.6
C-5	122.2
C-4	124.4
C-20	127.9
C-6	129.2
*C-28	129.7, 129.8 & 129.9
C-9	131.9
C-8	141.2
C-13	148.8
*C-26 & C-30	159.7, 159.8, 162.1 & 162.2
C-11	160.8
2-C=O	177.1

Chemical shift around δ 39.5 ppm is due to the solvent, DMSO-*d*₆.

*Splitting of signals is due to ¹³C-¹⁹F coupling.

Crystal data, data collection and refinement parameters of compound **5t**

Empirical formula	C ₅₂ H ₃₈ F ₄ N ₈ O ₆ S ₂
Formula weight	1011.02
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	$a = 12.8051(5)$ Å; $b = 12.8767(5)$ Å; $c = 15.6983(6)$ Å; $\alpha = 66.206(2)^\circ$; $\beta = 88.780(2)^\circ$; $\gamma = 81.531(2)^\circ$
Volume	2340.53 (16) Å ³
Z, calculated density	2, 1.435 mg m ⁻³
Absorption coefficient	0.192 mm ⁻¹
$F(000)$	1044
Crystal size	0.25 x 0.22 x 0.12 mm
θ range for data collection	1.42 -25.00°
Limiting indices	$-15 \leq h \leq 15$, $-15 \leq k \leq 15$, $-18 \leq l \leq 18$
Reflections collected /unique	26180/7948 [$R(\text{int}) = 0.0367$]
Completeness to $\theta = 25.00$	96.4 %
Absorption correction	Multi-scan
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	7948/3/664
Goodness-of-fit on F^2	1.349
Final R indices [I^2 sigma(I)]	$R1 = 0.0628$, $wR2 = 0.1751$
R indices (all data)	$R1 = 0.0977$, $wR2 = 0.2157$
Largest diff. peak and hole	0.971 and -0.486 e.Å ⁻³
CCDC	936,526