Electronic Supplementary Material (ESI) for RSC Advances.

$Cu(OTf)_2$ catalyzed three component strategy for the synthesis of thienopyridine containing spirooxindoles and their cytotoxic evaluation

K. Parthasarathy,[a,b] C. Praveen,[a] P. Senthil Kumar,[c] C. Balachandran,[d] P. T. Perumal[a,]*

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[c]*Department of Bioinformatics, Orchid Chemicals & Pharmaceuticals Ltd., Research & Development Centre, Shozanganallur, Chennai 600 119, India.*

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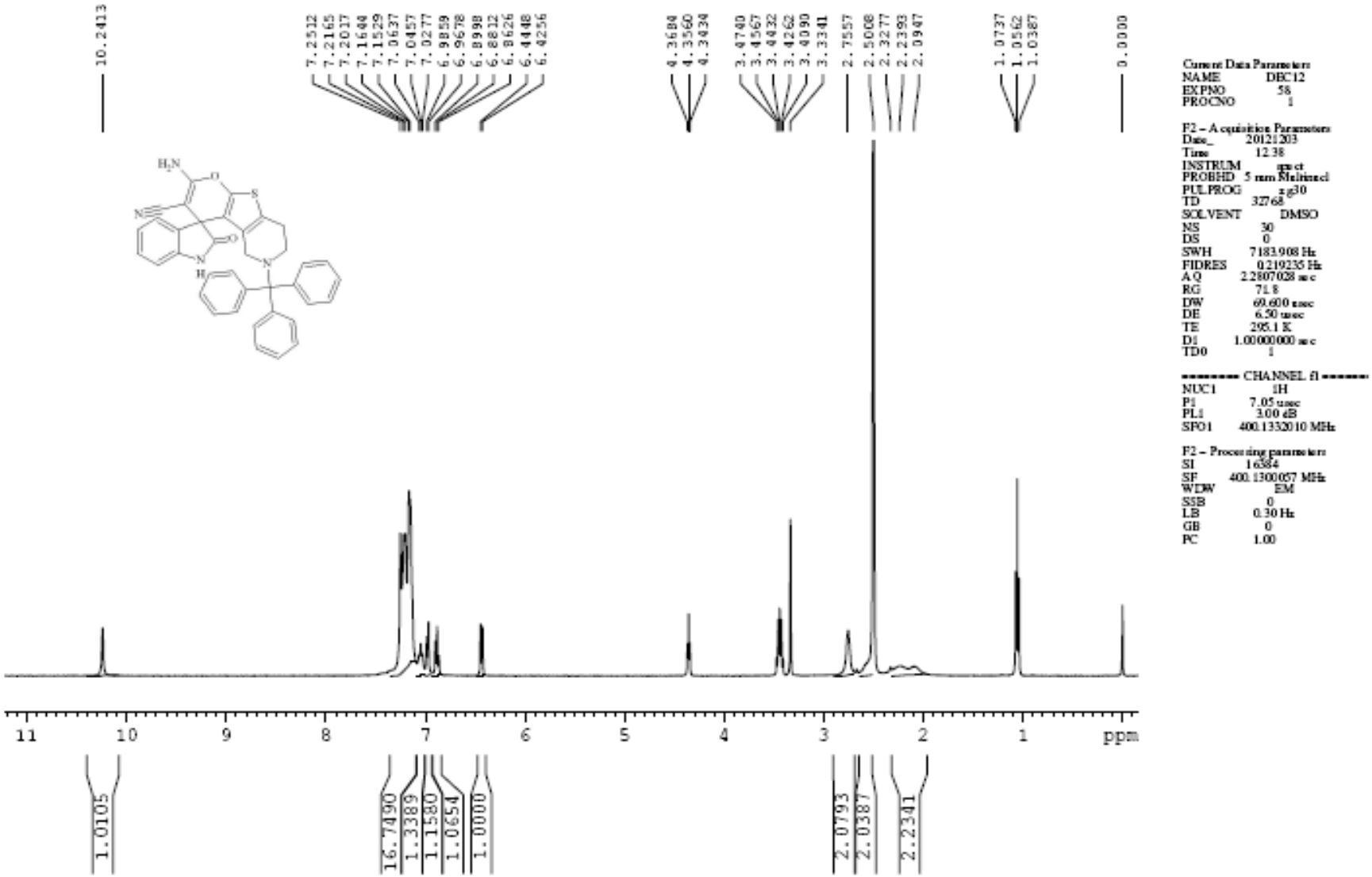
IR, ^{1}H NMR, ^{13}C NMR and mass spectra of selected compounds



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PROCNO 1

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RG 71.8
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DE 6.50 usec
TE 295.1 K
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PL1 3.00 dB
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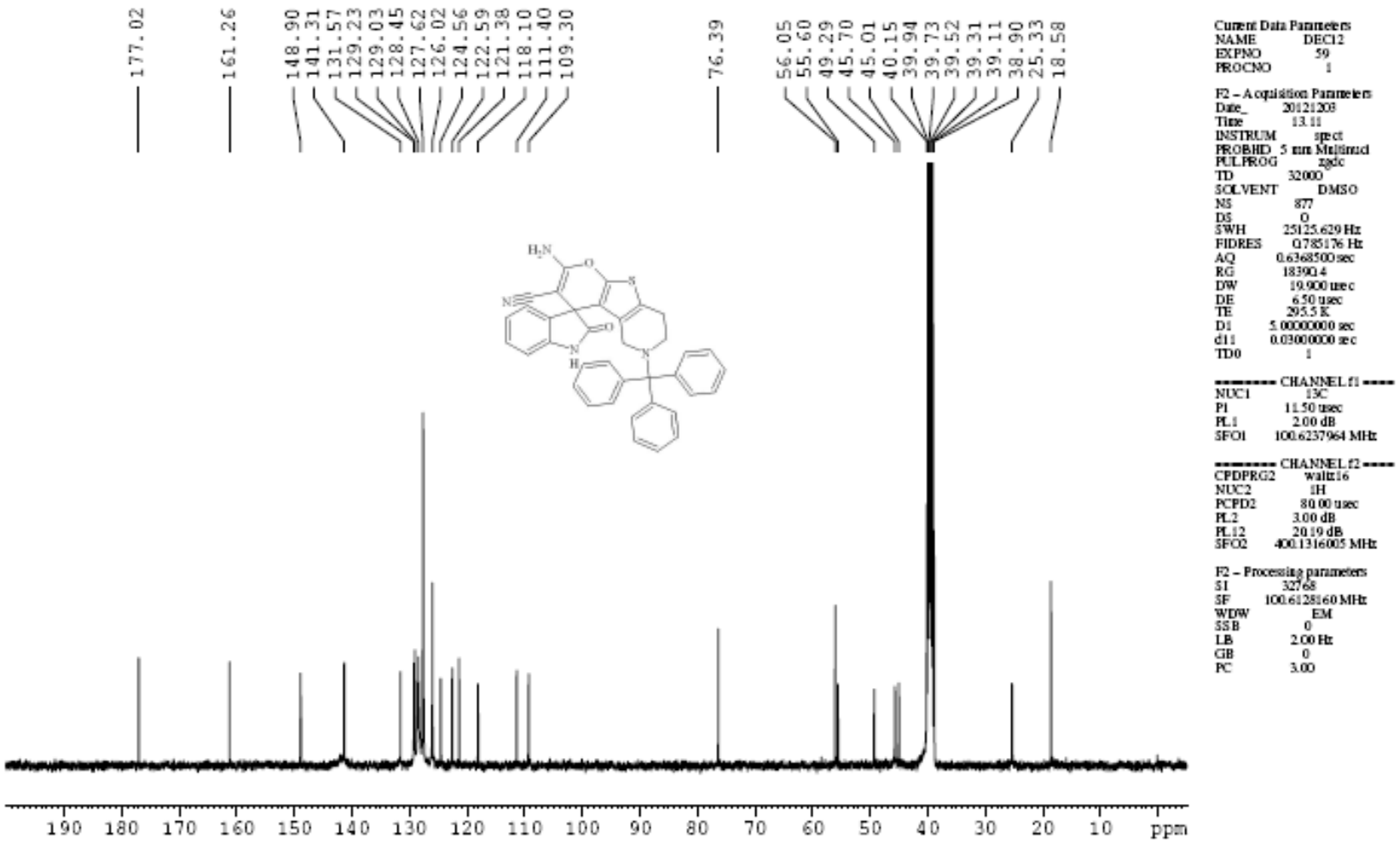
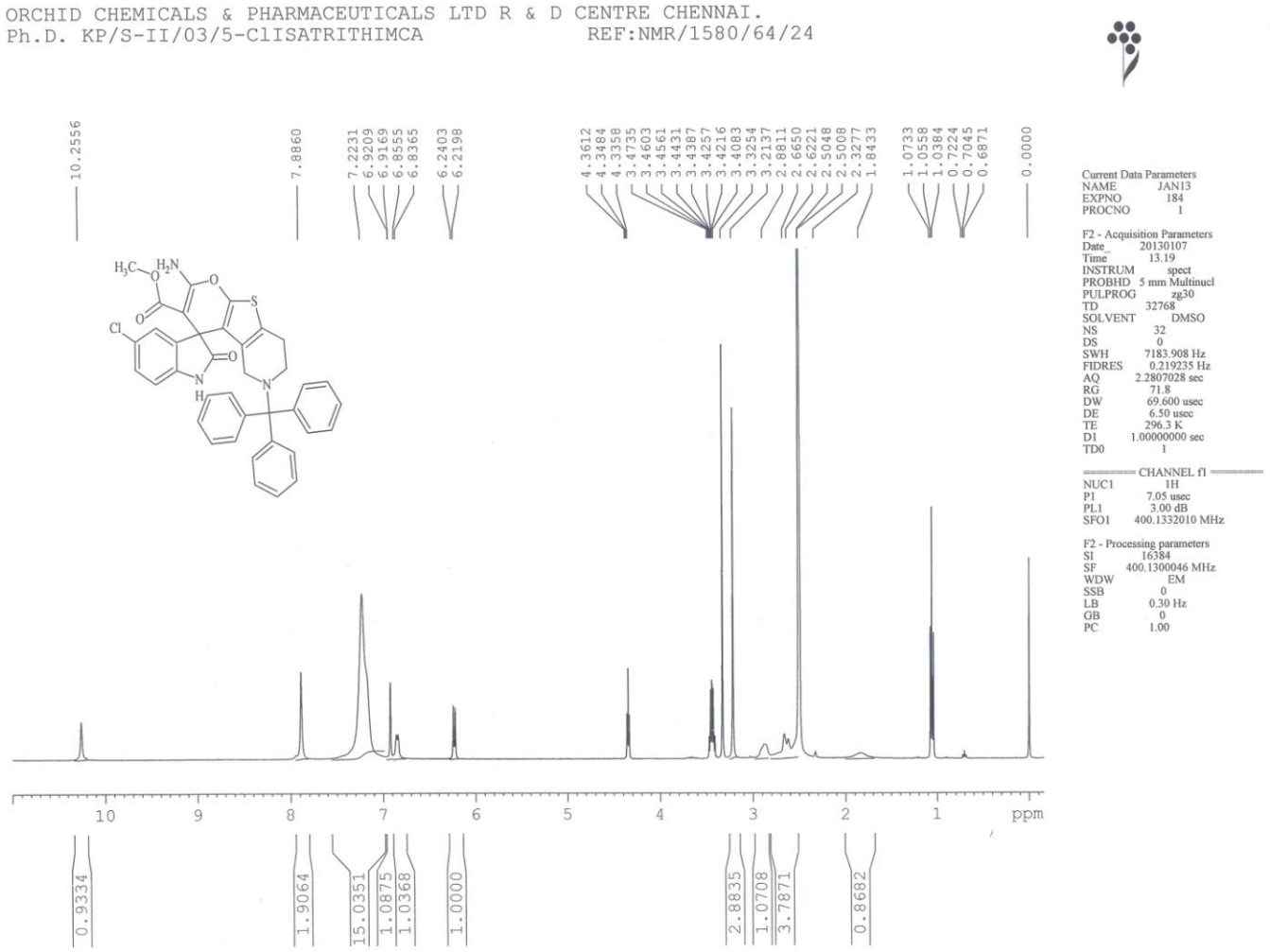
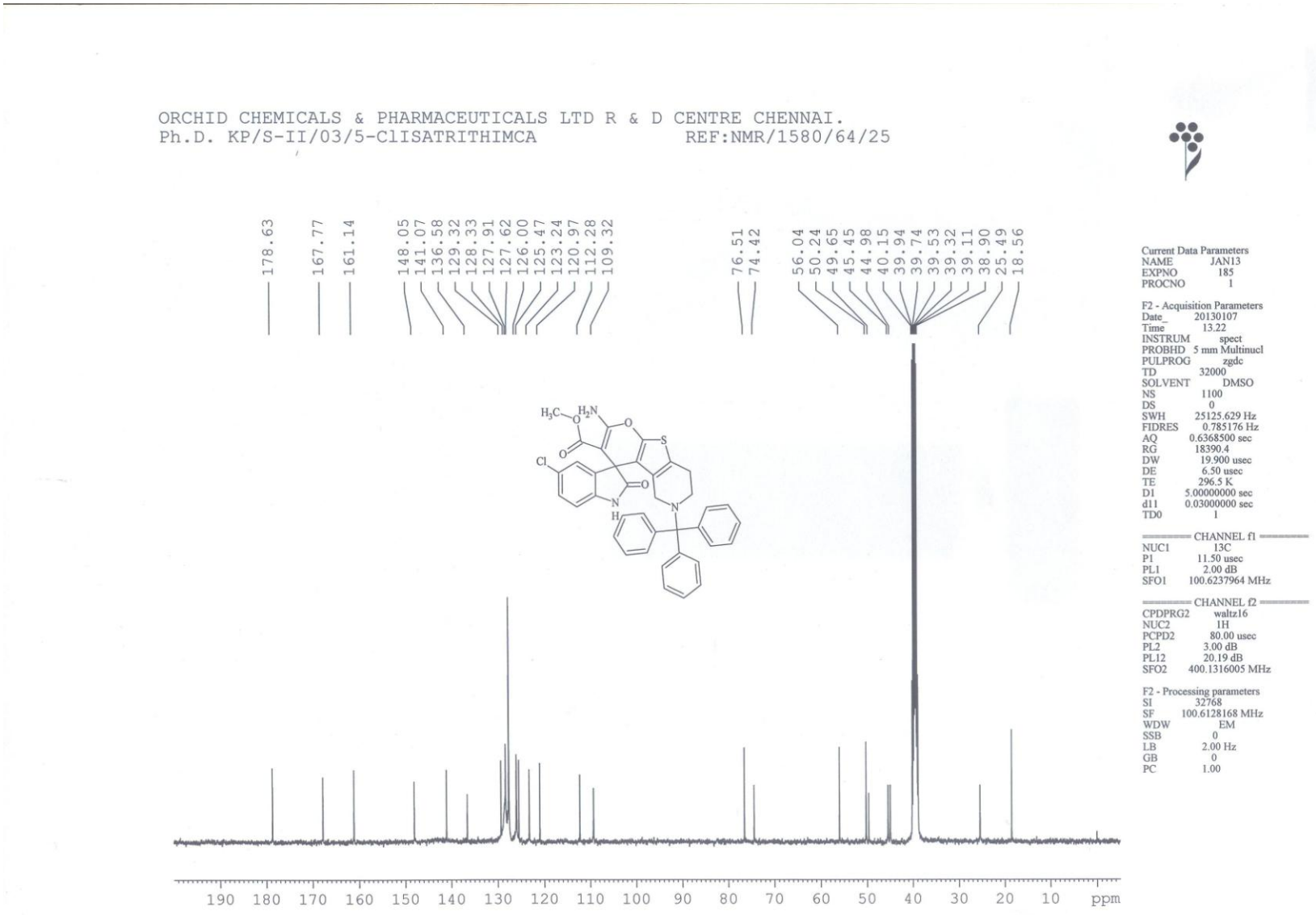
^{1}H NMR spectrum of 5a



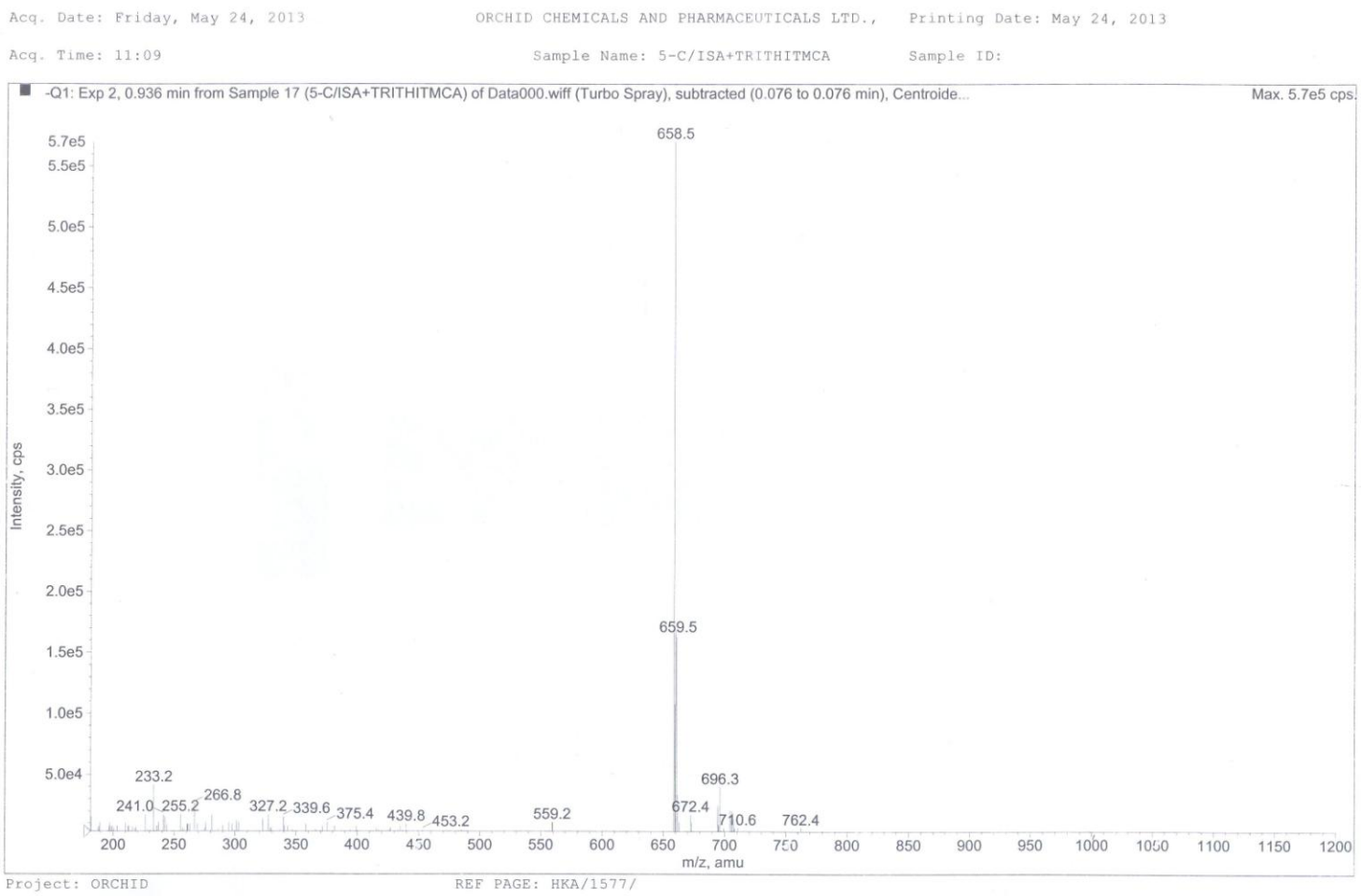
^{13}C NMR spectrum of 5a



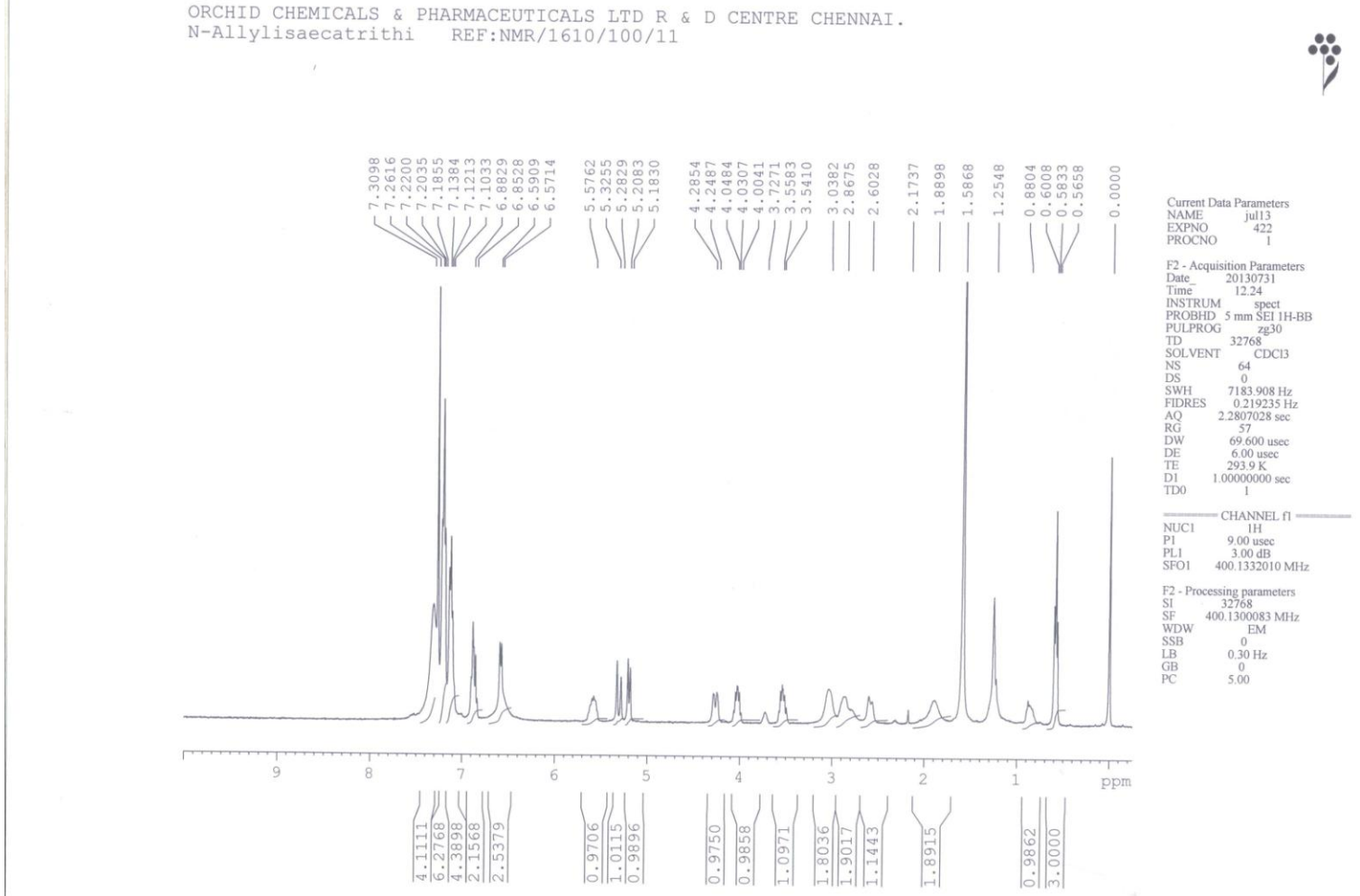
^{1}H NMR spectrum of 5c



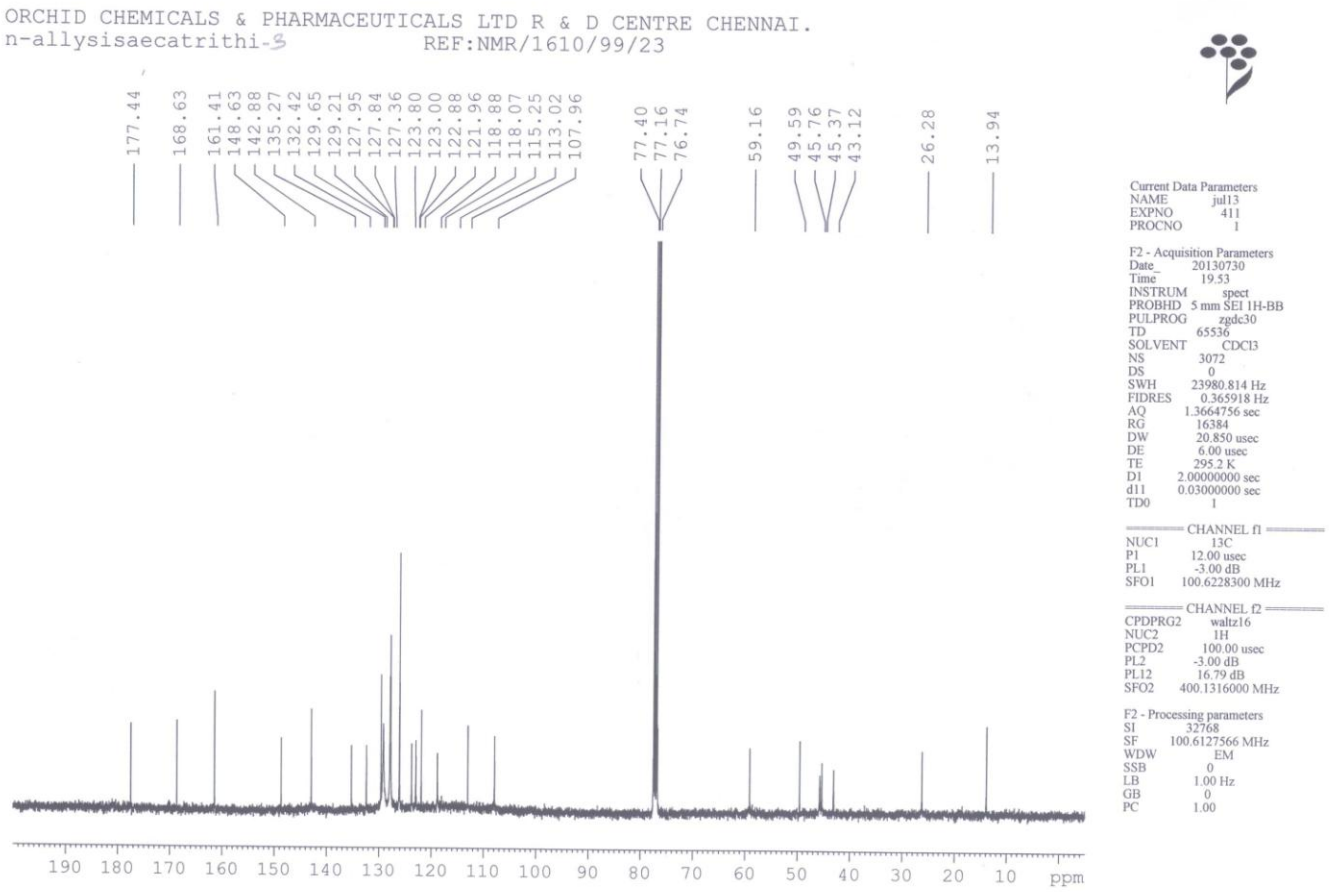
^{13}C NMR spectrum of 5c



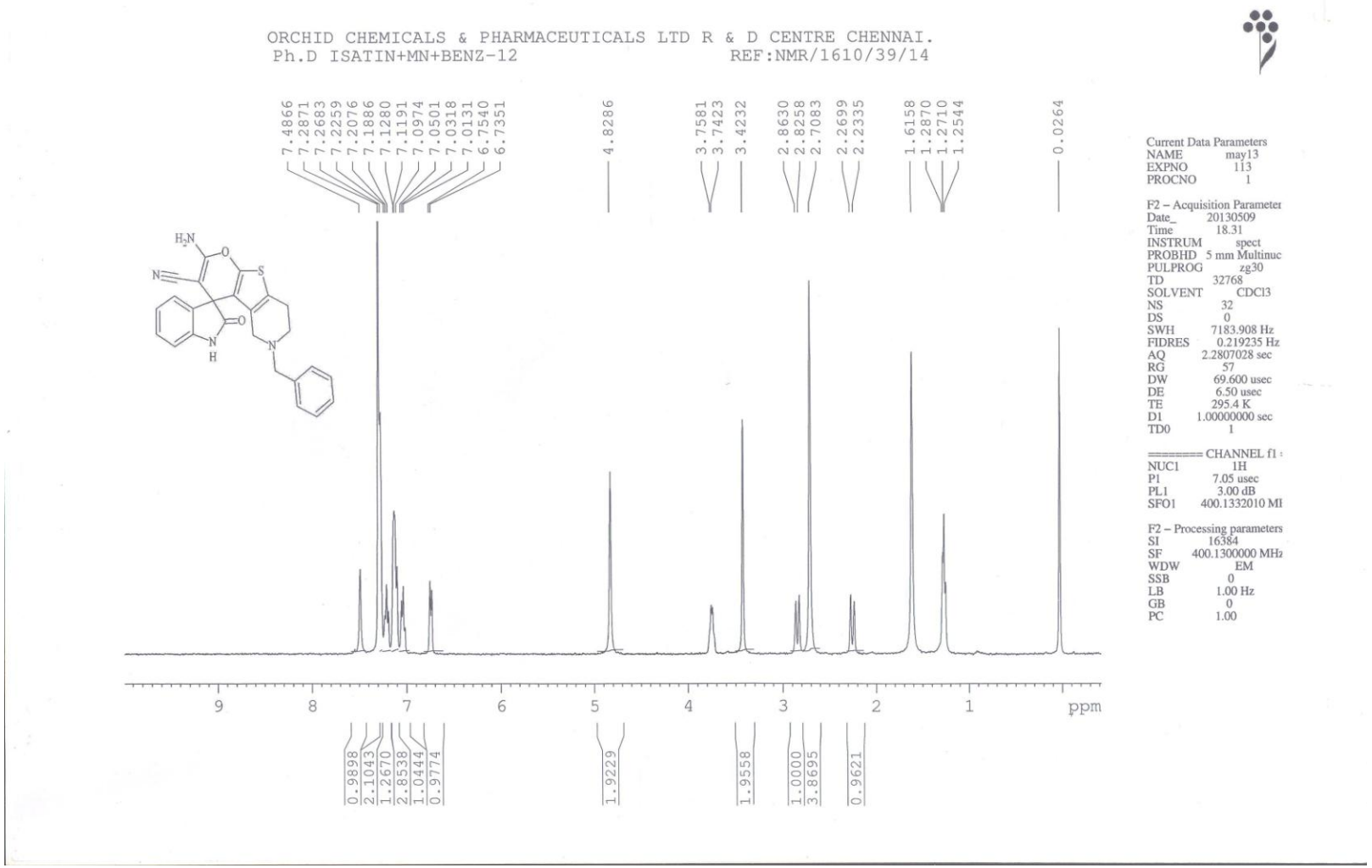
Mass spectrum of 5c



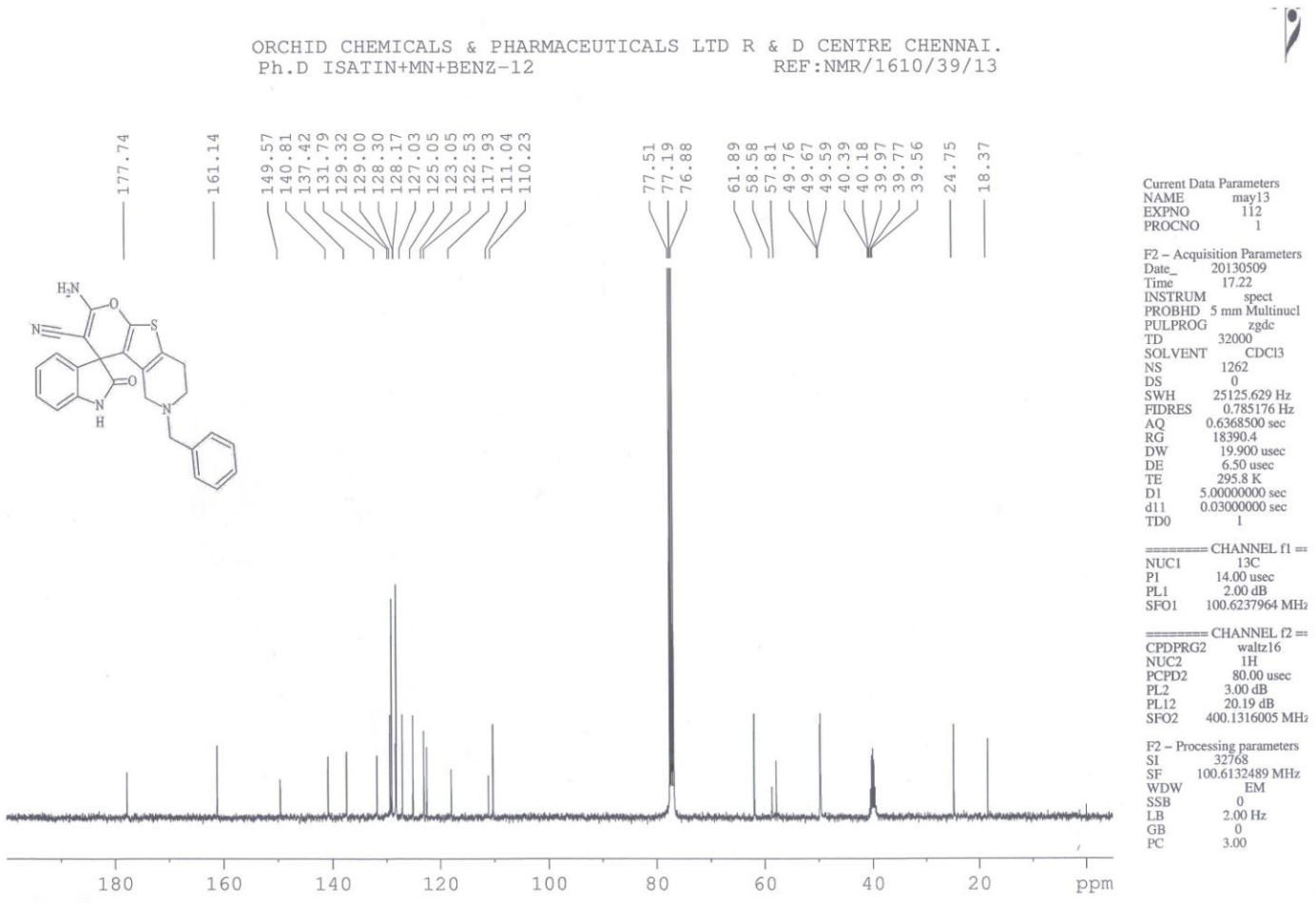
^{1}H NMR spectrum of 5i



^{13}C NMR spectrum of 5i



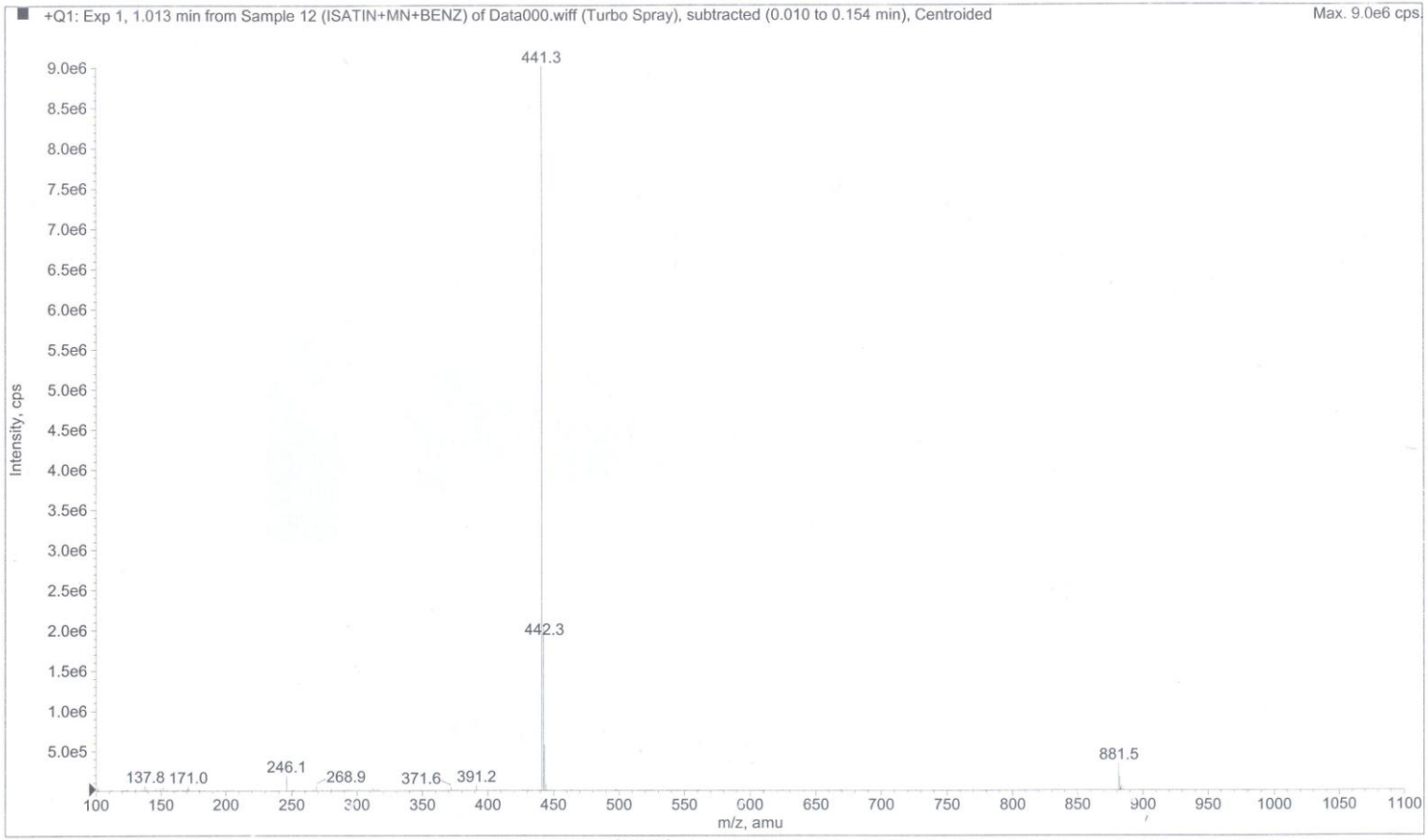
^{1}H NMR spectrum of 5j



^{13}C NMR spectrum of 5j

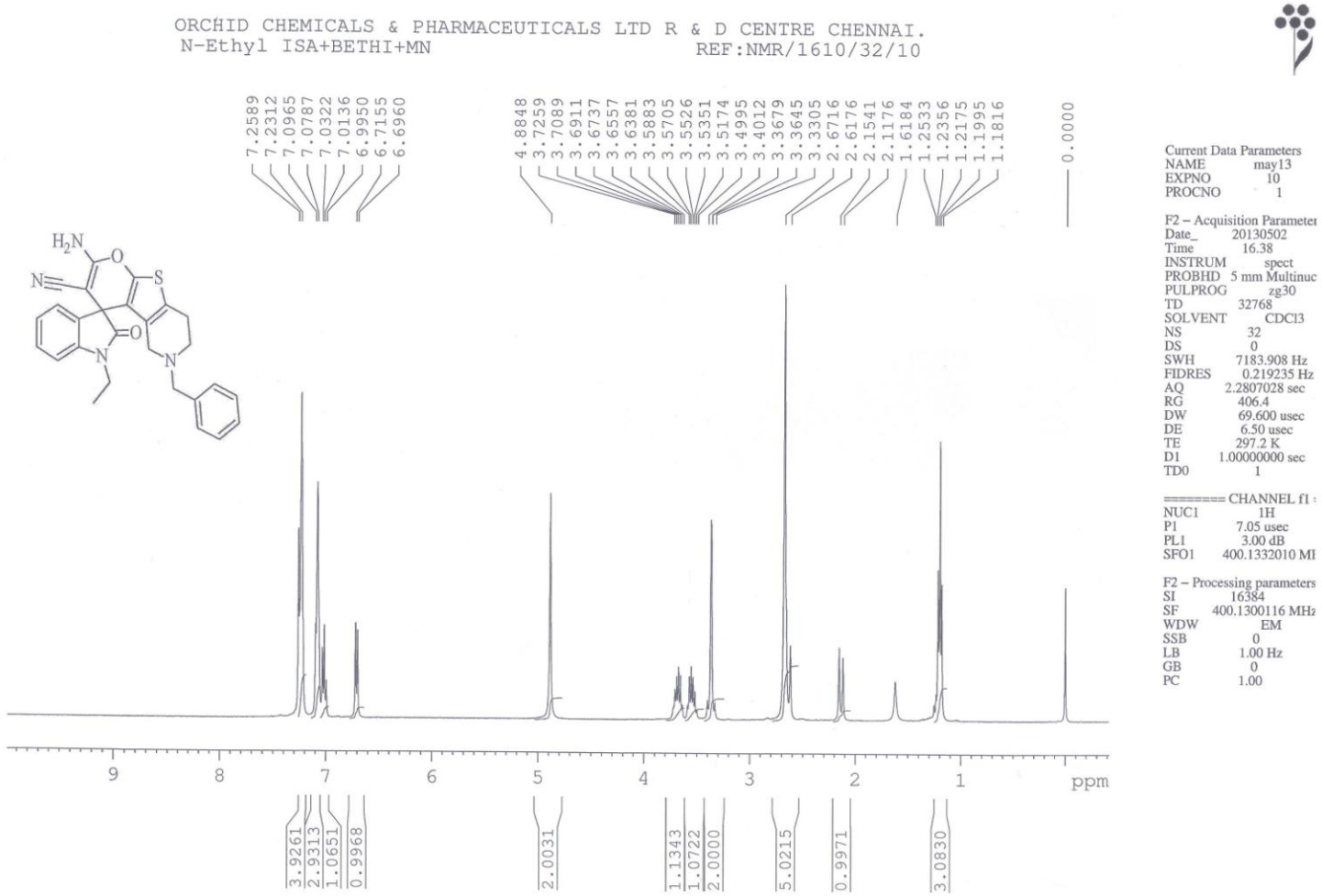
Acq. Date: Friday, May 24, 2013 ORCHID CHEMICALS AND PHARMACEUTICALS LTD., Printing Date: May 24, 2013

Acq. Time: 10:42 Sample Name: ISATIN+MN+BENZ Sample ID:

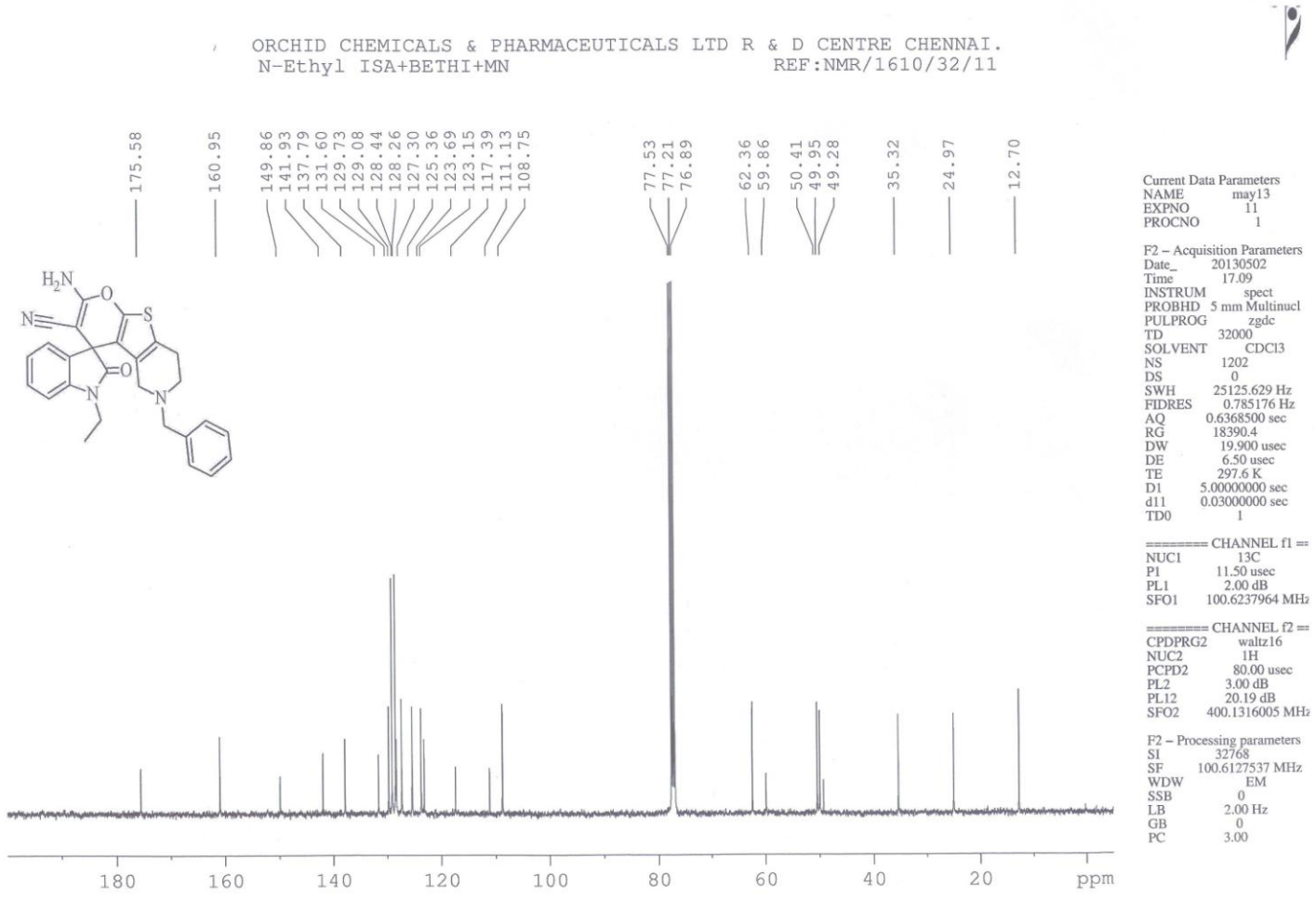


Project: ORCHID REF PAGE: HKA/1577/

Mass spectrum of 5j



^{1}H NMR spectrum of 5k



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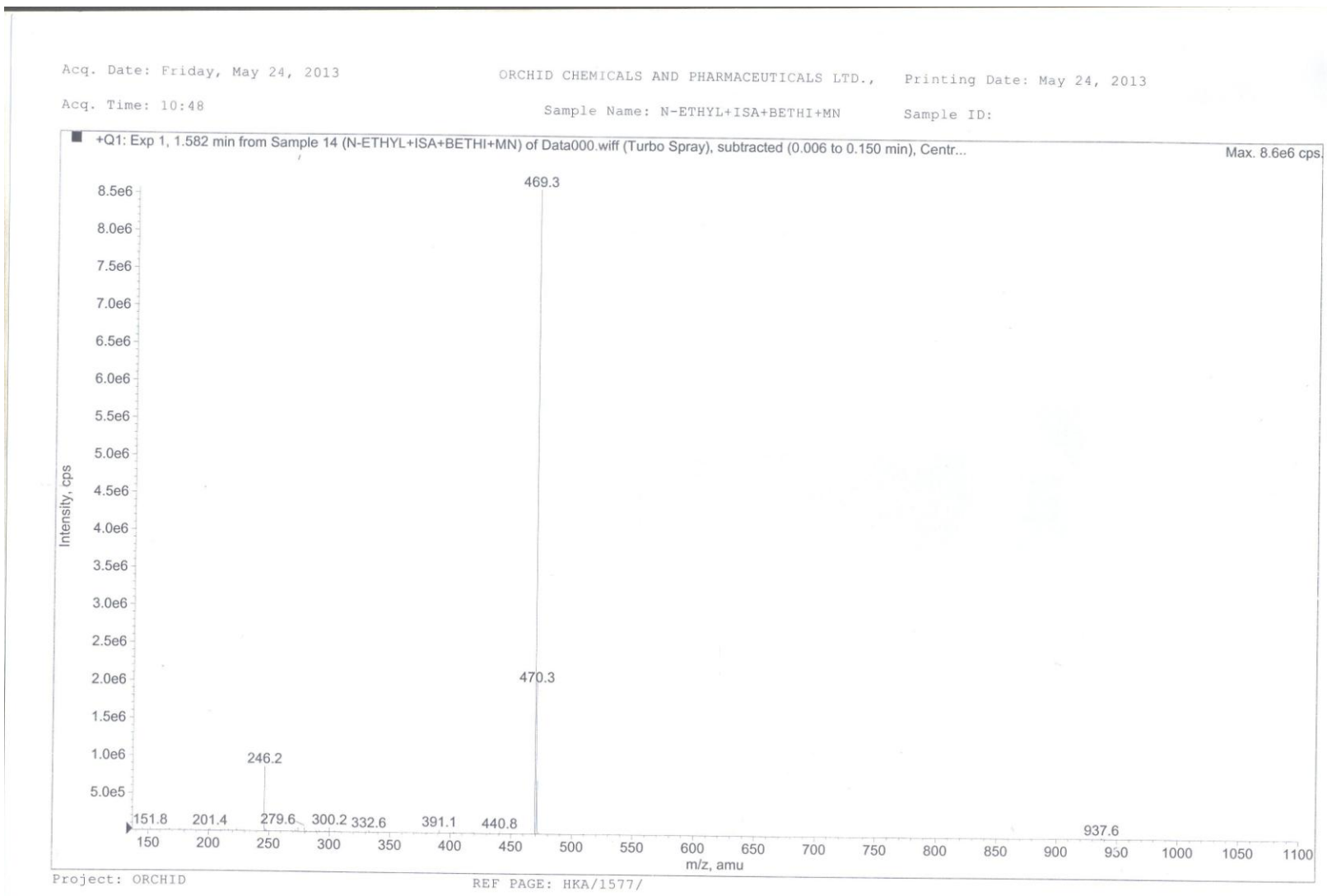

Mass spectrum of 5k

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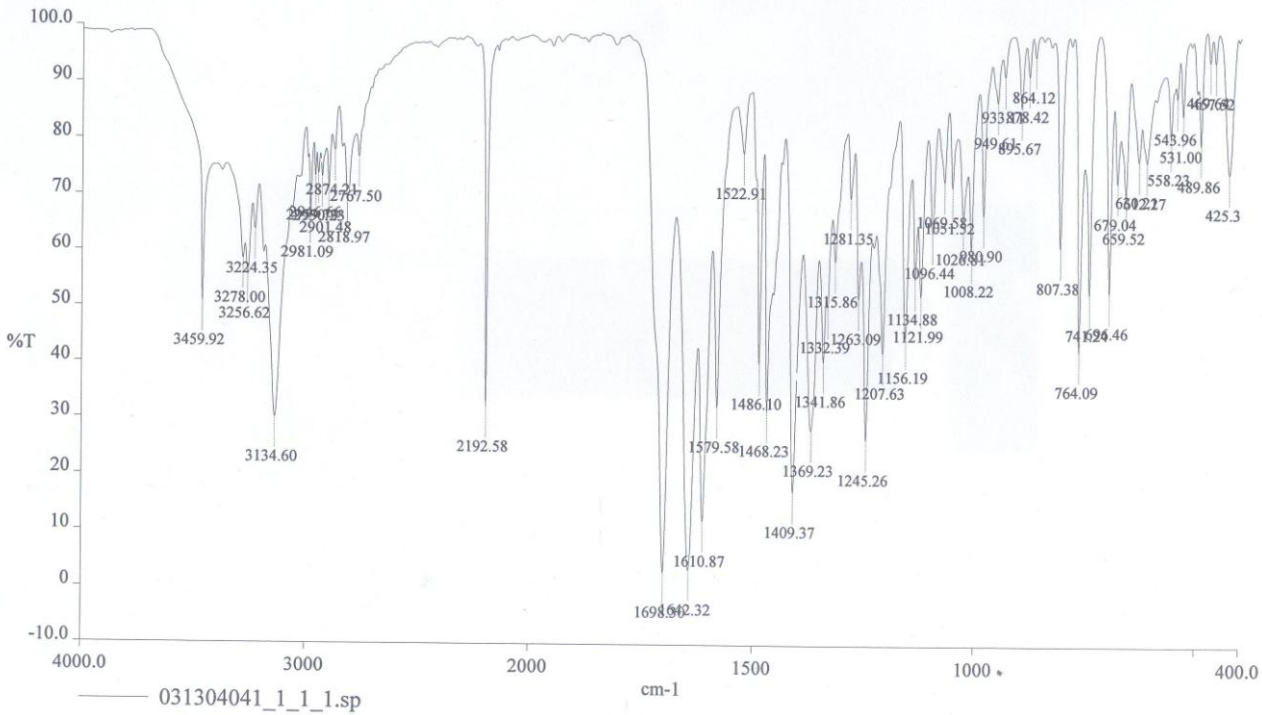

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Date Created: Tuesday, April 23, 2013 11:53

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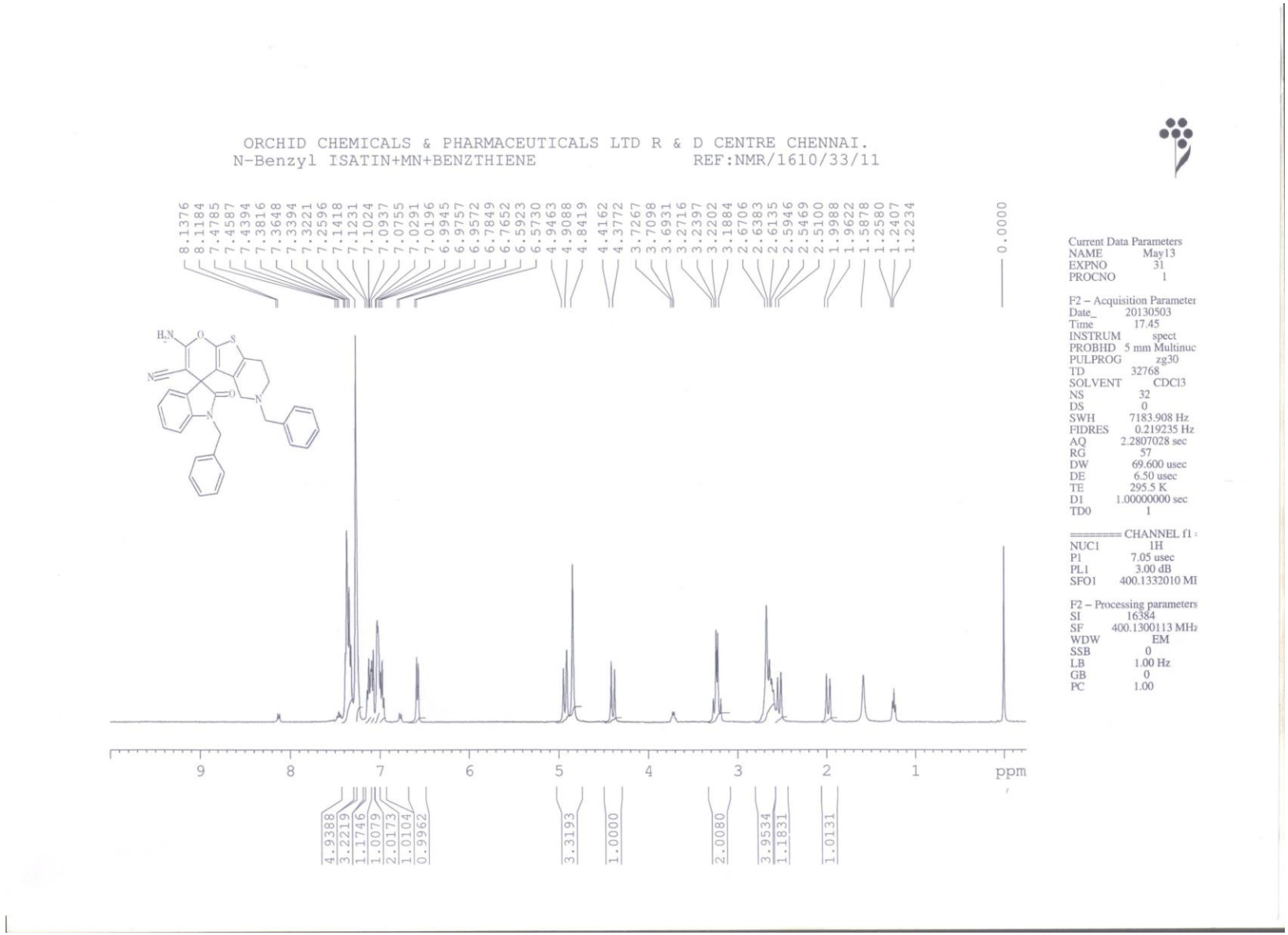
Analyst:

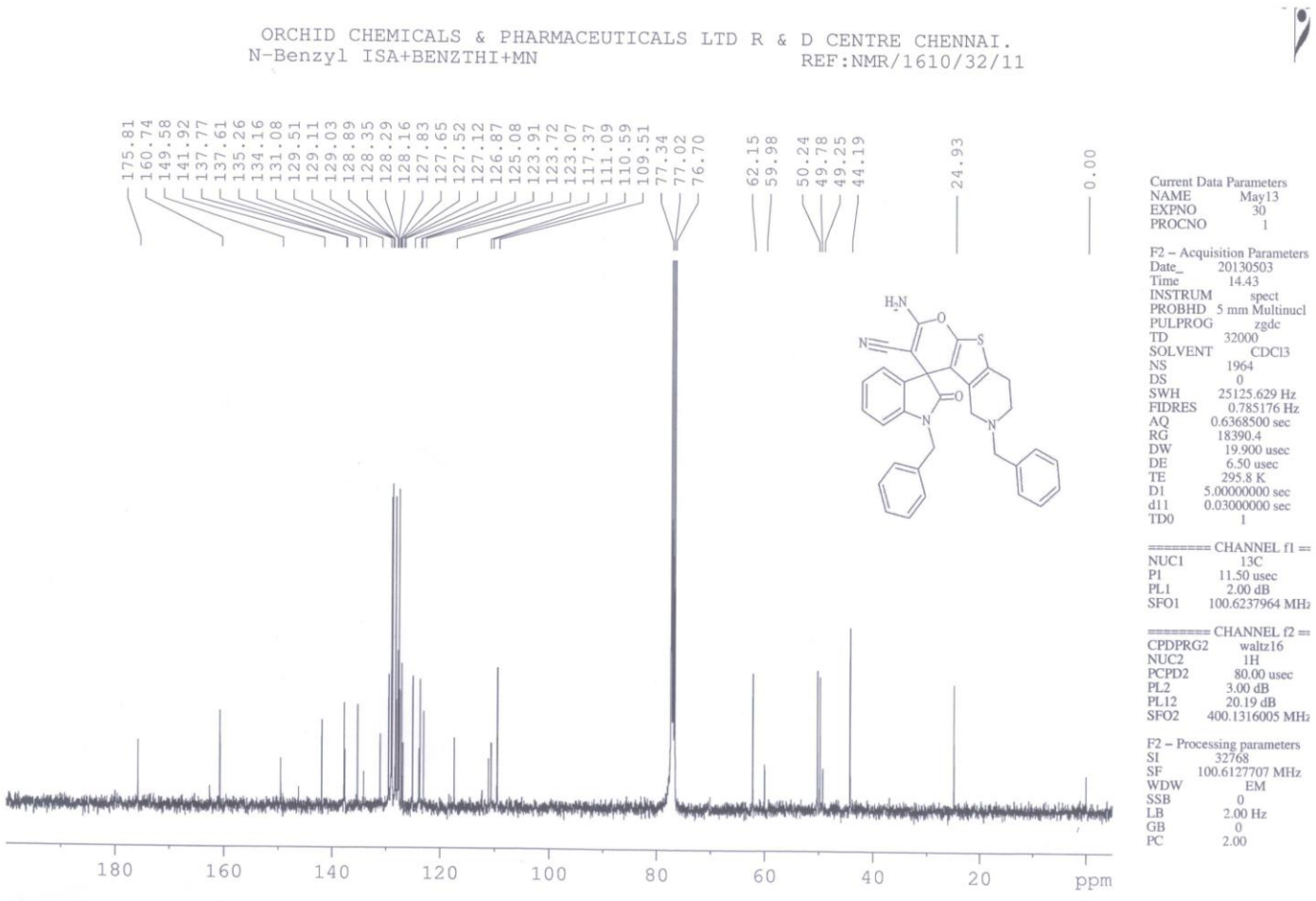
Technique:KBr/~~Neat/Nujol~~

Reference: Ra/1559/107/15

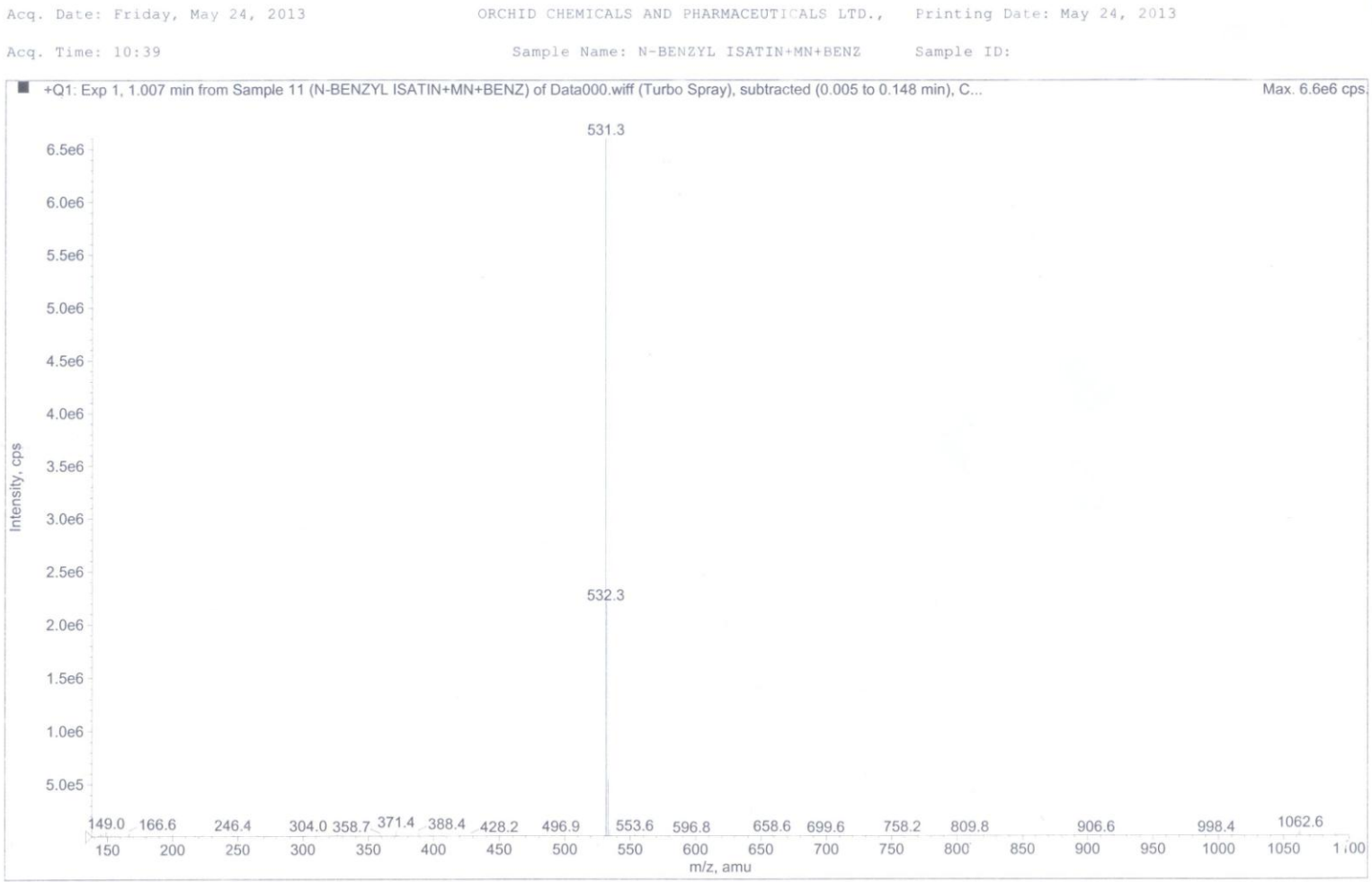
IR spectrum of 5k



^{1}H NMR spectrum of 5l



^{13}C NMR spectrum of 5l



Mass spectrum of 5l

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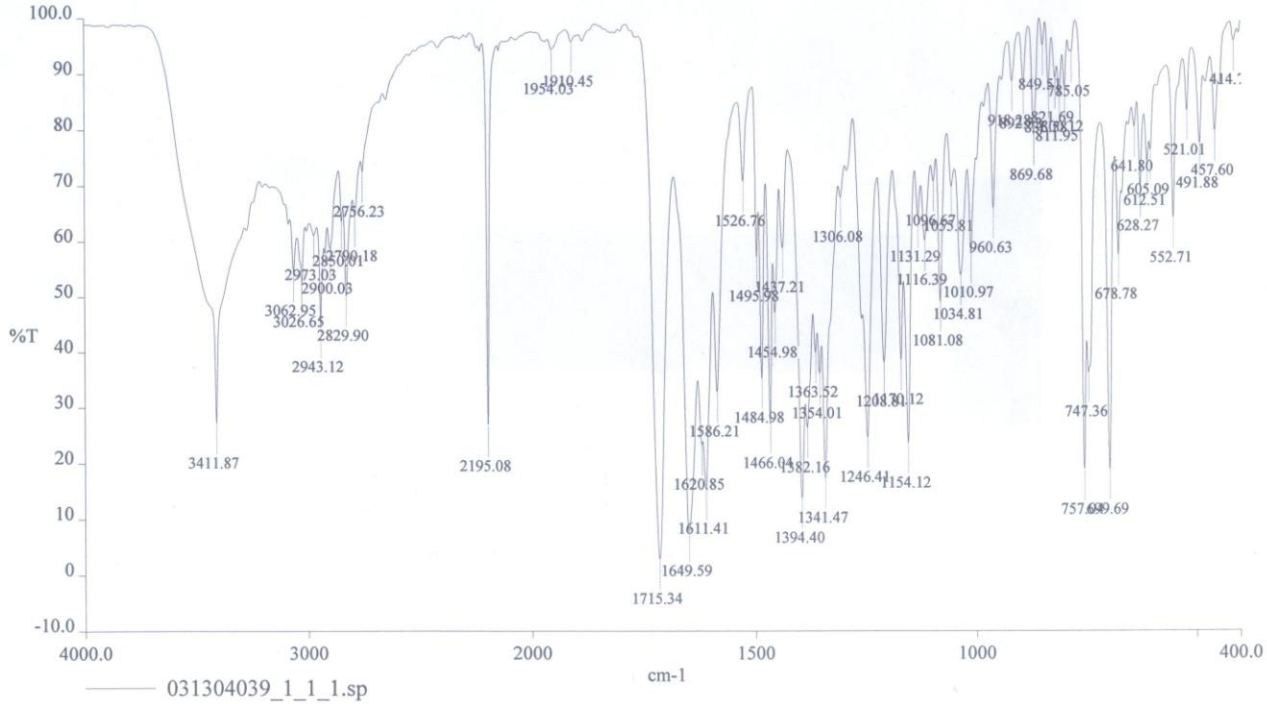

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Date Created: Tuesday, April 23, 2013 11:39

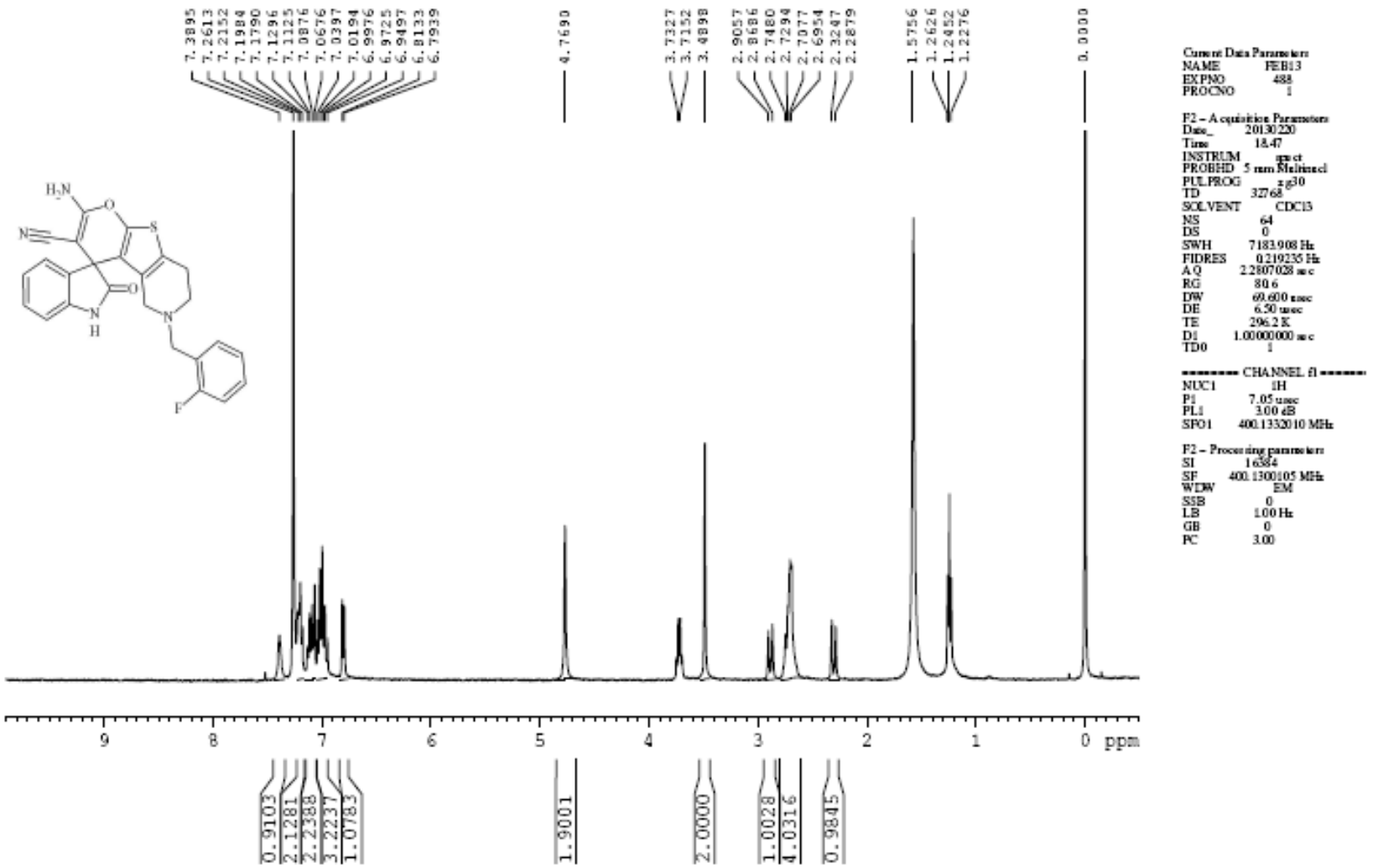
Description: N-BENZYL ISATIN+MN+BENZ-II

Analyst: 25/4/13

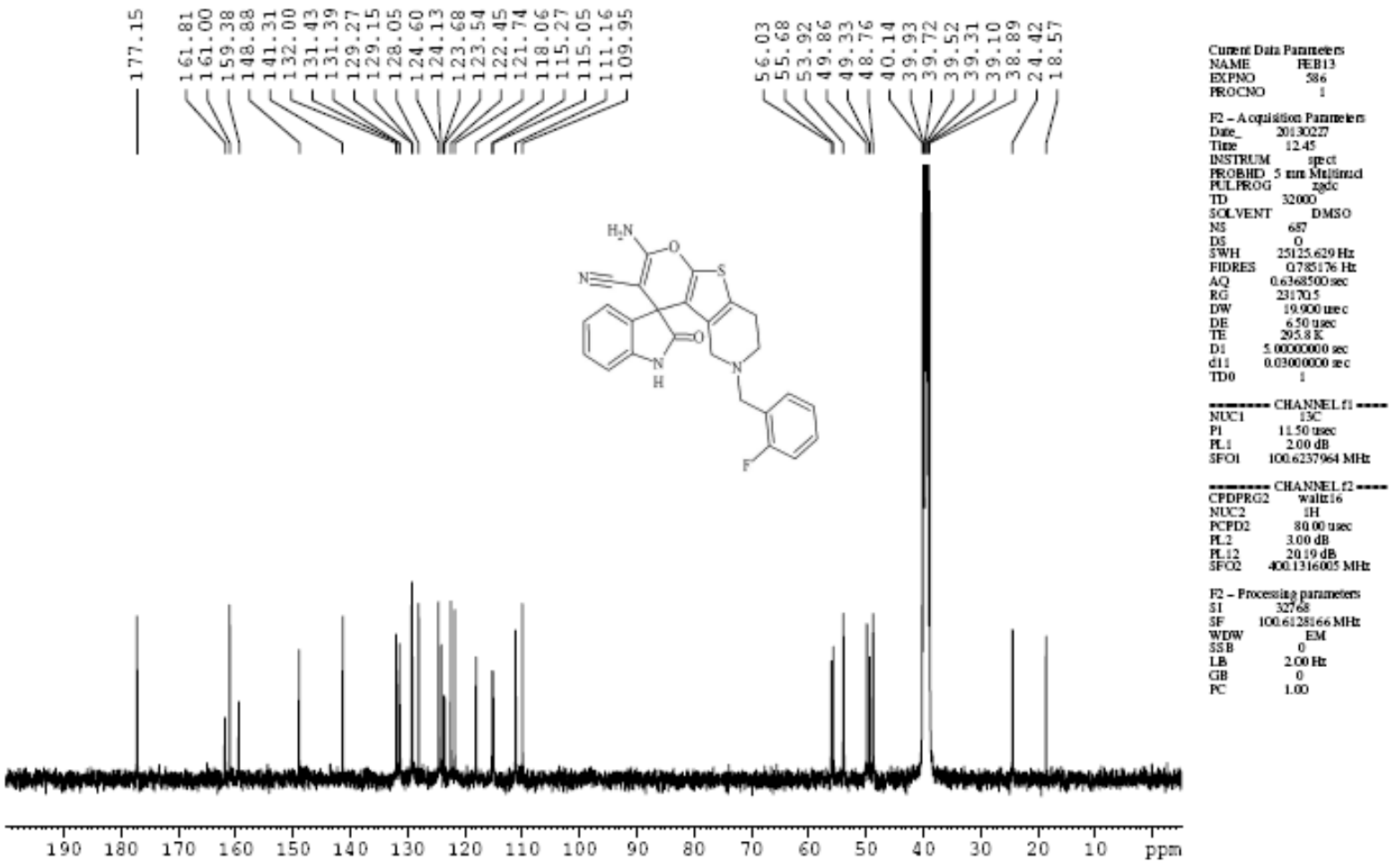
Technique: KBr/~~Neat~~/~~Nujol~~

Reference: DR/1555/1071/13

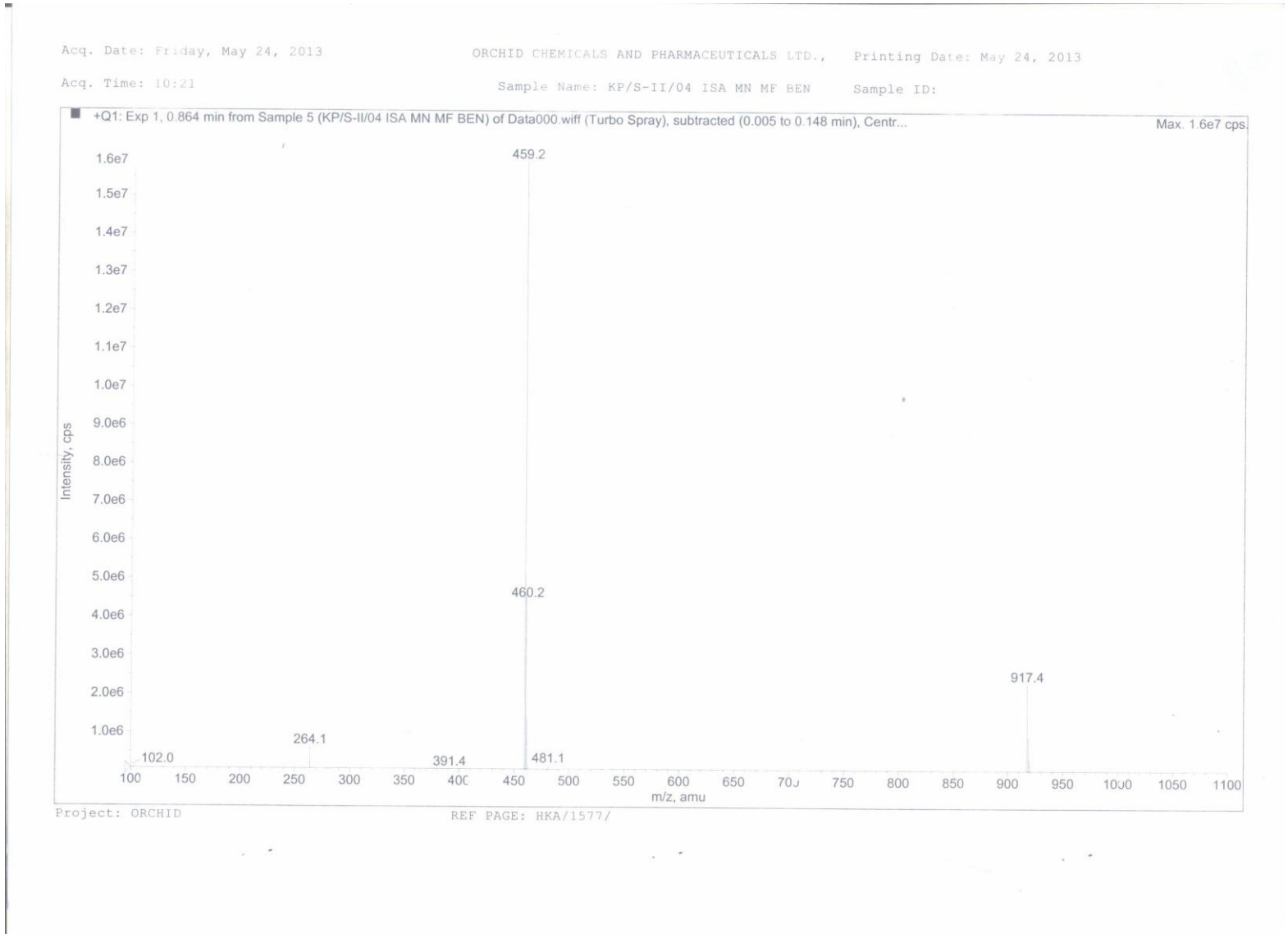
IR spectrum of 5l



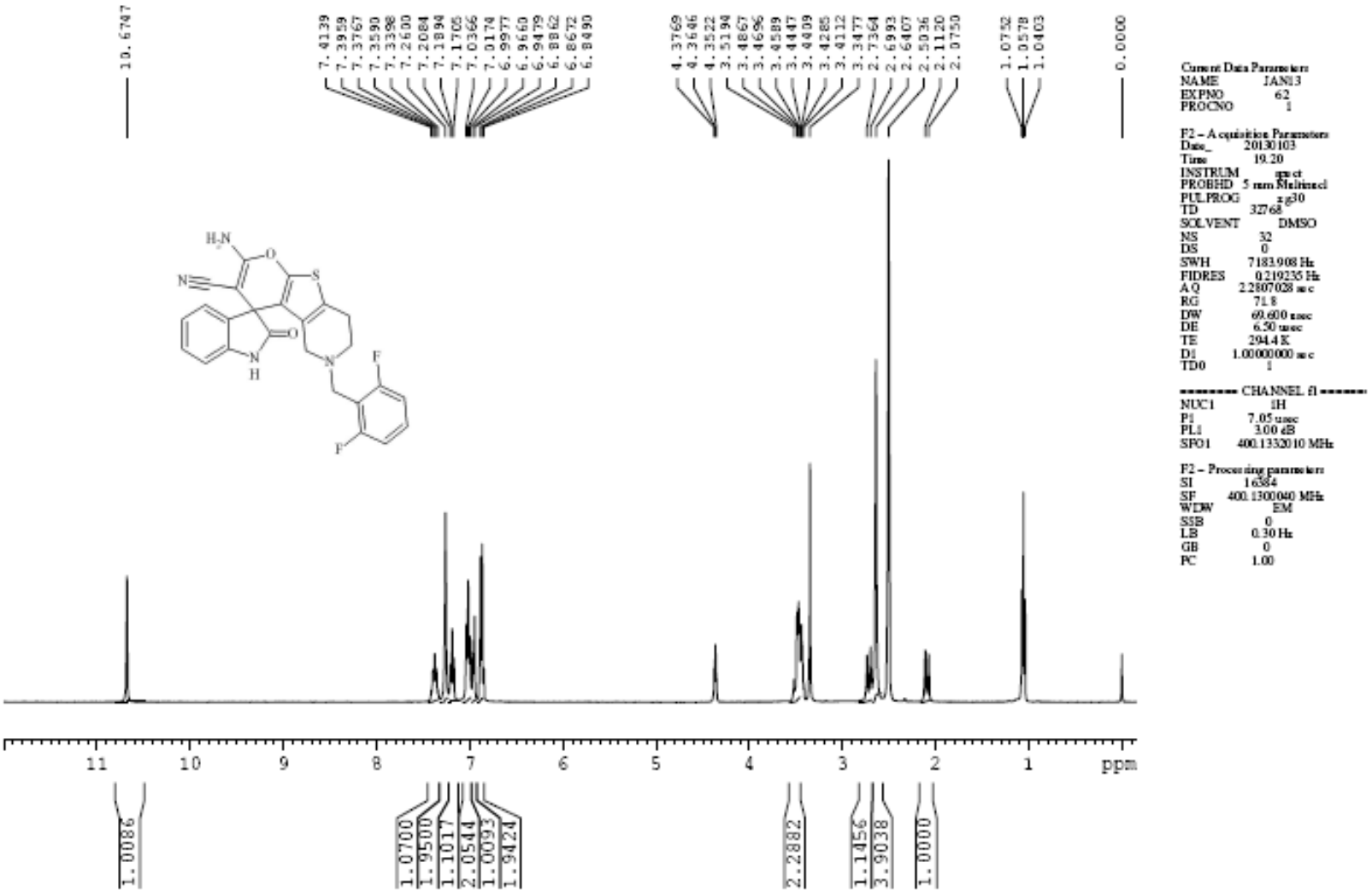
^{1}H NMR spectrum of 5o



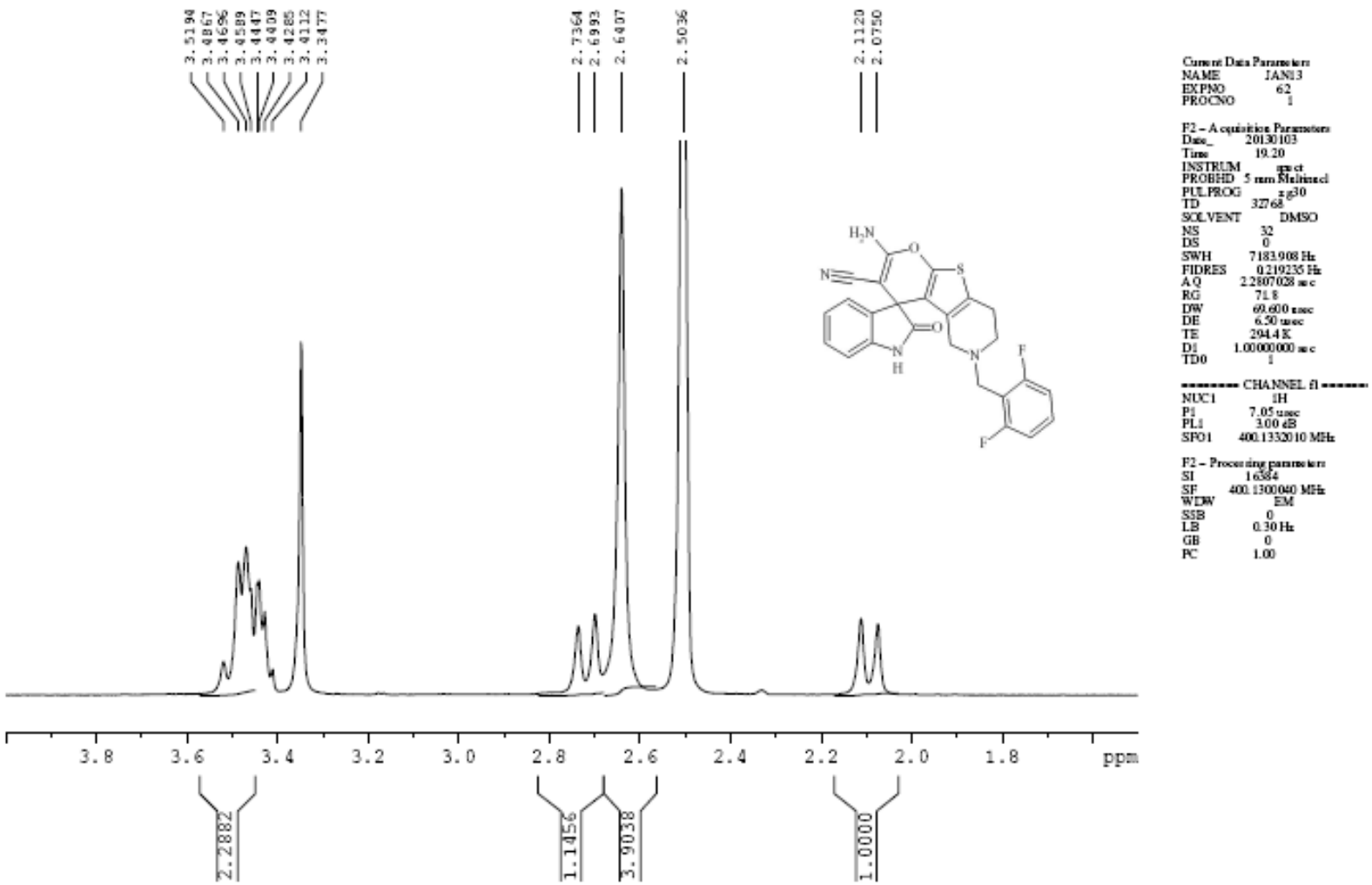
^{13}C NMR spectrum of 5o



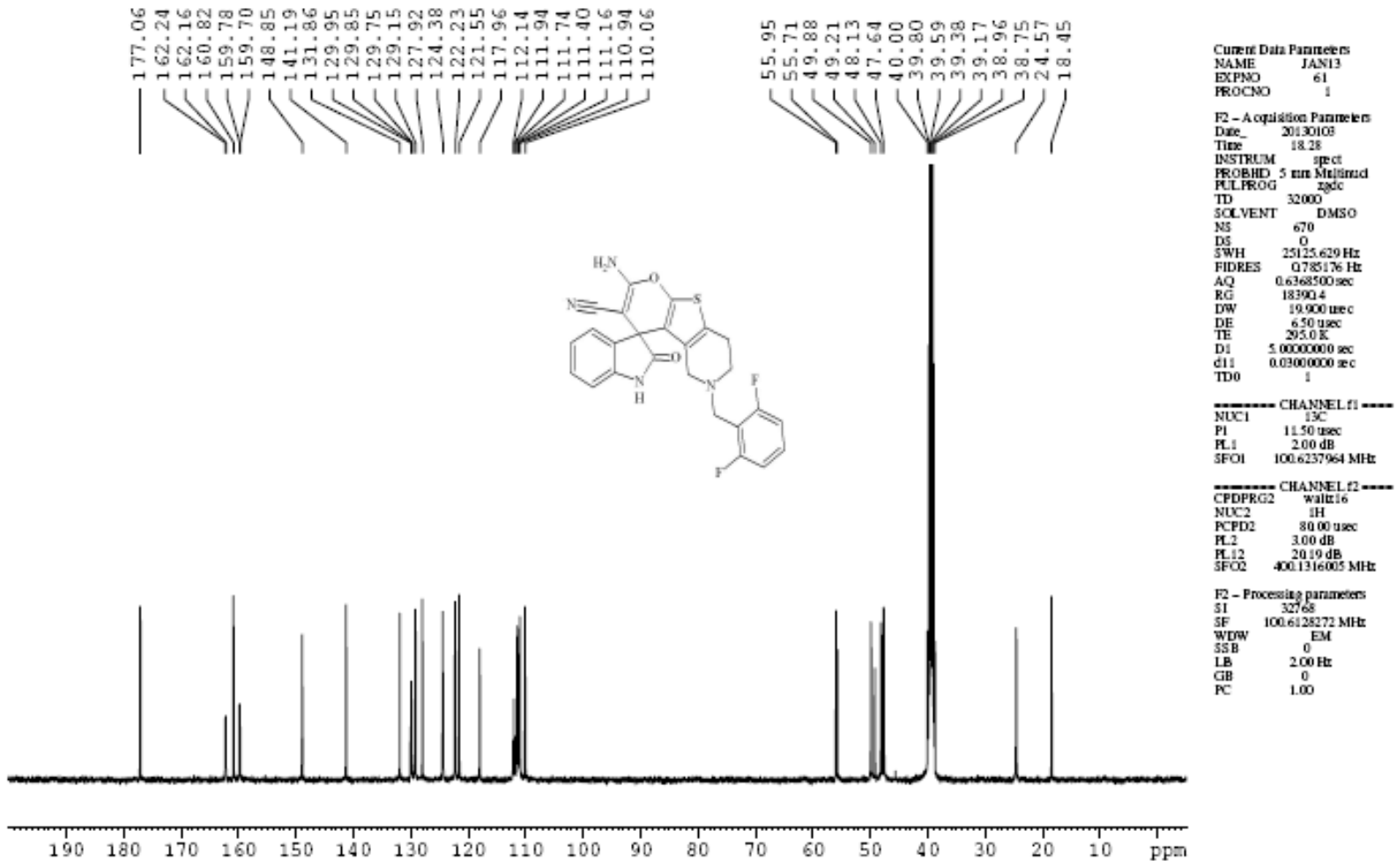
Mass spectrum of 5o



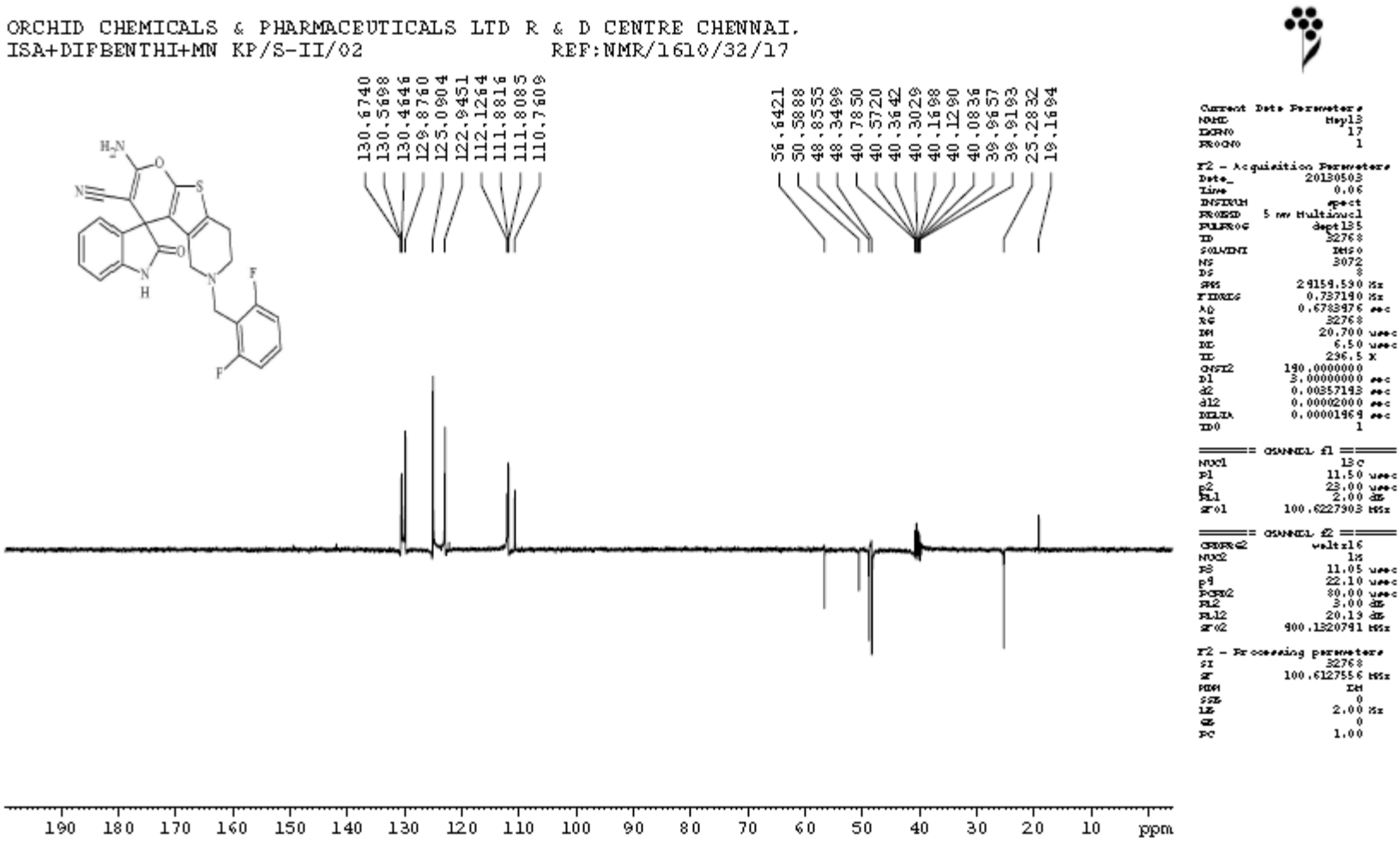
^{1}H NMR spectrum of 5t



Expanded ^{1}H NMR spectrum of 5t



^{13}C NMR spectrum of 5t



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PL1 2.00 dB
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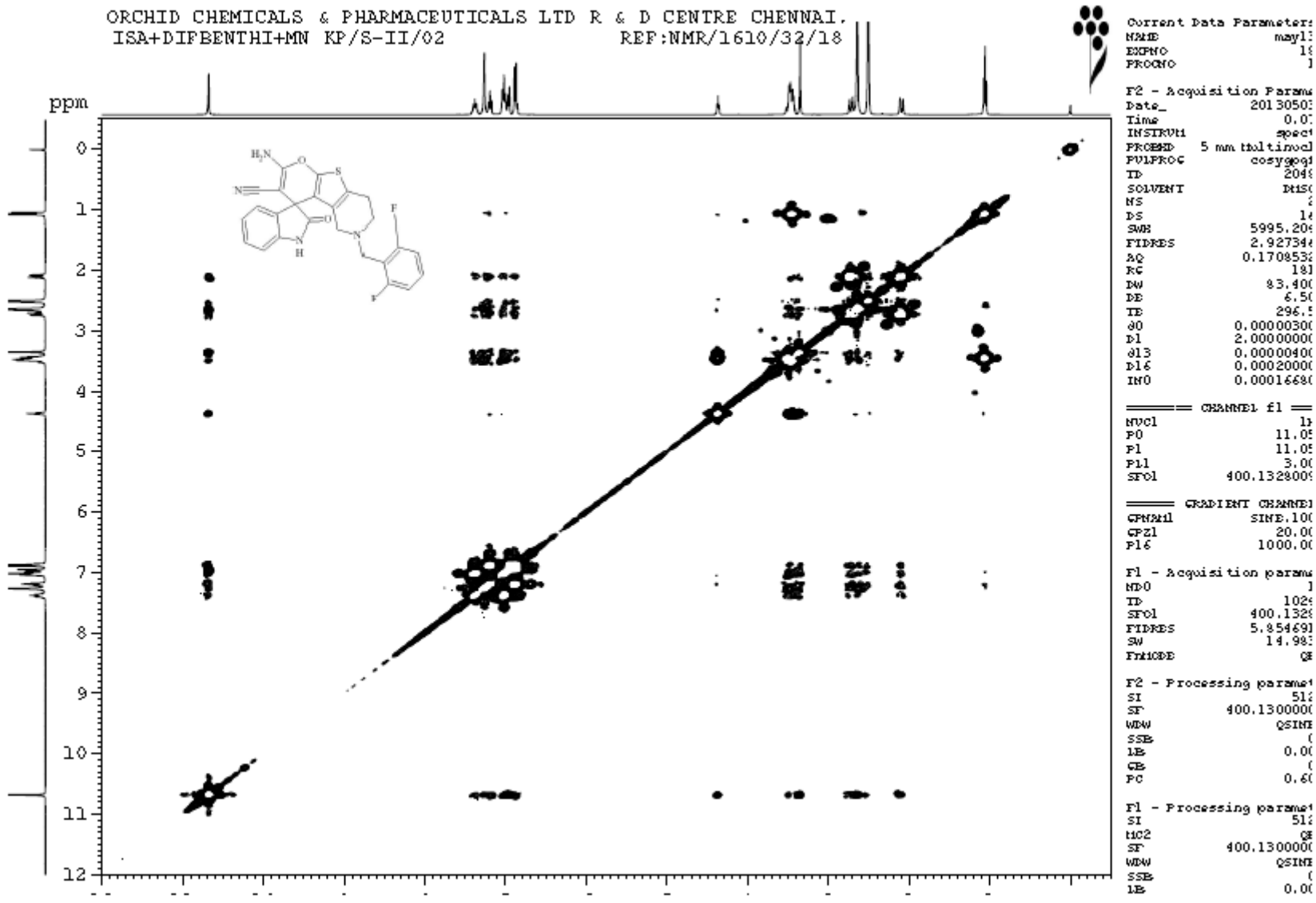
DEPT-135 ^{13}C NMR of compound 5t



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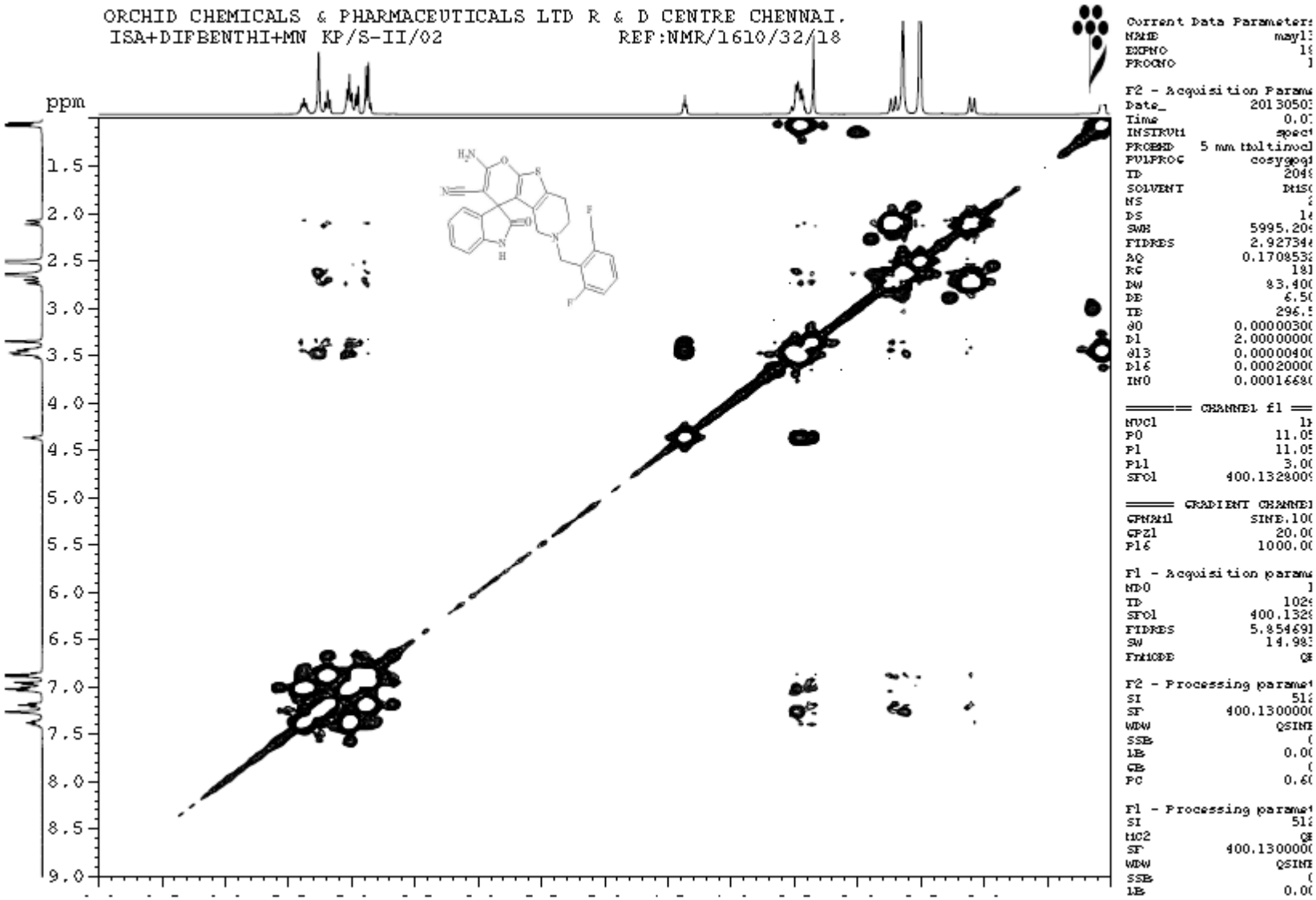
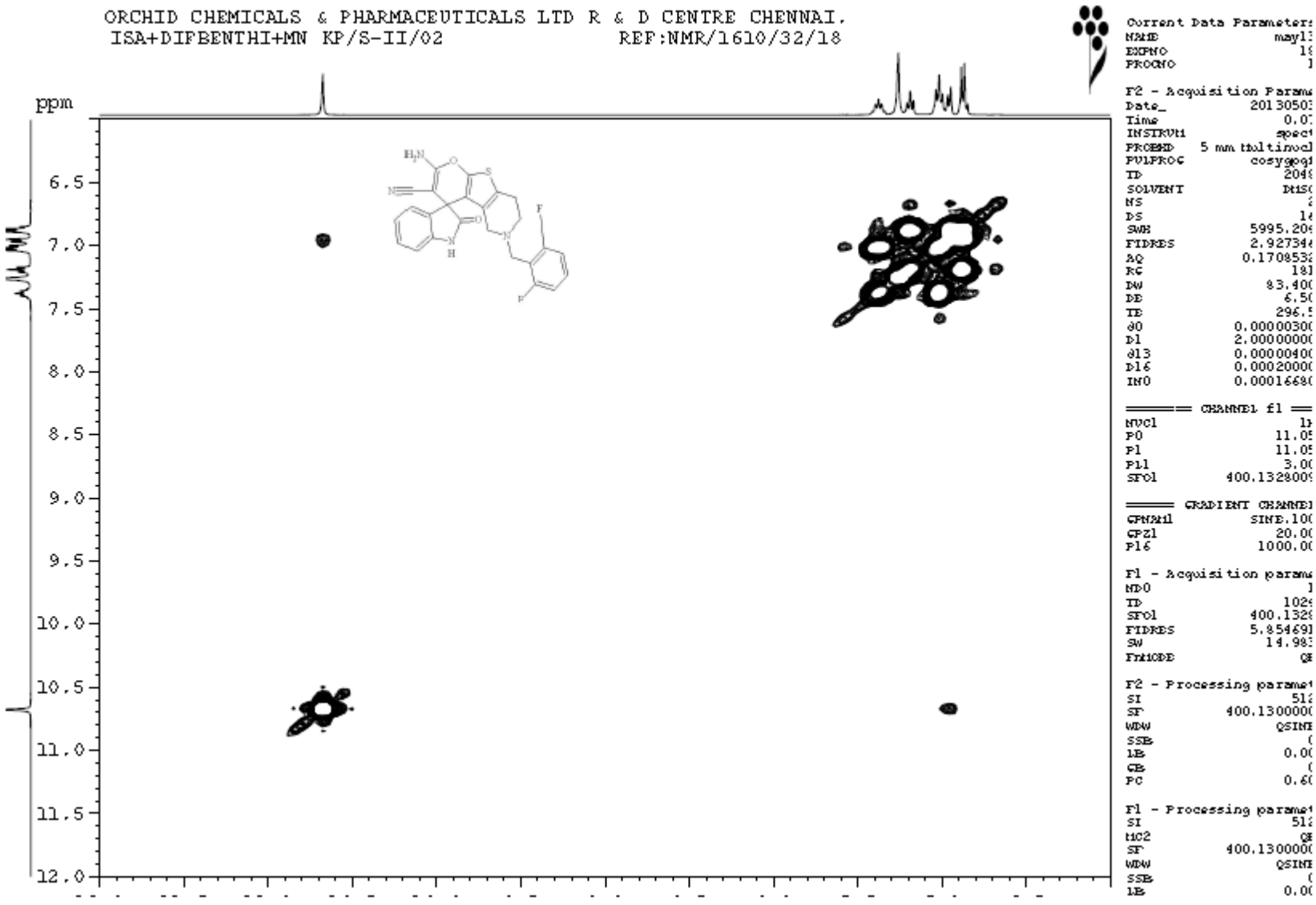
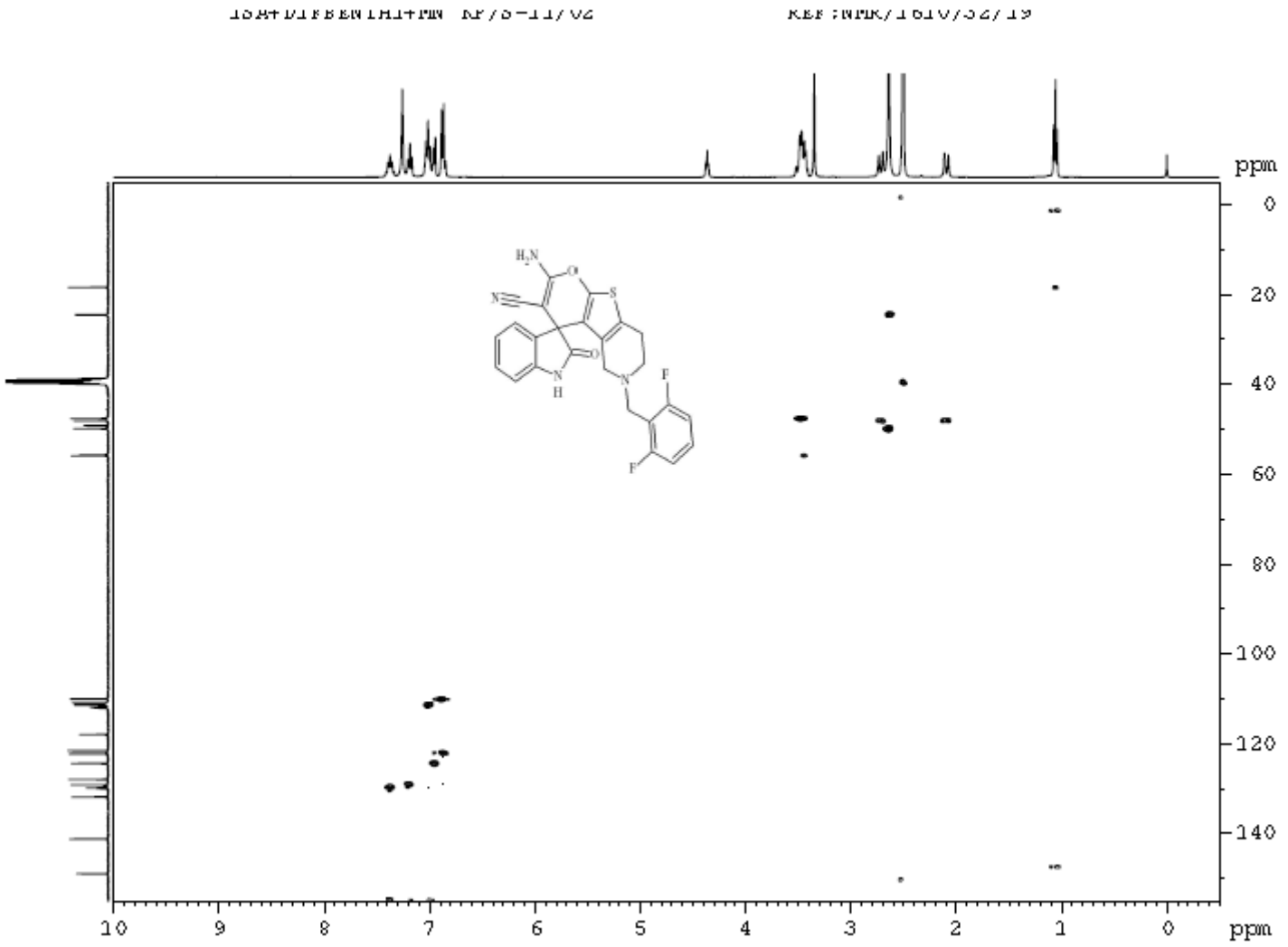


Expanded COSY spectrum of compound 5t



Expanded COSY spectrum of compound 5t



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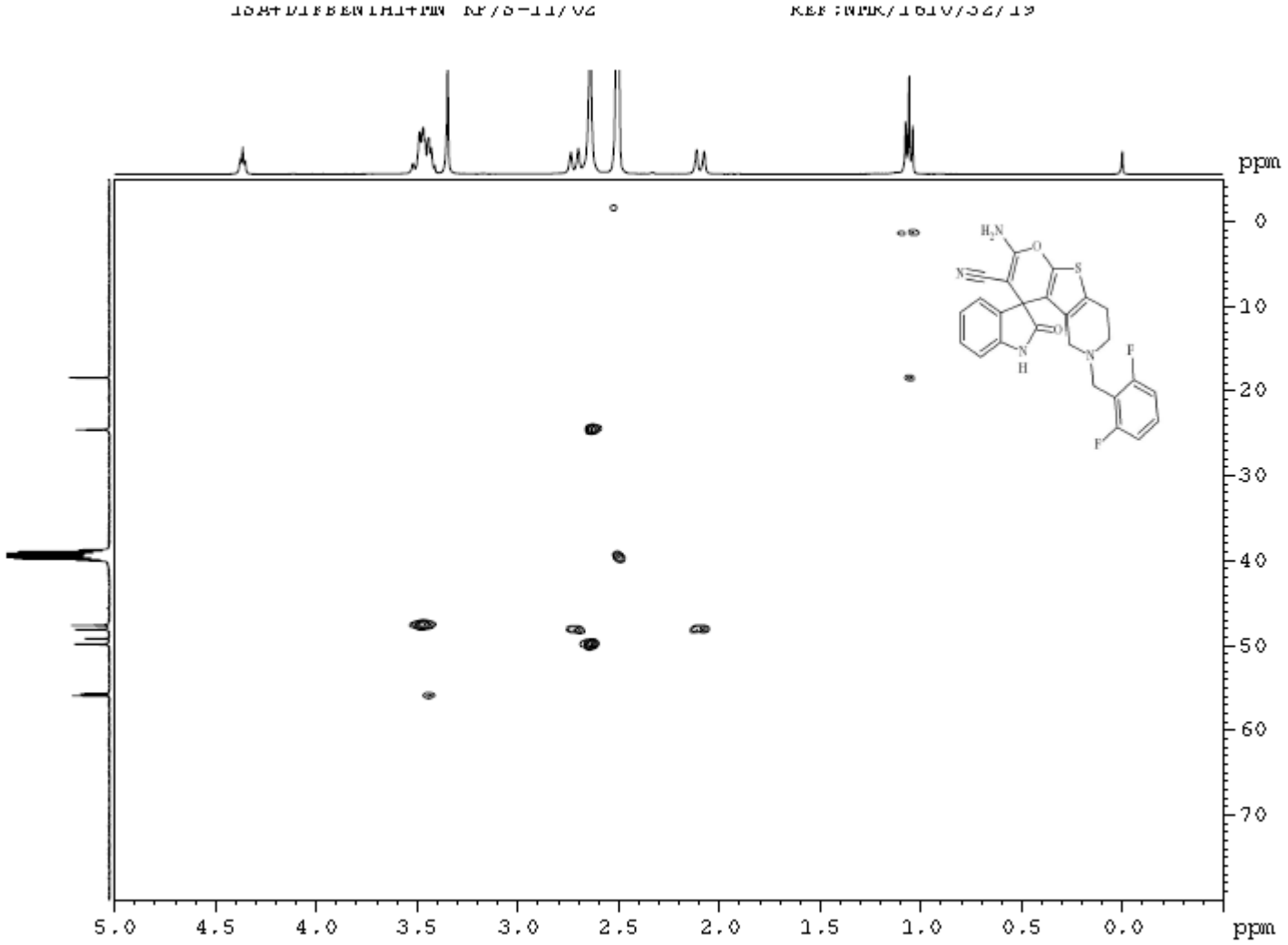
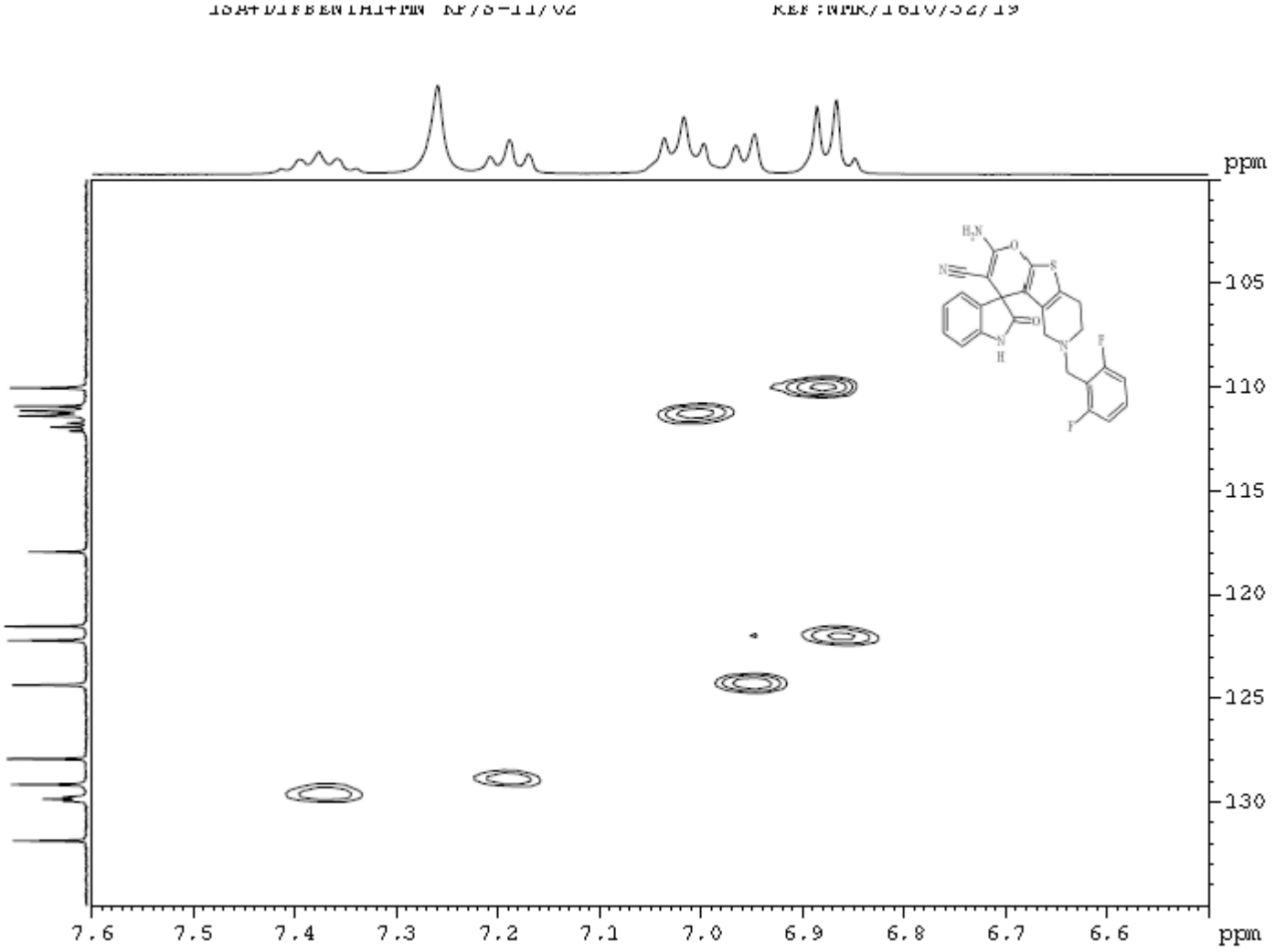
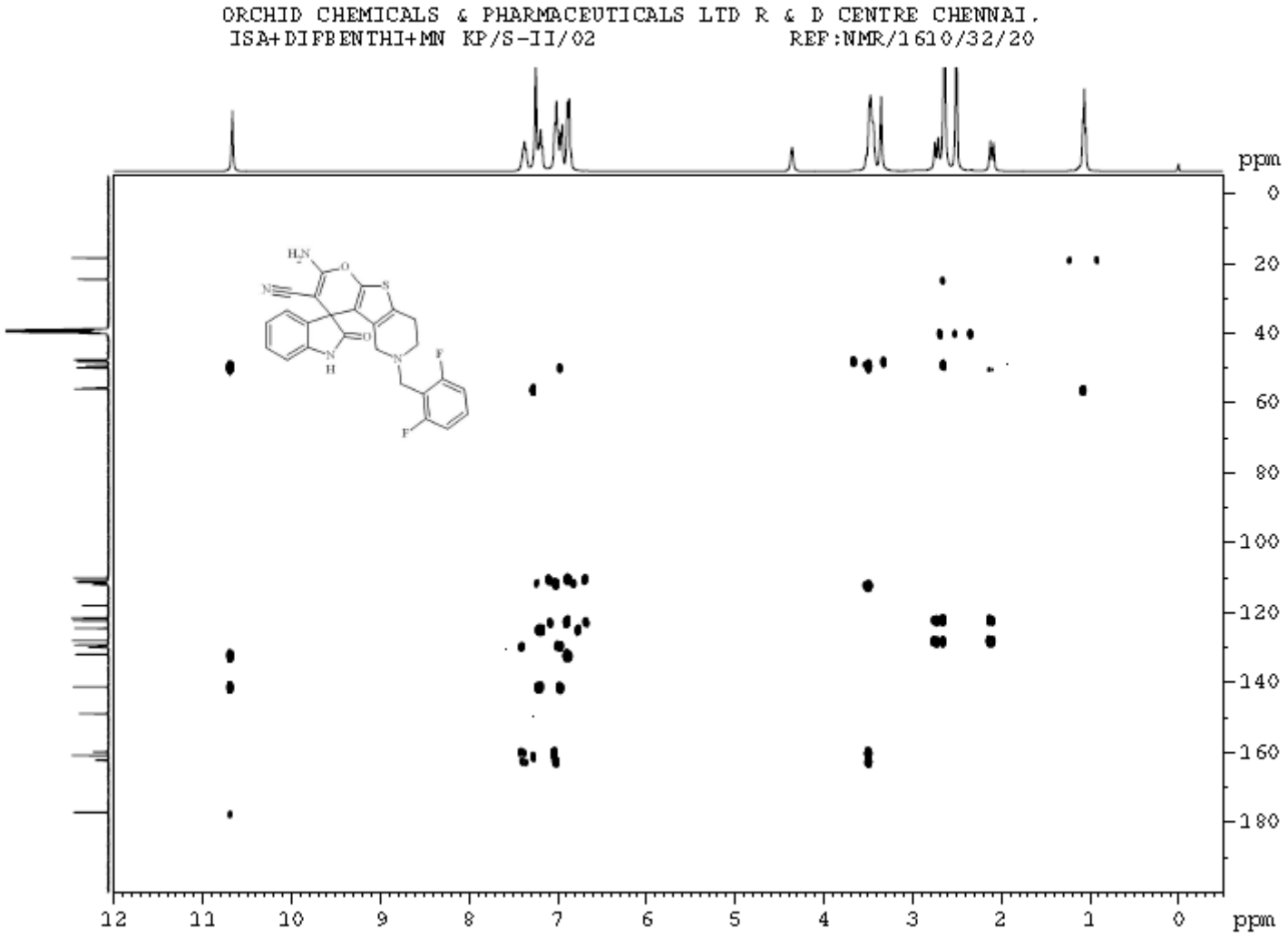


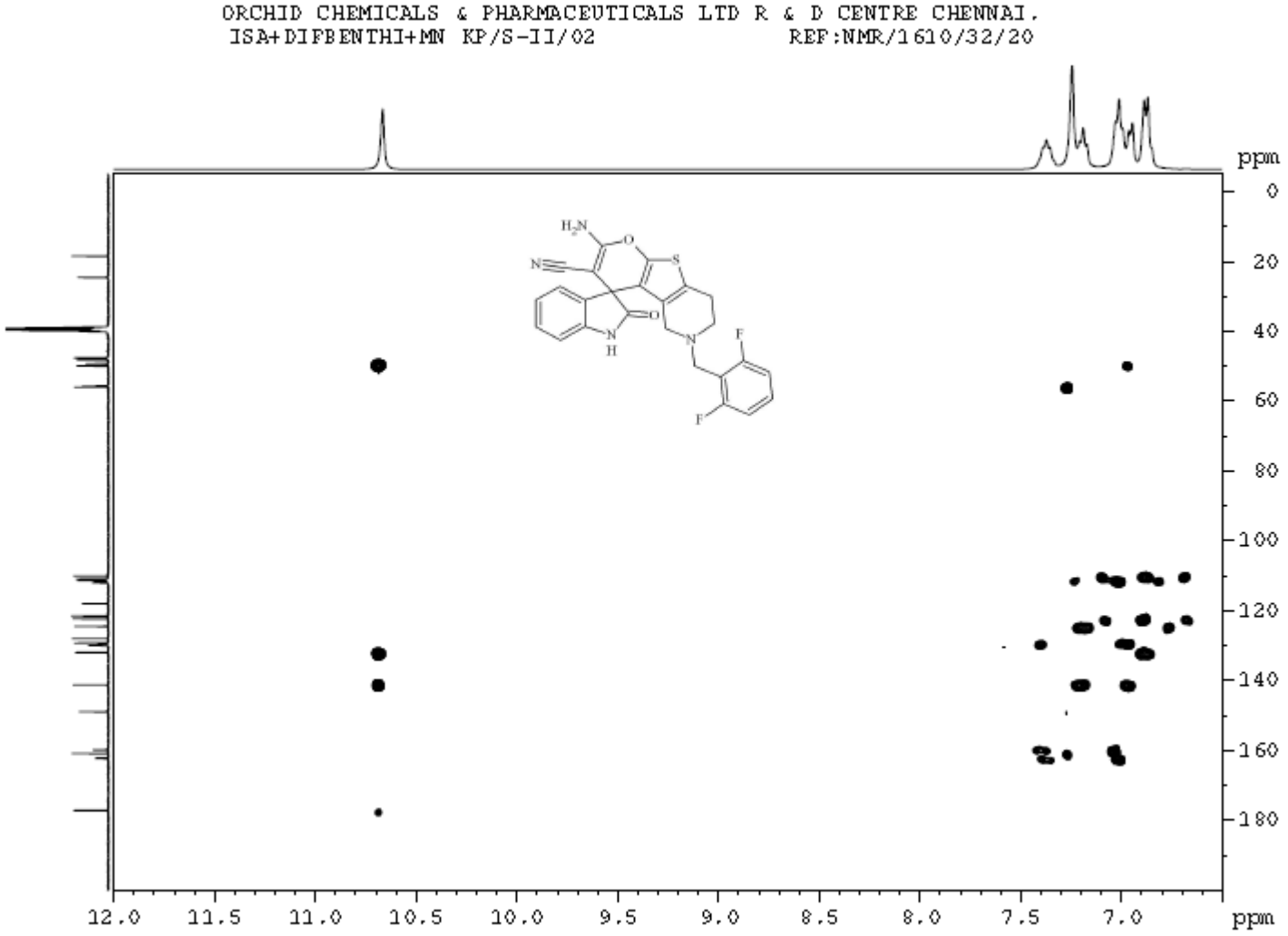
Expanded HSQC spectrum of compound 5t



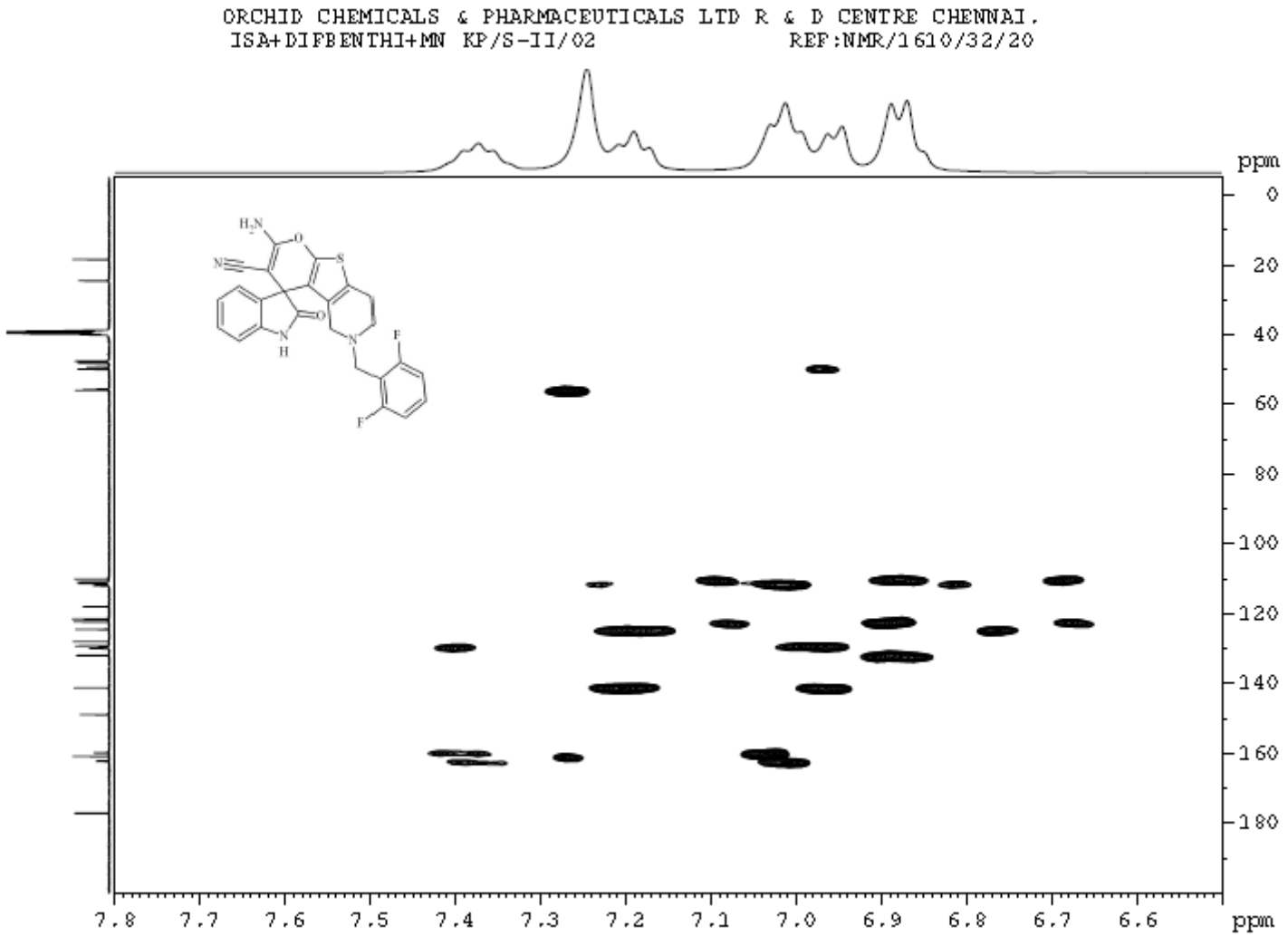
Expanded HSQC spectrum of compound 5t



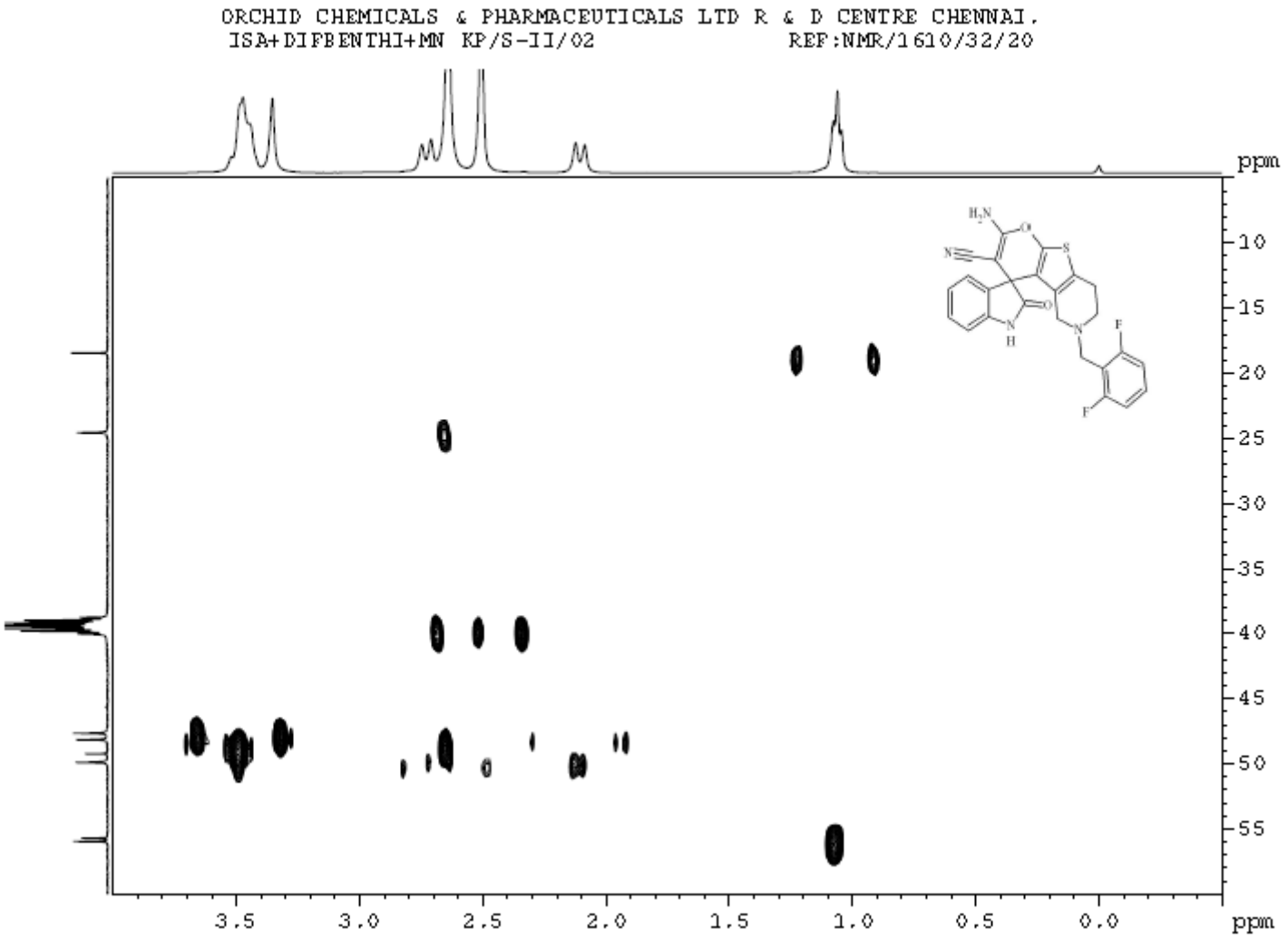
HMBC spectrum of compound 5t



Expanded HMBC spectrum of compound 5t



Expanded HMBC spectrum of compound 5t



Expanded HMBC spectrum of compound 5t

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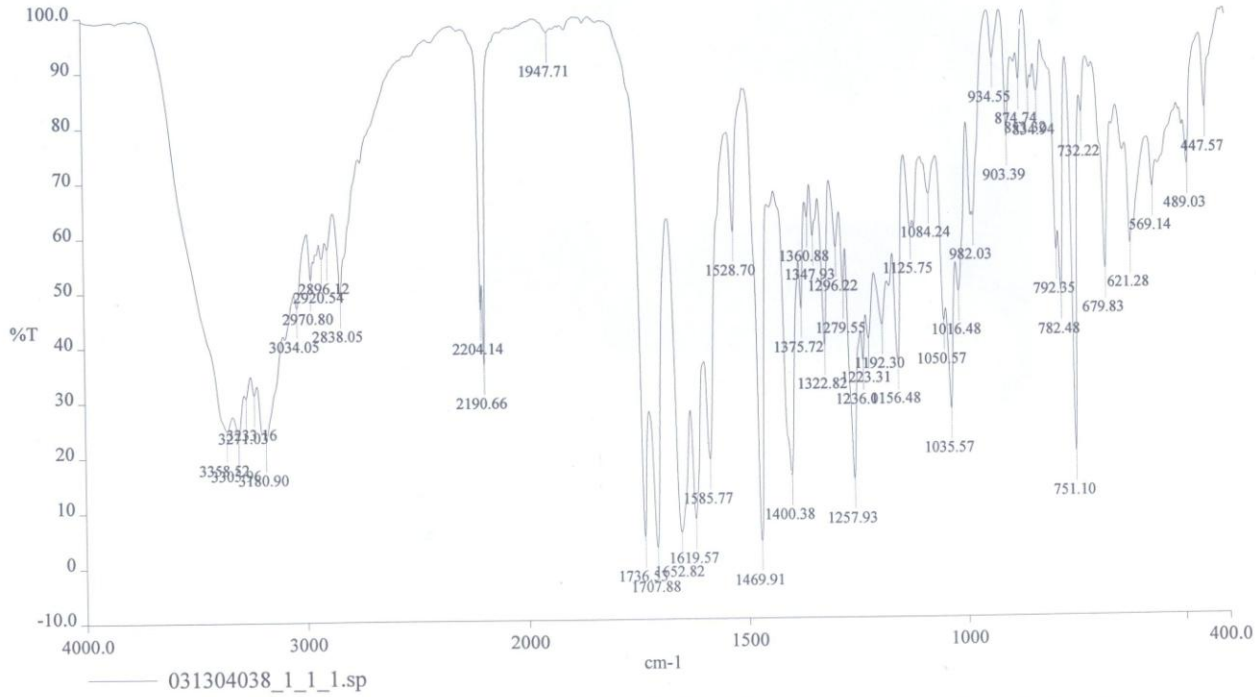

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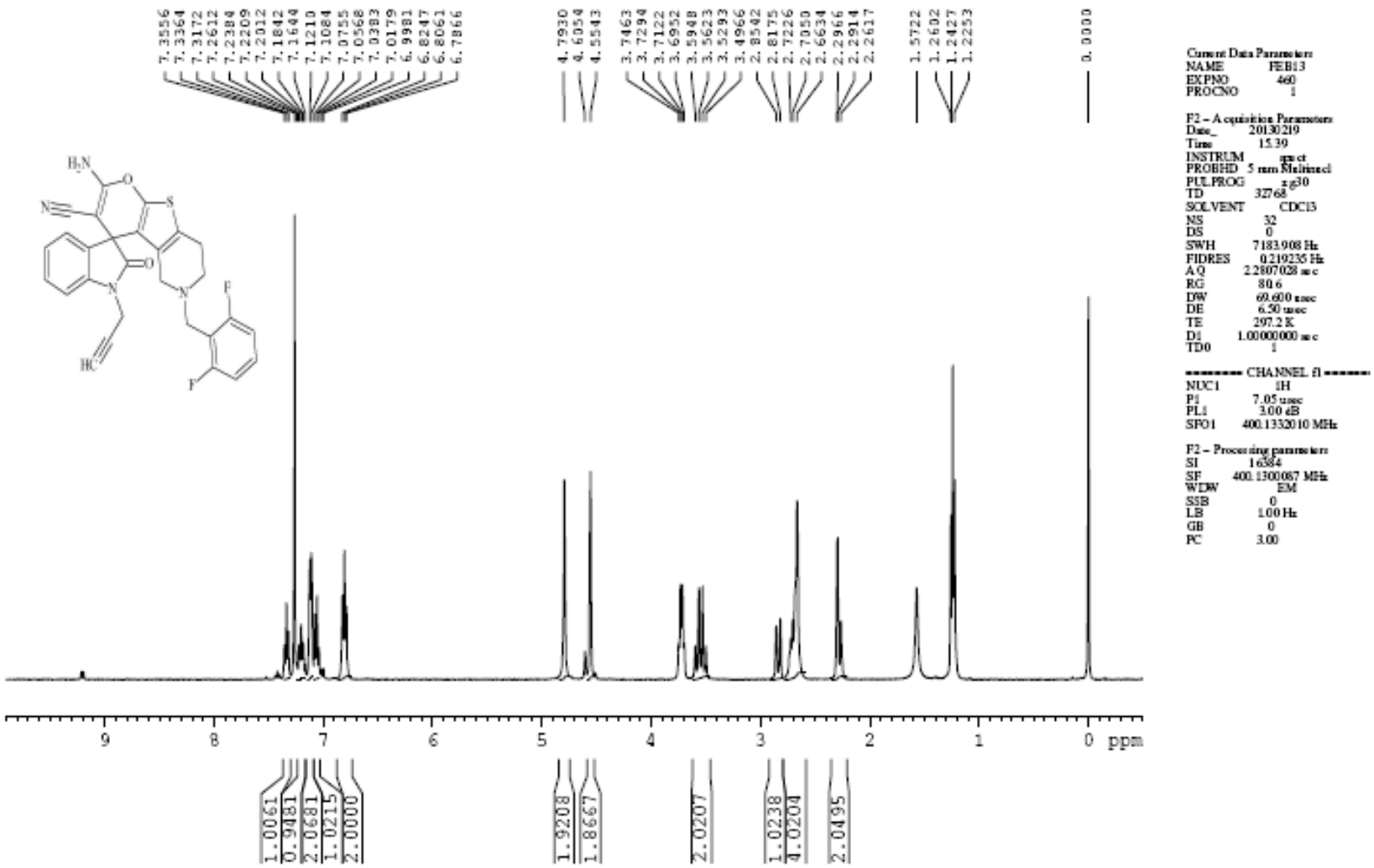
Analyst: 25/23/04/13

Technique: ~~KBr~~/~~Neat~~/~~Nujol~~

Reference: Da 1155 / 107 / 12

IR spectrum of 5t



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TE 297.2 K
D1 1.00000000 sec
TD0 1

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PL1 3.00 dB
SFO1 400.1332010 MHz

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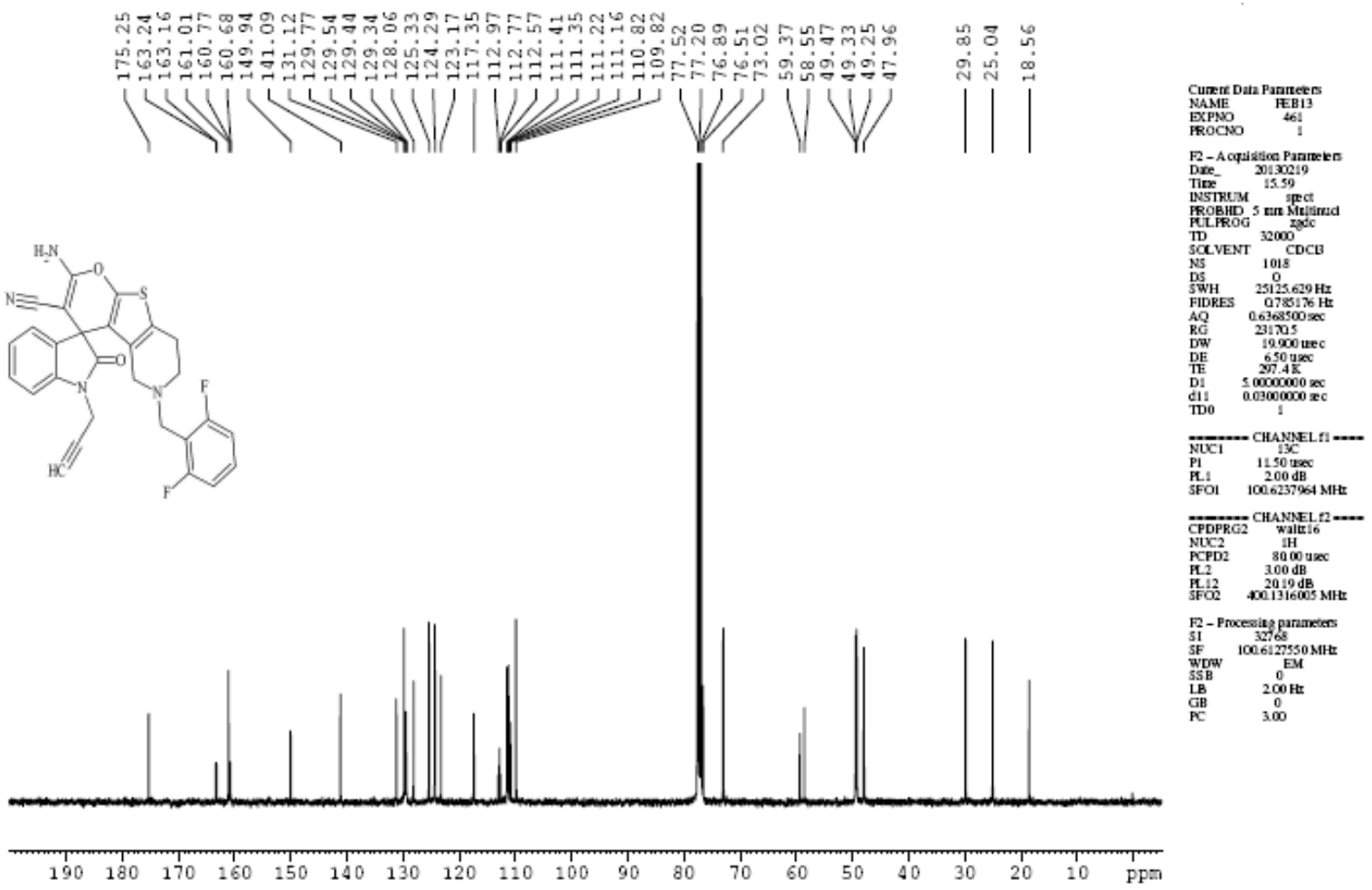
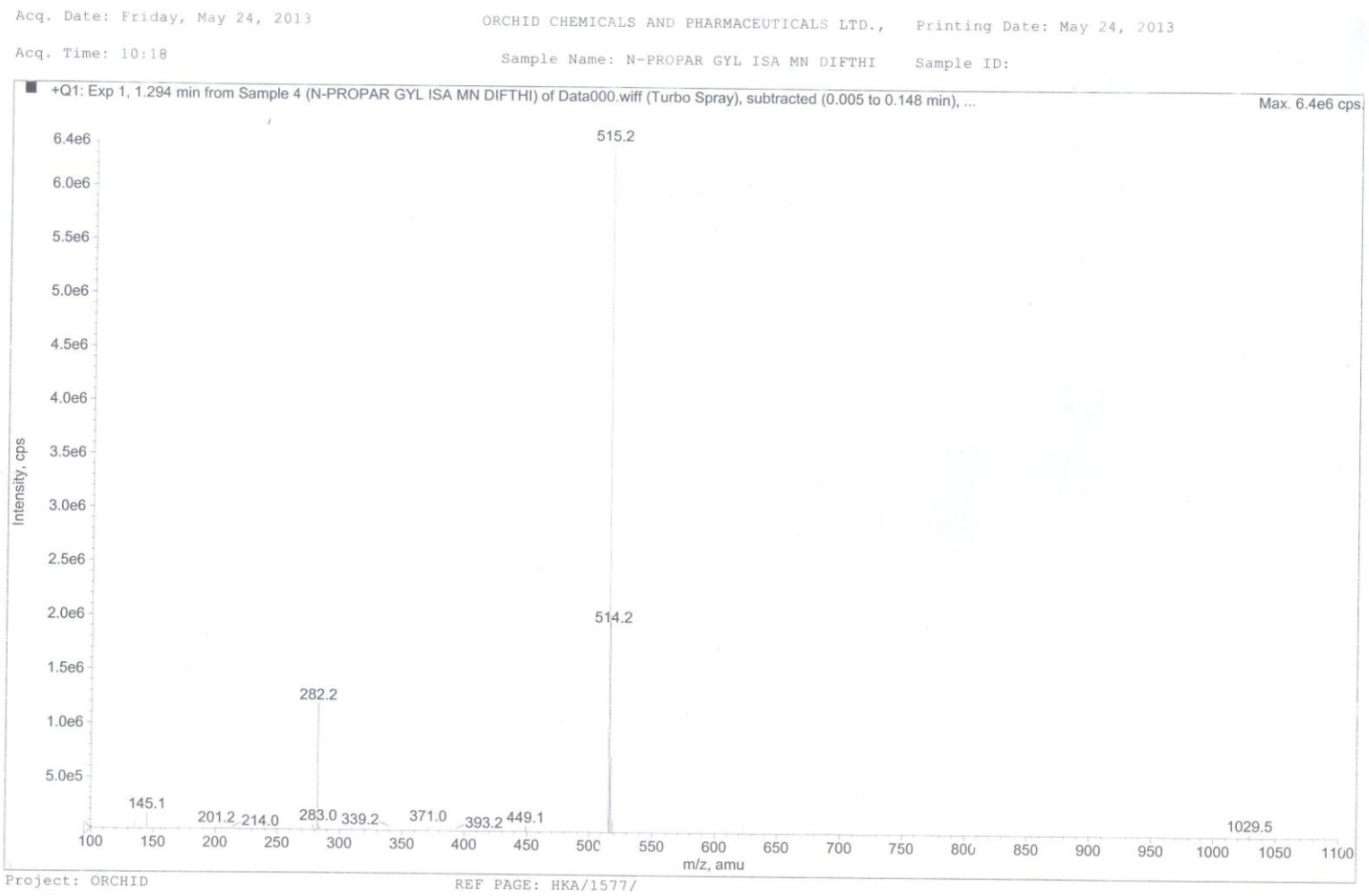
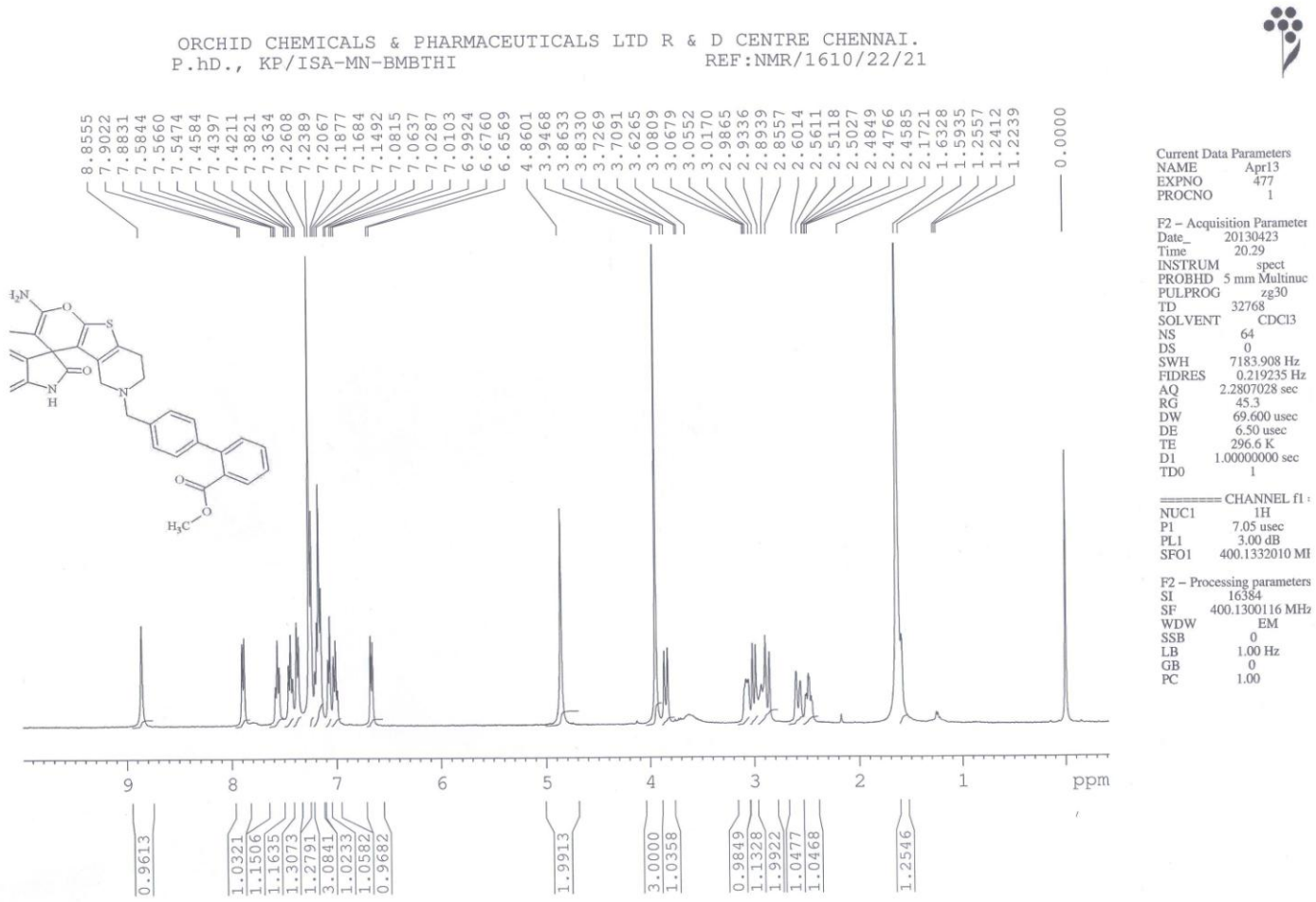
^{1}H NMR spectrum of 5u



^{13}C NMR spectrum of 5u



Mass spectrum of 5u



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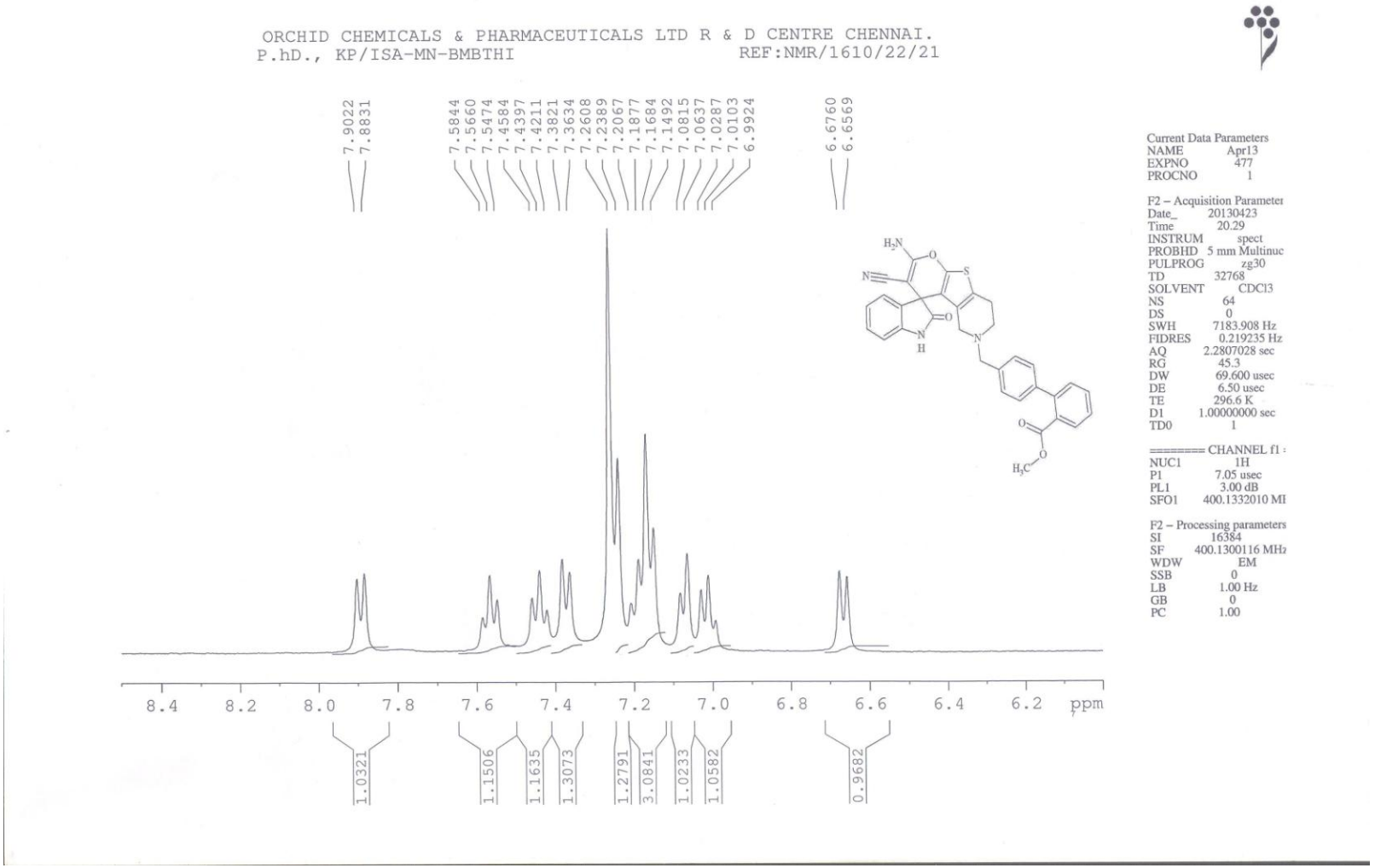
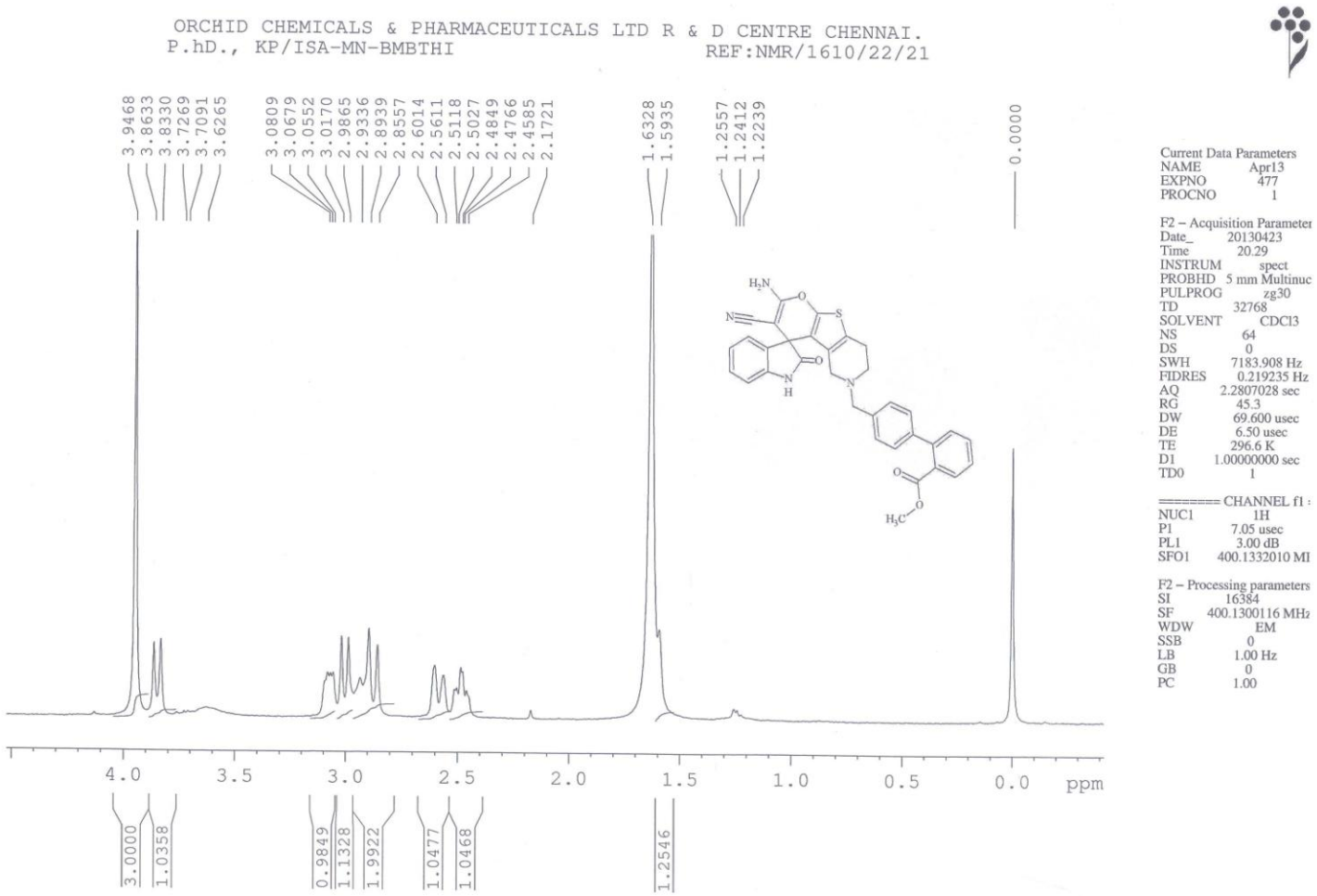
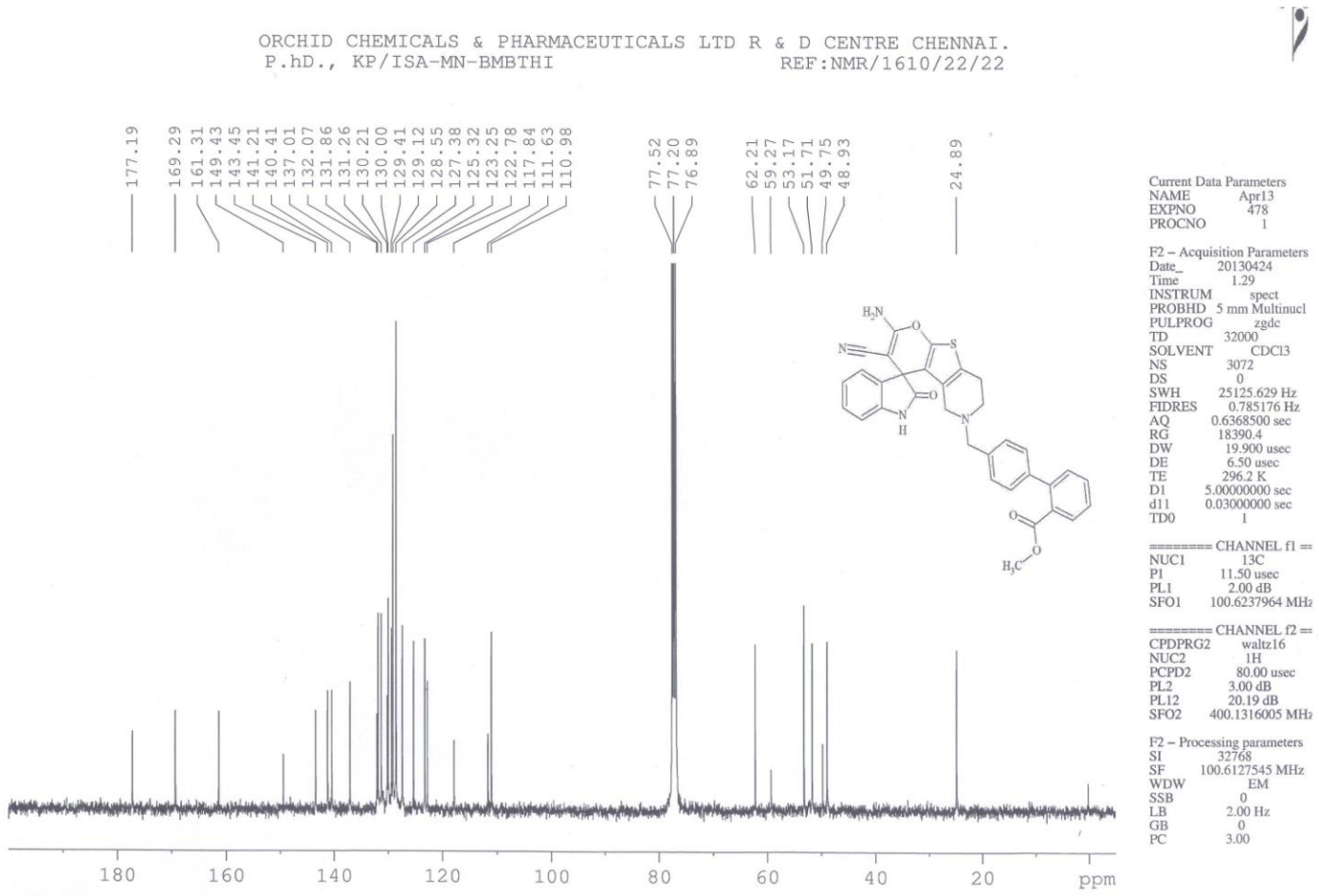


Expanded ^{1}H NMR spectrum of 5w



Expanded ^{1}H NMR spectrum of 5w



^{13}C NMR spectrum of 5w

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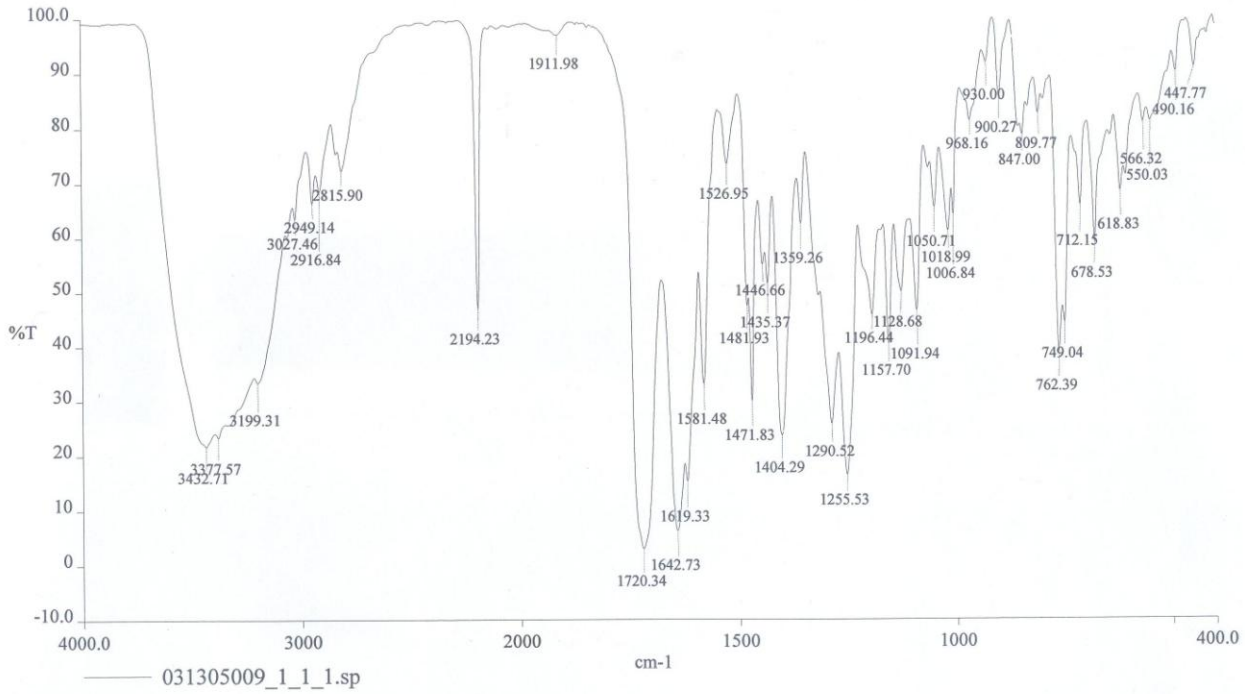

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Analyst:

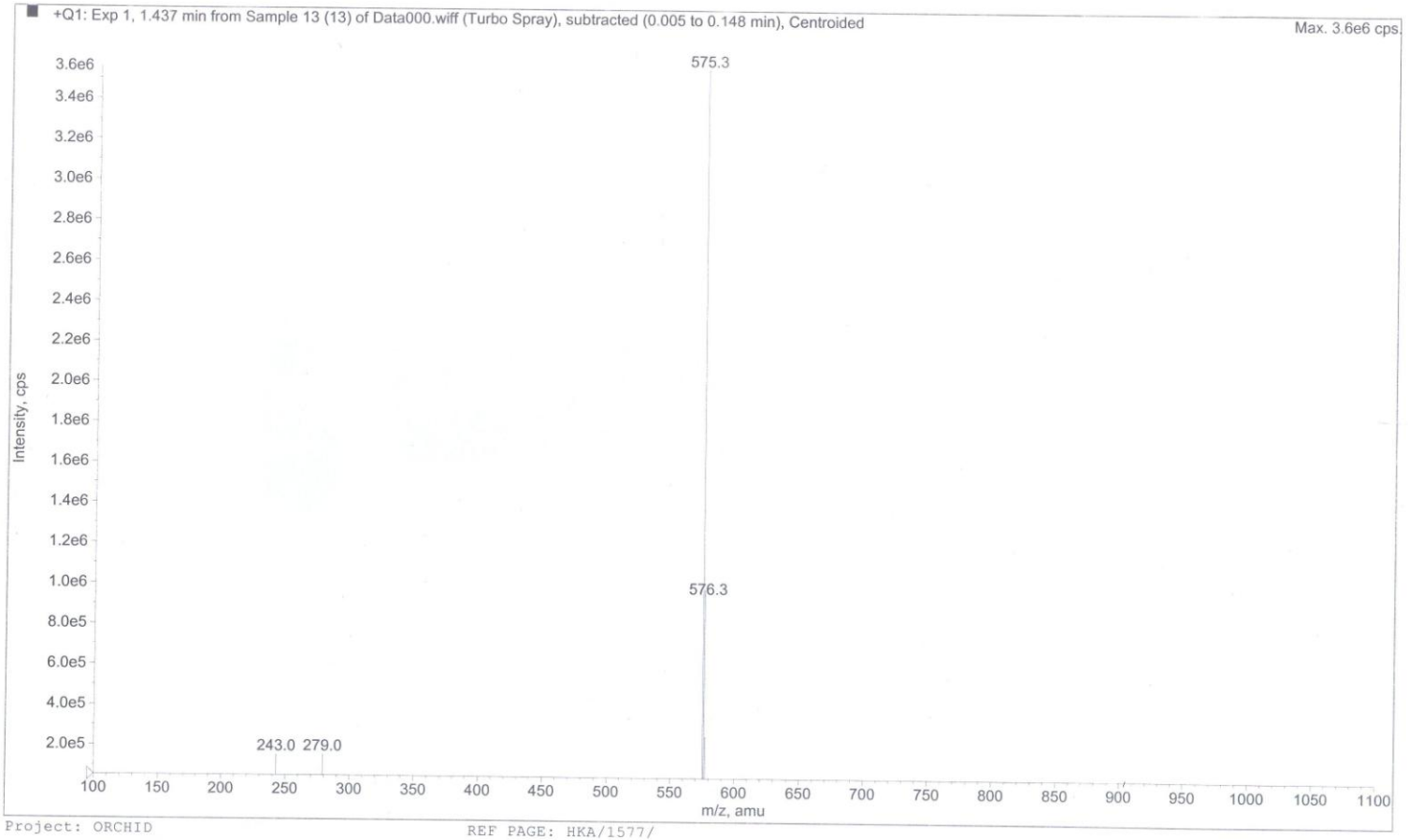
Technique:KBr/~~Neat~~/~~Nujol~~

Reference: IR/1559/111/9

IR spectrum of 5w

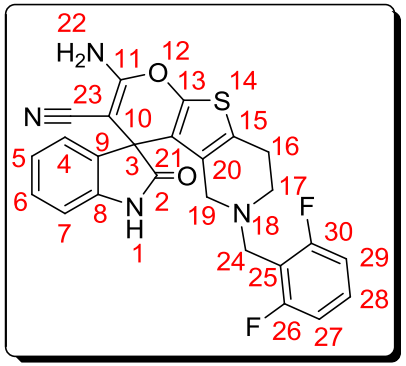
Acq. Date: Friday, May 24, 2013 ORCHID CHEMICALS AND PHARMACEUTICALS LTD., Printing Date: May 24, 2013

Acq. Time: 10:45 Sample Name: 13 Sample ID:



Project: ORCHID REF PAGE: HKA/1577/

Mass spectrum of 5w



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

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

Chemical Shift δ (ppm)	Multiplicity	Number of Hydrogen(s)	Coupling constant $^{2}J_{HH}$ & $^{3}J_{HH}$ (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 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.

^{13}C 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 ^{13}C-^{19}F coupling.

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

Empirical formula	$C_{52}H_{38}F_4N_8O_6S_2$
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; α = 66.206(2)^o; β = 88.780(2)^o; γ = 81.531(2)^o
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^o
Limiting indices	$-15 \leq h \leq 15$, $-15 \leq k \leq 15$, $-18 \leq l \leq 18$
Reflections collected /unique	26180/7948 [R(int) = 0.0367]
Completeness to θ = 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