Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Catalytic regioselective synthesis of pyrazole based pyrido[2,3-*d*]pyrimidine-diones and their biological evaluation

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Supporting information

1. Experimental part of biological evaluation

1.1 Instruments and Materials

All reactions were performed with commercially available reagents. They were used without further purification. The solvents used were of analytical grade. All reactions were monitored by thin-layer chromatography (TLC) on aluminum plates coated with silica gel 60 F_{254}, 0.25 mm thickness (Merck). Detection of the components was made by exposure to iodine vapors or UV light. Melting points were taken in melting point apparatus µThermoCal$_{10}$ (Analab Scientific Pvt. Ltd, India) and are uncorrected. The IR spectra were recorded in KBr on a Perkin-Elmer Spectrum GX FT-IR Spectrophotometer (Perkin-Elmer, USA) and only the characteristic peaks are reported in cm^{-1}. Mass spectra were recorded on Shimadzu LCMS 2010 spectrometer (Shimadzu, Tokyo, Japan) purchased under PURSE program of DST at Sardar Patel University, Vallabh Vidyanagar. The synthesized compounds were identified by 1H and 13C NMR spectra recorded in DMSO-d6 as a solvent on a Bruker Avance 400 MHz spectrometer (Bruker Scientific Corporation Ltd., Switzerland) using the residual solvent signal as an internal standard at 400 MHz and 100 MHz respectively. Chemical shifts are reported in parts per million (ppm). The elemental analysis was carried out by using Perkin-Elmer 2400 series-II elemental analyzer (Perkin-Elmer, USA) and all compounds are within ± 0.4% of the theoretical compositions. Ampicillin, griseofulvin, isoniazid and nystatin were purchased from local market.

1.2. In vitro antimicrobial assay

The *in vitro* antimicrobial activity of all synthesized pyrazole based pyrido [2,3- *d*]pyrimidinones derivatives **4a-r** was carried out by broth micro dilution method. Mueller – Hinton broth was used as nutrient medium to grow and dilute the compound suspension for the test bacteria. Sabouraud Dextrose broth was used for fungal nutrition. 2% DMSO in water was used as the diluent to get desired concentration of compounds to test upon standard bacterial strains. Inoculum size for test strain was adjusted to 10^8 CFU mL^{-1} by comparing the turbidity. Serial dilutions were prepared in primary and secondary screening. Each synthesized compound and the standard drugs were diluted obtaining 2000 µg/mL concentration as a stock solution. The drugs which were found to be active in primary screening (i.e. 500, 250 and 200 µg/mL concentrations) were further screened in their second set of dilution at 100, 50, 25 and 12.5 µg/mL concentration against all microorganisms. 10 micro liter suspensions were further inoculated on appropriate media and growth was noted after 1 and 2 days. The control tube containing no antibiotic was instantaneously sub cultured (before inoculation) by spreading a loopful evenly over an area of plate of medium suitable for the growth of the test organism. The tubes were then put for incubation at 37°C overnight. The highest dilution preventing appearance

of turbidity after spot subculture was considered as minimal inhibitory concentration (MIC, µ/L). All the tubes showing no visible growth (same as control tube) were subcultured and incubated overnight at 37 °C. The amount of growth from the control tube before incubation was compared. In this study Ampicillin, Norfloxacin and Chloramphenicol were used as the standard antibacterial drugs. Nystatin and Griseofulvin were used as standard antifungal drugs. The results are summarized in **Table 4**.

1.3. In vitro antituberculosis assay

The antitubercular activity of all synthesized compound against *Mycobacterium tuberculosis* H37Rv was performed by Lowensteine-Jensen method [1] with minor modification where 250 µg/mL and 100 µg/mL dilution of each compound was added to Lowensteine-Jensen medium and then media was uncontaminated by inspissation method. A culture of *Mycobacterium tuberculosis* H37Rv growing on Lowensteine-Jensen medium was harvested in 0.85% saline in bijou bottle. The stock solutions of the all compounds were prepared in 2% DMSO IN WATER i.e. 250 µg/mL and 100 µg/mL. These tubes were then incubated at 37 °C for 24 h followed by streaking of *Mycobacterium tuberculosis* H37Rv (5 _ 104 bacilli per tube). The growth of bacilli was seen after two weeks, three weeks and finally after four weeks of incubation. The tubes having the compounds were compared with control tubes where medium alone was incubated with *Mycobacterium tuberculosis* H37Rv. The concentration at which complete inhibition of colonies occurred was taken as active concentration of the tested compound. The standard strain *Mycobacterium tuberculosis* H37Rv was tested with known drug isoniazid and rifampicin for comparison purpose. The results are summarized in **Table 5**.

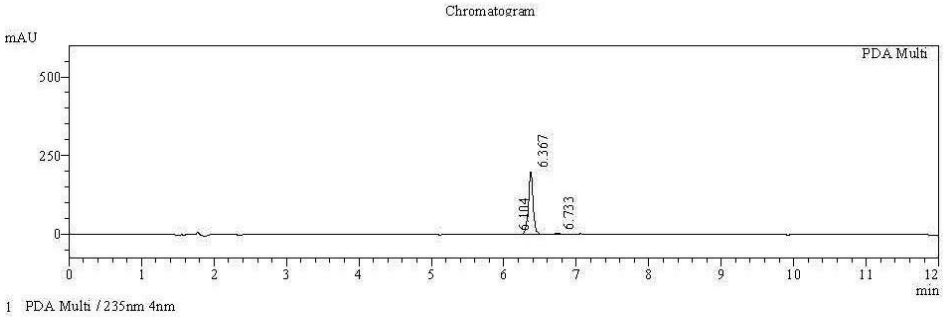
1.4. In vitro antimalarial assay

All the synthesized compounds **4a-r** were screened for their antimalarial activity against the *P. falciparum* strain. The *P. falciparum* strain was acquired from Shree R. B Shah Mahavir Super-speciality hospital, Surat, Gujarat, India, and was used in the vitro tests. The *P. falciparum* strains were cultivated by a modified method described by Trager and Jensen [2]. Compounds were dissolved in 2% DMSO in water. The final concentration of DMSO used was not toxic and did not interfere with the assay. The antiparasitic effect of the compounds was measured by growth inhibition percentage as described by Carvalho and Krettli [3]. For experimental purposes, the cultures were synchronized with 5% D-sorbitol when the parasites were in the ring stage [4]. The parasite suspension, consisting of predominately the ring stage, was adjusted to a 1-2 % parasitaemia and 2.5 % haematocrit in hypoxanthine-free RPMI-1640 culture medium with 10% human plasma and was exposed to 7 concentrations of each compound for a single cycle of parasite growth of 48 h at 37 °C. A positive control with reference to antimalarial drugs

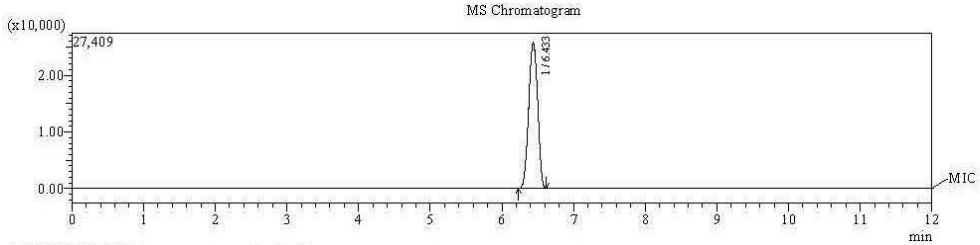
in standard concentrations was used in each experiment. The stock solutions were additionally diluted in whole medium (RPMI 1640 plus 10% human serum) to each of the used concentrations. The concentration that inhibited 50% of parasite growth (IC_{50} value) was determined by interpolation using Microcal Origin software. The standard drugs chloroquine and quinine were used as the reference antimalarial agents, blood smears were read blind and each duplicate experiment was repeated three times. The results are summarized in **Table 6**.

LC-MS of compound **4a**

Acquired by : Admin
Sample ID : 16
Vial# : 16
Injection Volume : 10
Data File : JAG-1-1-2-12.lcd
Method file : X-BRIDGE_150_12min_AA-FA.lcm



1 PDA Multi / 235nm 4nm



PDA Ch2 235nm 4nm PeakTable

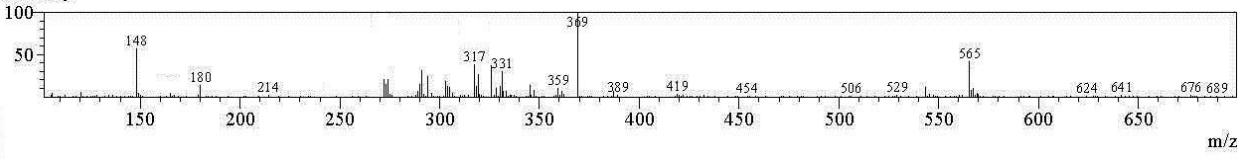
Peak#	Ret. Time	Area	Height	Area %
1	6.104	3962	826	0.442
2	6.367	879809	199559	98.143
3	6.733	12681	2990	1.415
Total		896452	203375	100.000

MSTABLE MIC1

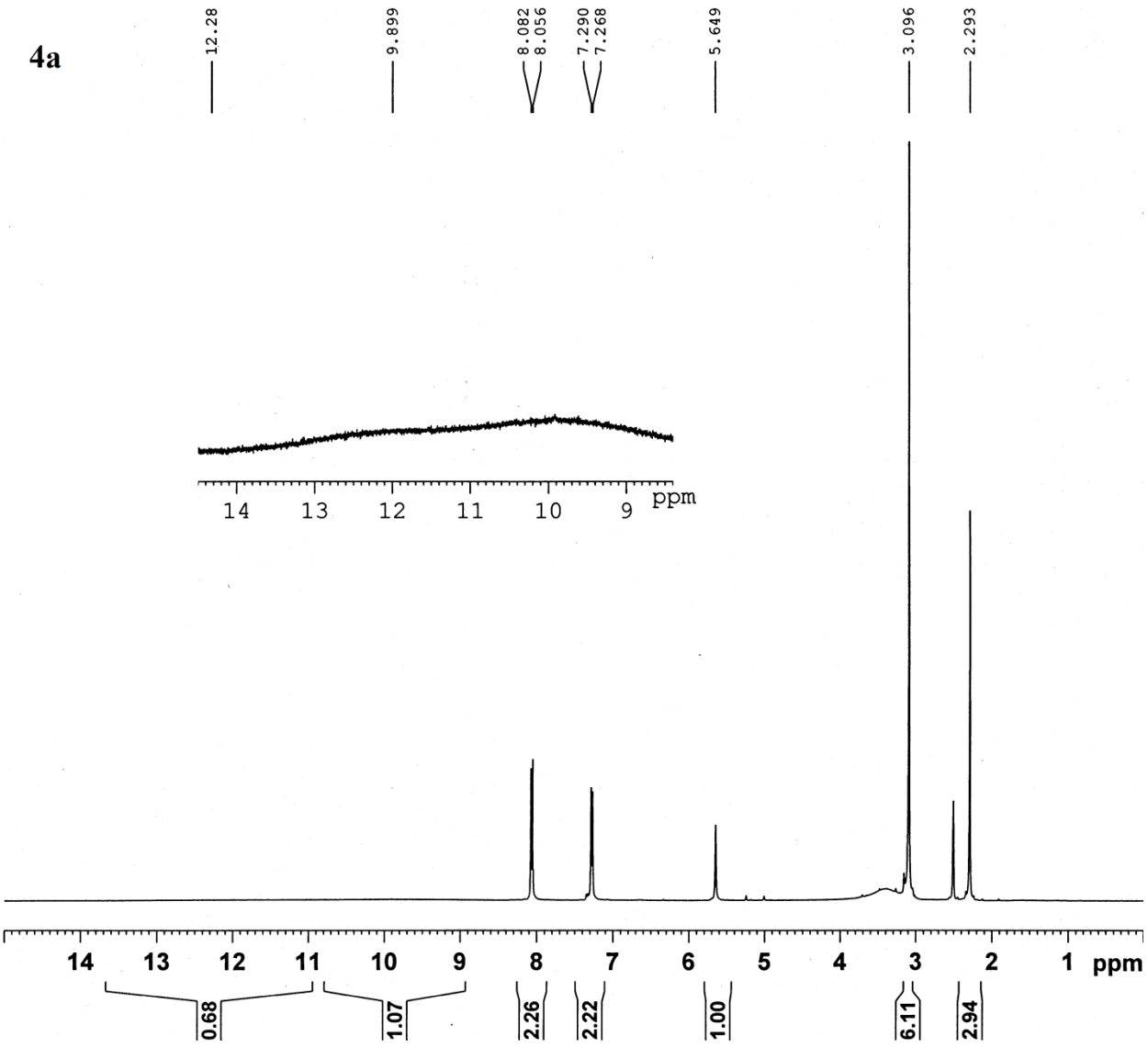
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	6.433	369.20	224750	100.00	1-1
Total			224750	100.00	

MS Spectrum Graph

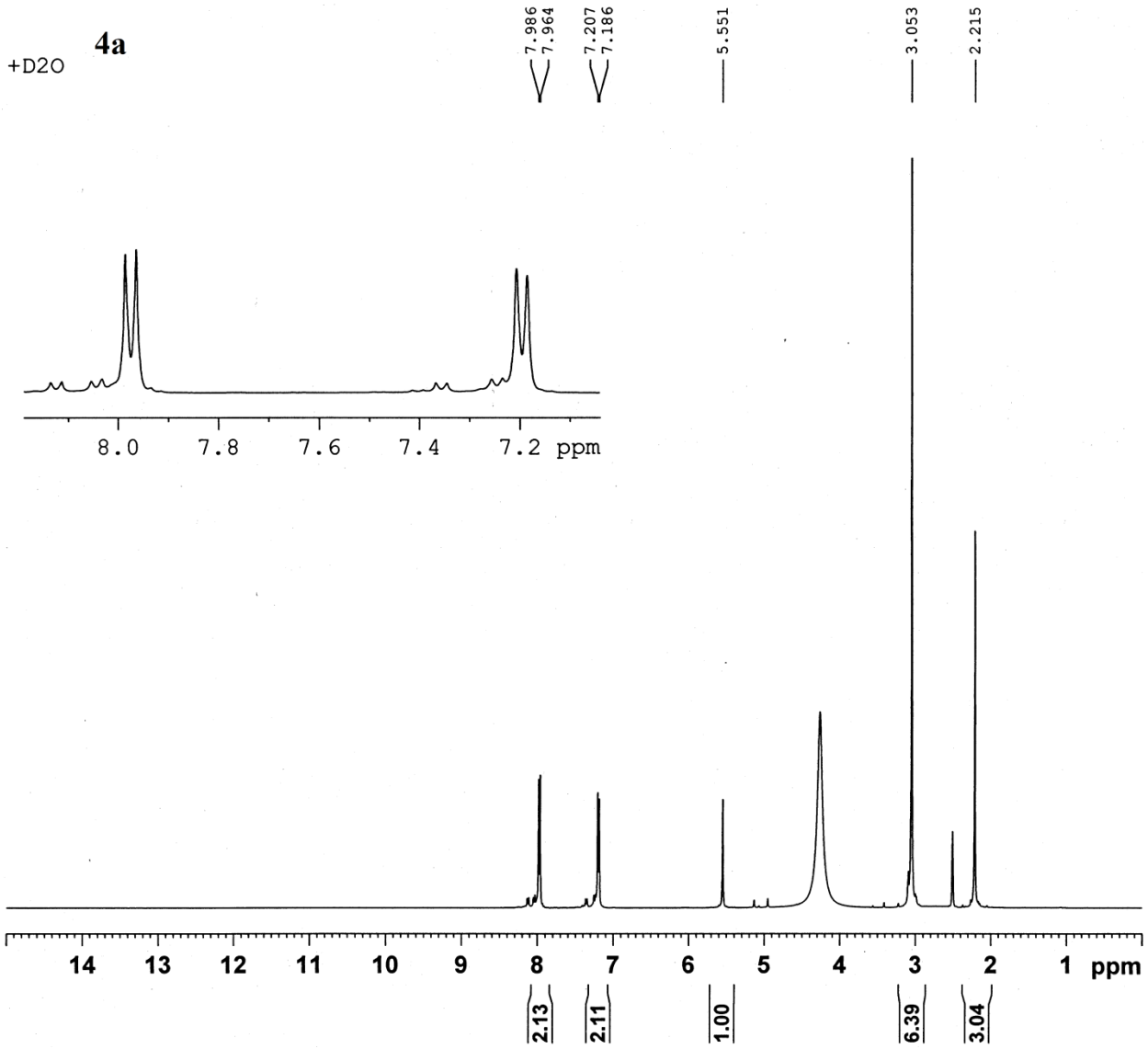
#:1 Ret.Time:Averaged 6.417-6.450(Scan#:386-388)
BG Mode:Calc 6.233<->6.617(375<->398)
Base Peak:369.20(41512) Polarity:Pos Segment1 - Event1
Intensity



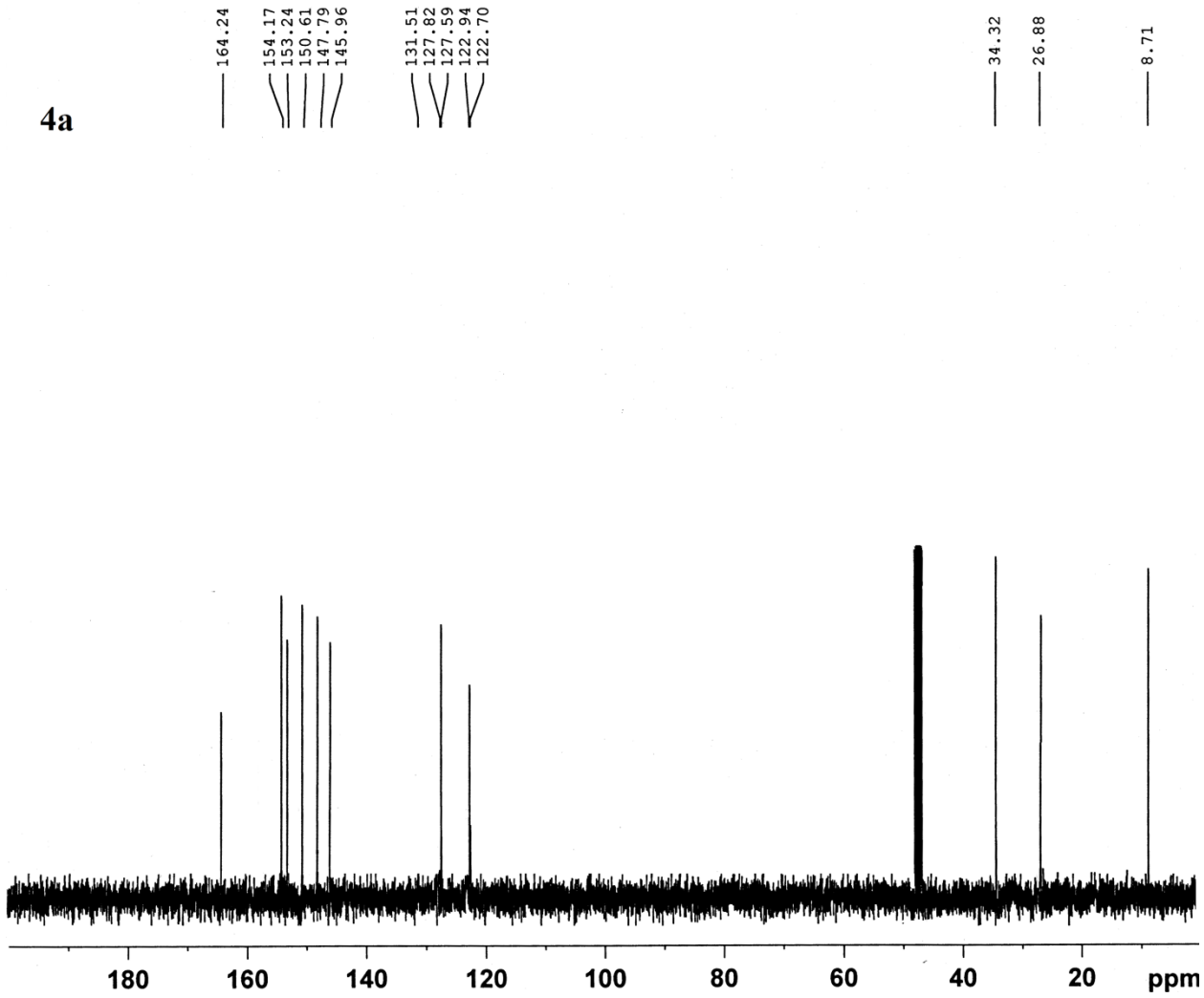
^{1}HNMR of compound **4a**



^{1}HNMR of compound **4a** (DMSO+D_2O)

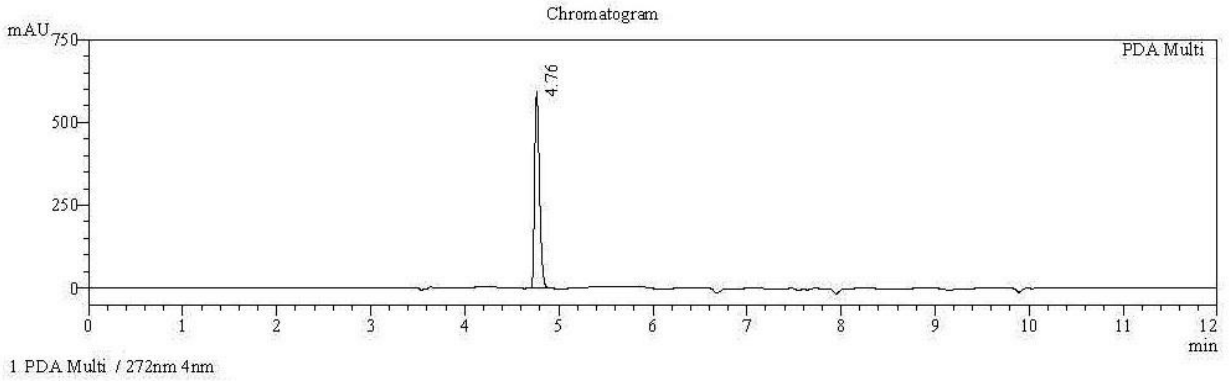


^{13}CNMR of compound **4a**

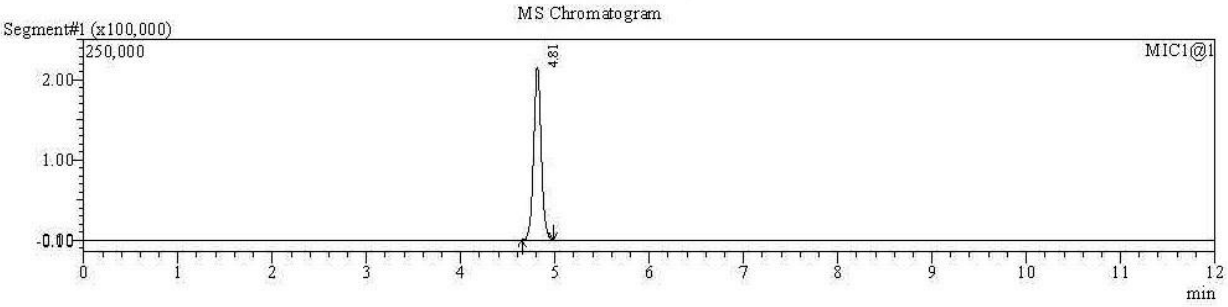


LC-MS of compound **4b**

Acquired by	: Admin
Sample ID	: 2
Vial#	: 2
Injection Volume	: 10
Data File	: VTX-622-D-43-FINAL-AP--4.lcd
Method file	: GENESIS-LCMS-ESI-150.lcm



1 PDA Multi / 272nm 4nm



PeakTable

PDA Ch1 272nm 4nm

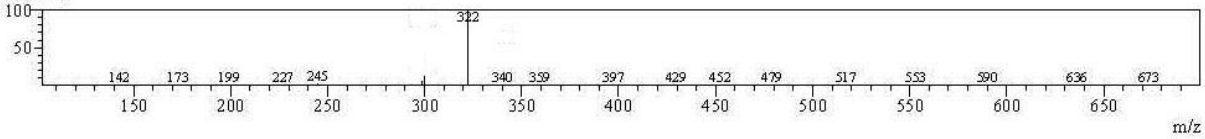
Peak#	Ret. Time	Area	Height	Area %
1	4.76	2126211	591590	100.00
Total		2126211	591590	100.00

MSTABLE MIC1

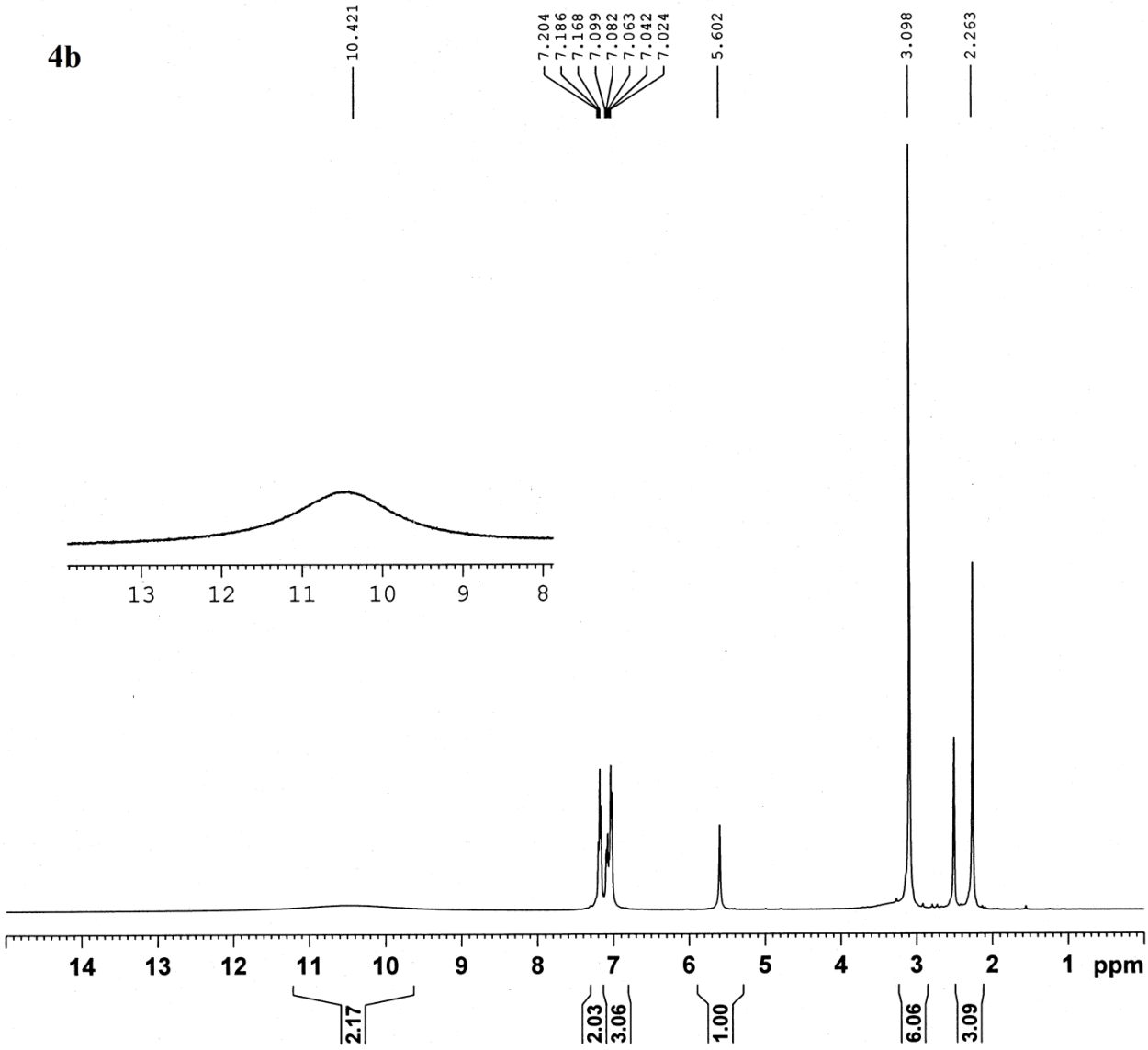
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	4.81	322.20	1203267	100.00	1-1
Total			1203267	100.00	

MS Spectrum Graph

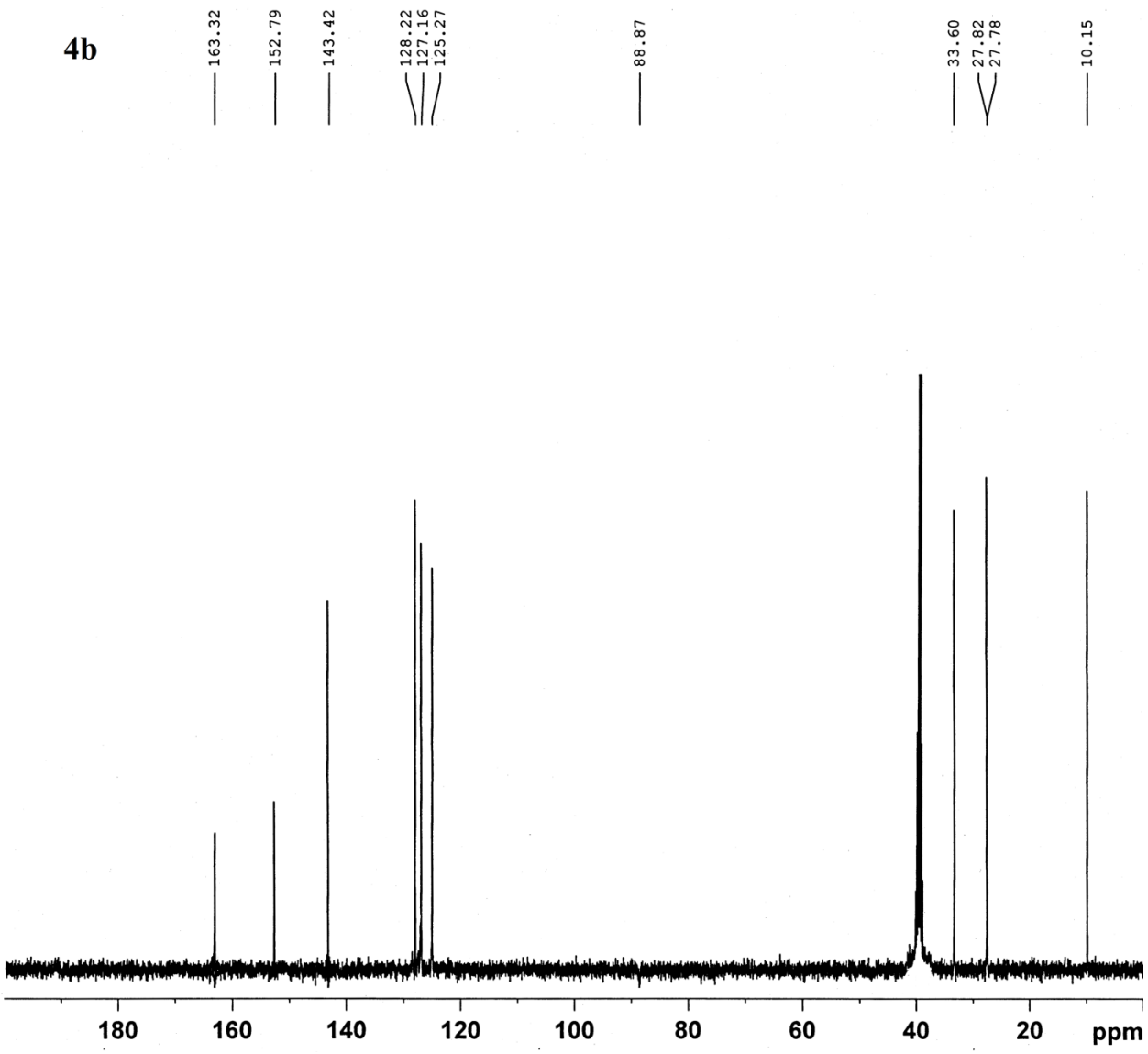
#1 Ret.Time:Averaged 4.783-4.817(Scan#:288-290)
BG Mode:Calc 4.650<->4.983(280<->300)
Base Peak:322.20(514975) Polarity:Pos Segment1 - Event1
Intensity



^{1}HNMR of compound **4b**

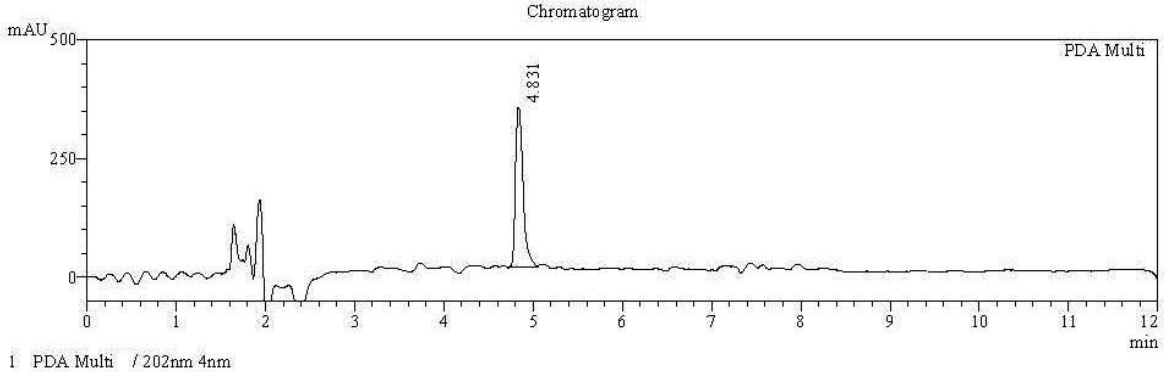


^{13}CNMR of compound **4b**

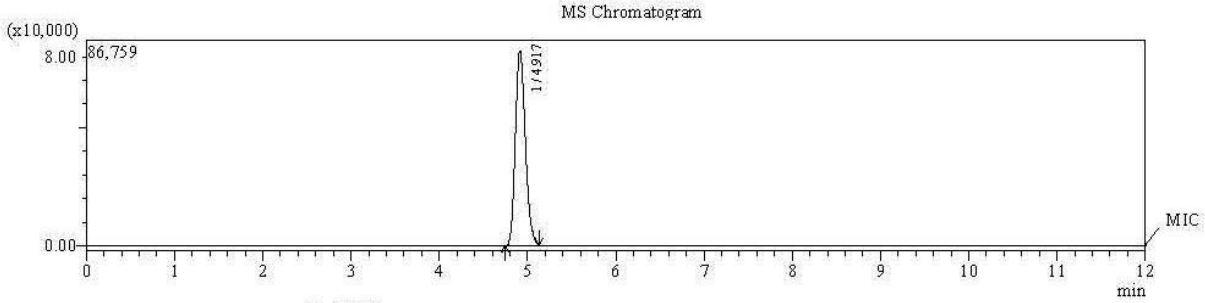


LC-MS of compound **4c**

Acquired by	: Admin
Sample ID	: 19
Vial#	: 19
Injection Volume	: 20
Data File	: JAG-09-AA-52-PURE_2.lcd
Method file	: X-BRIDGE_150_12min_AA-FA.lcm



1 PDA Multi / 202nm 4nm



PeakTable

PDA Ch1 202nm 4nm

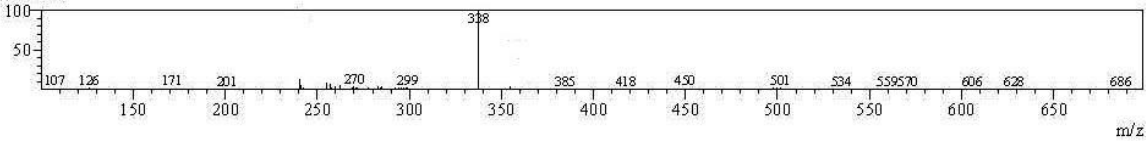
Peak#	Ret. Time	Area	Height	Area %
1	4.831	1980348	336766	100.000
Total		1980348	336766	100.000

MSTABLE MIC1

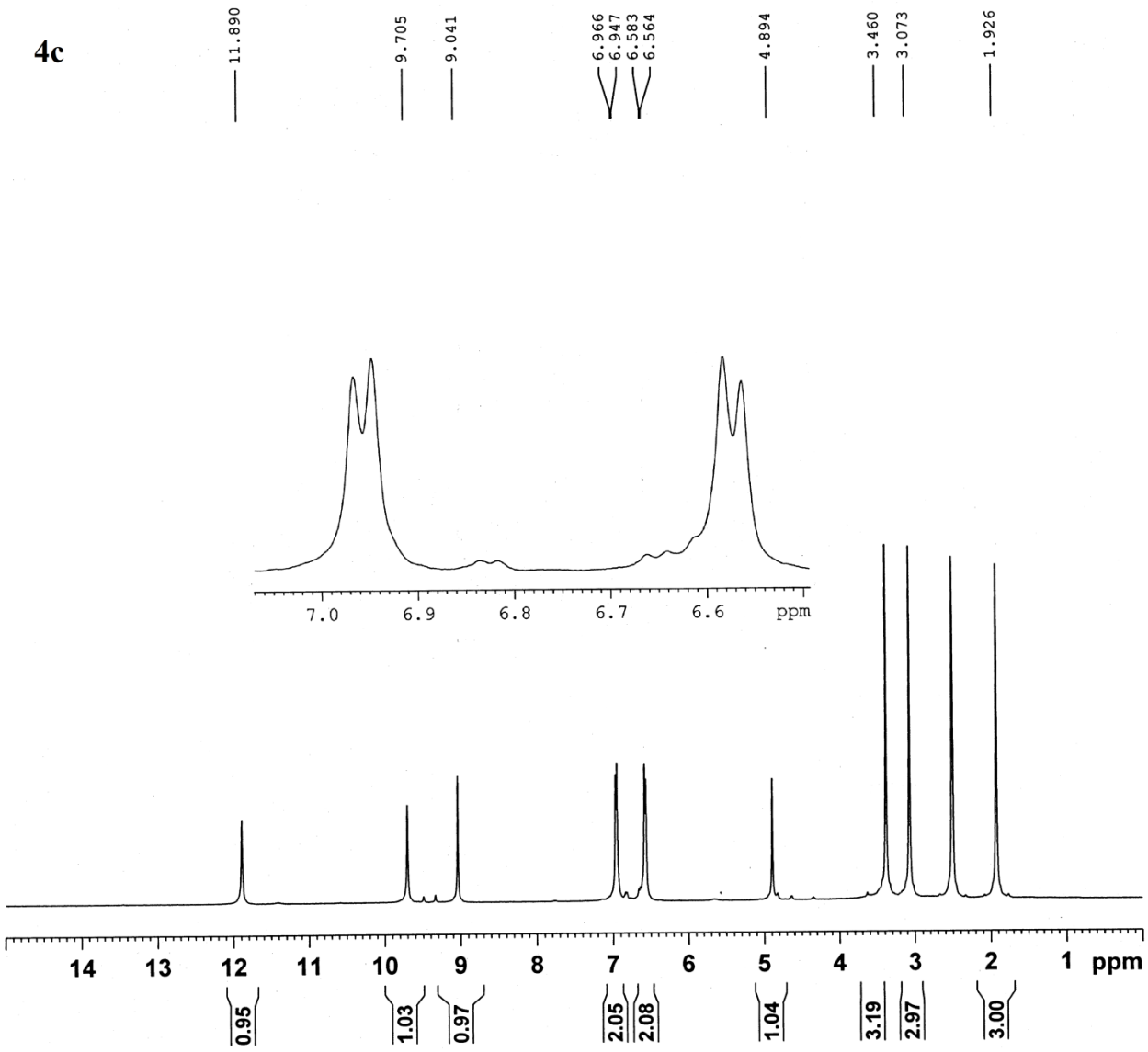
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	4.917	338.15	647559	100.00	1-1
Total			647559	100.00	

MS Spectrum Graph

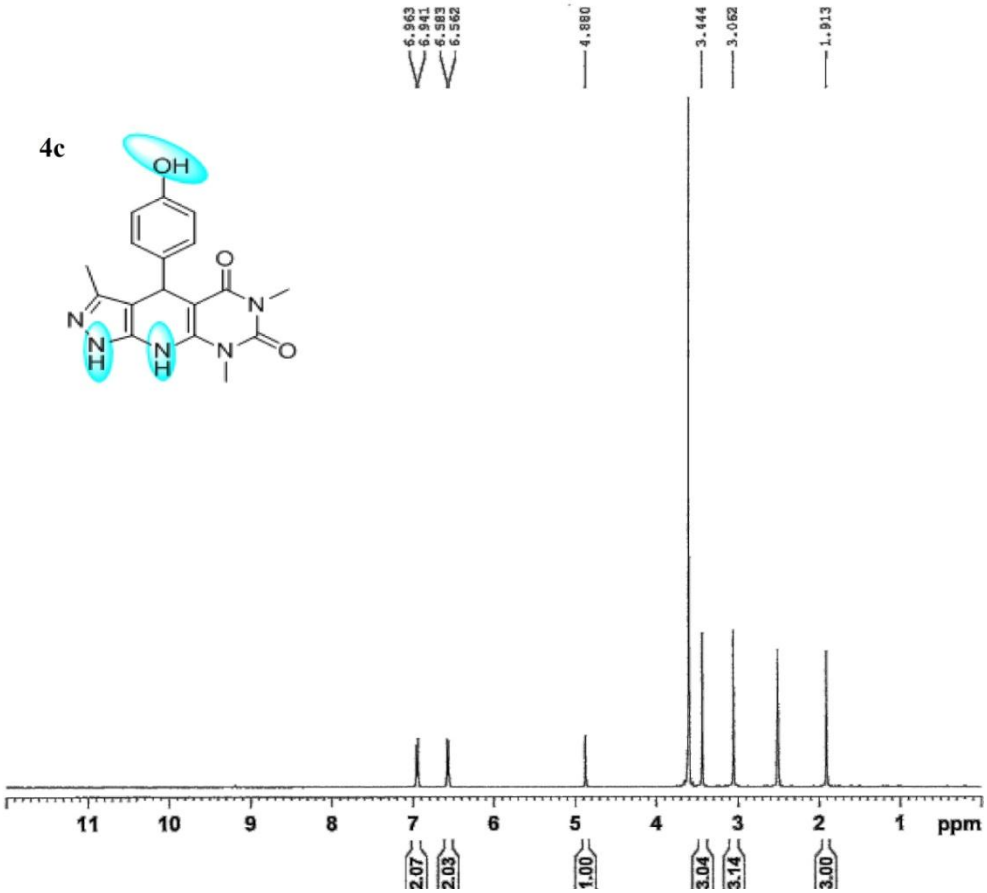
#1 Ret.Time:Averaged 4.900-4.933(Scan# 295-297)
BG Mode:Calc 4.750<->5.133(286<->309)
Base Peak:338.15(106016) Polarity:Pos Segment1 - Event1



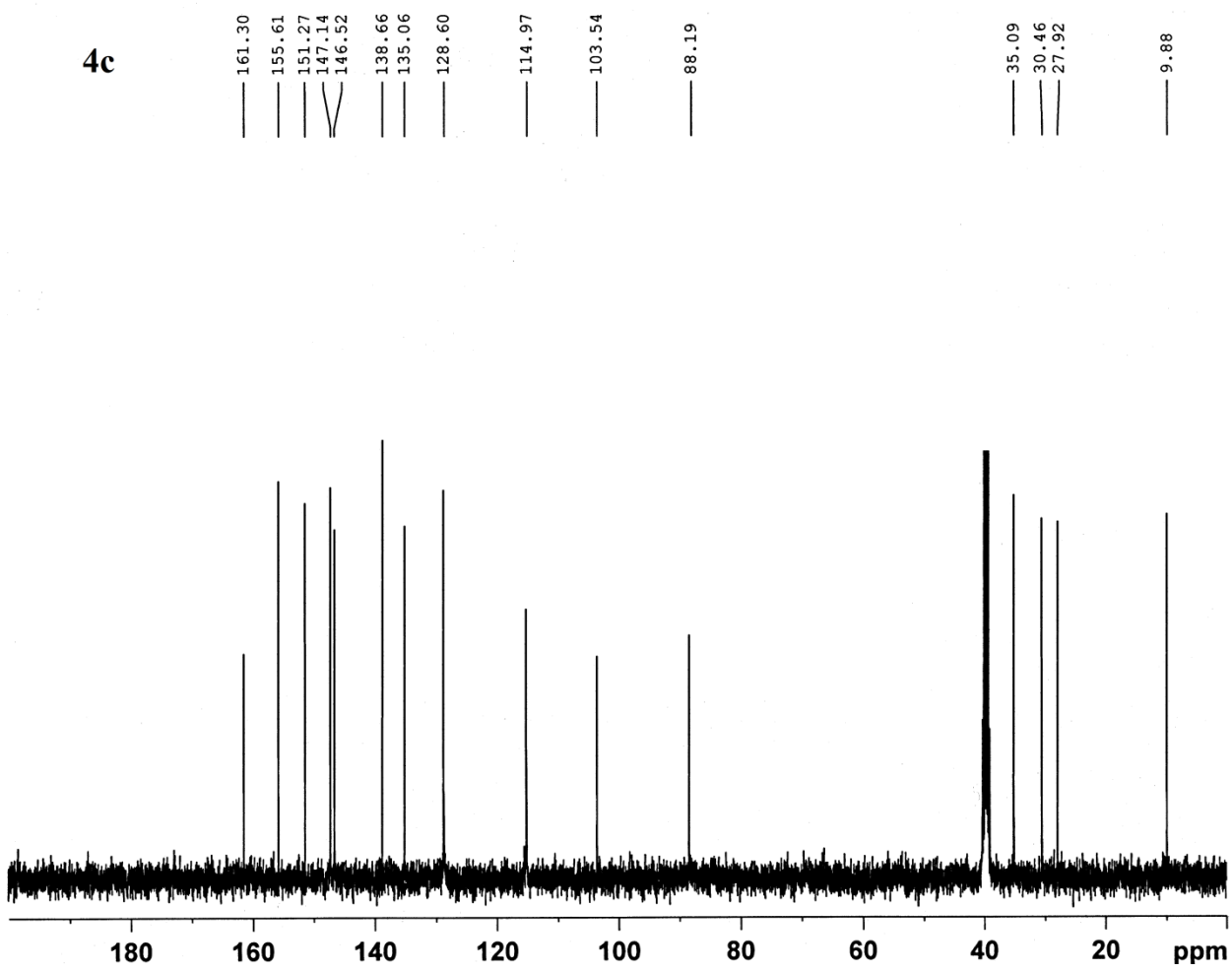
^{1}HNMR of compound **4c**



^{1}HNMR of compound **4c** (DMSO+D_2O)



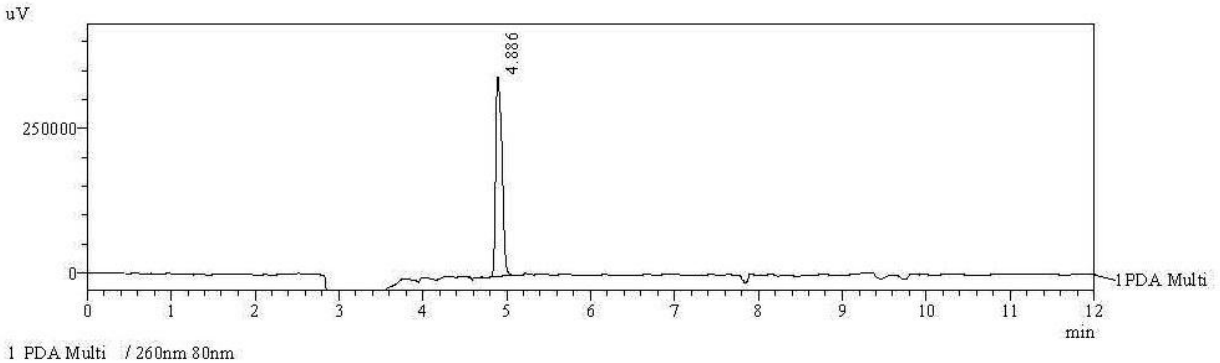
^{13}CNMR of compound **4c**



LC-MS of compound **4d**

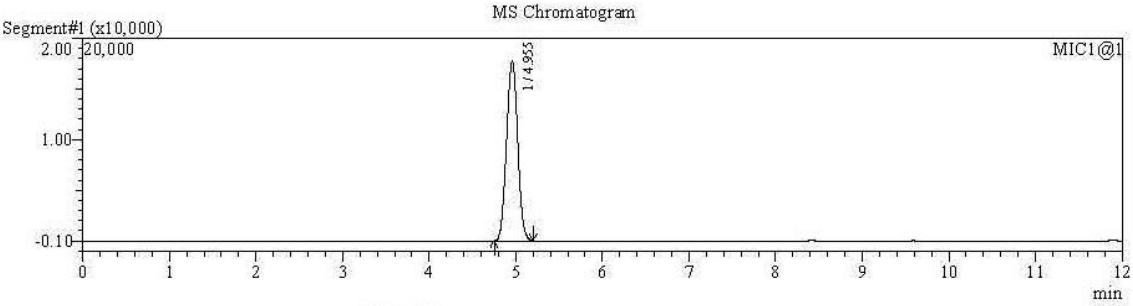
Acquired by : Admin
Sample ID : 1
Vial# : 17
Injection Volume : 6
Data File : BDX-615-E-46-FINAL001.lcd
Method file : GENESIS-LCMS-ESI-150.lcm

Chromatogram



1 PDA Multi / 260nm 80nm

MS Chromatogram



PeakTable

PDA Ch2 260nm 80nm

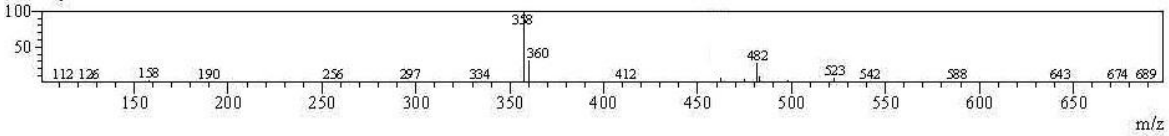
Peak#	Ret. Time	Area	Height	Area %
1	4.886	1796147	344704	100.000
Total		1796147	344704	100.000

MSTABLE MIC1

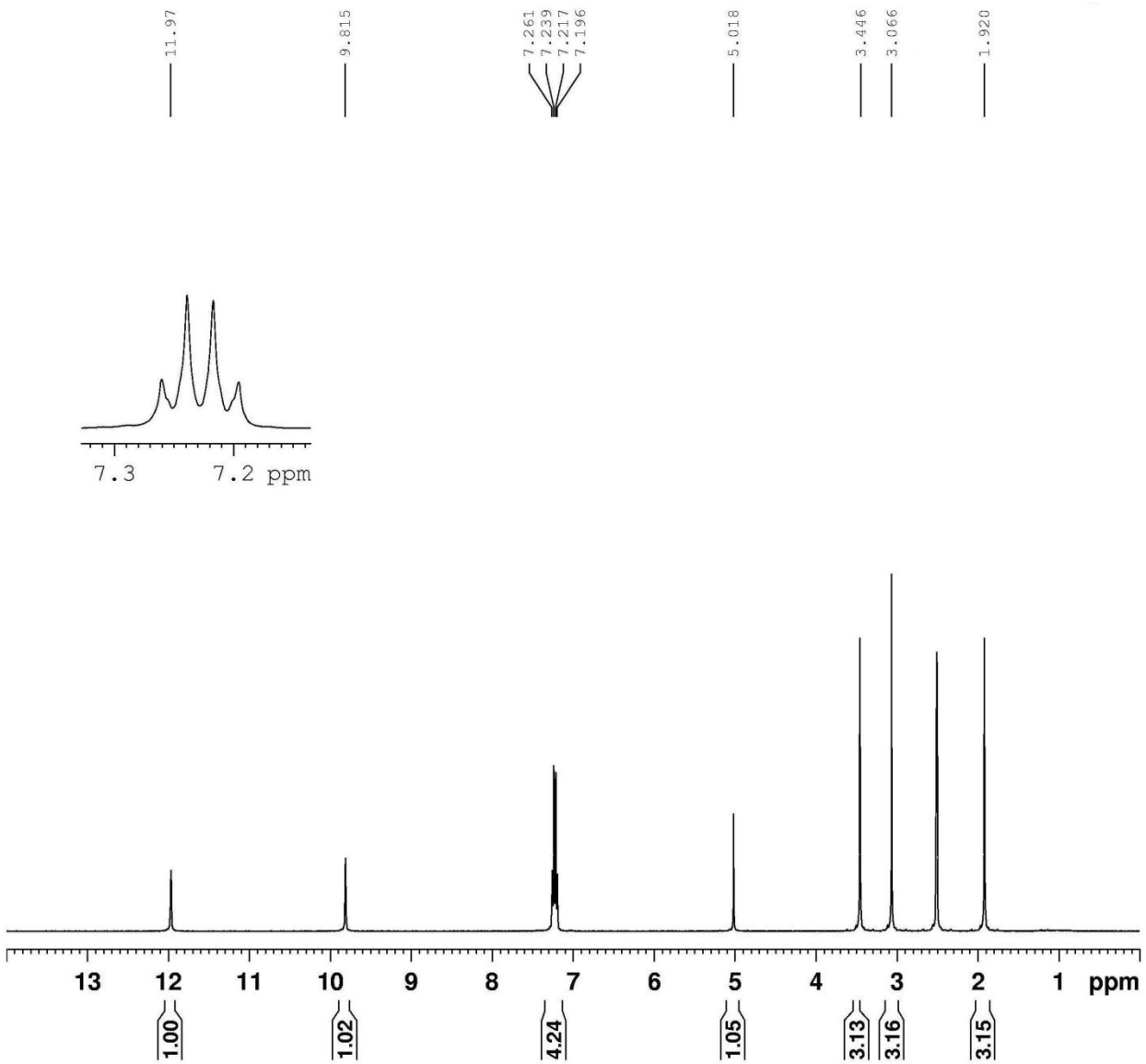
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	4.955	358.54	159085	100.00	1-1
Total			159085	100.00	

MS Spectrum Graph

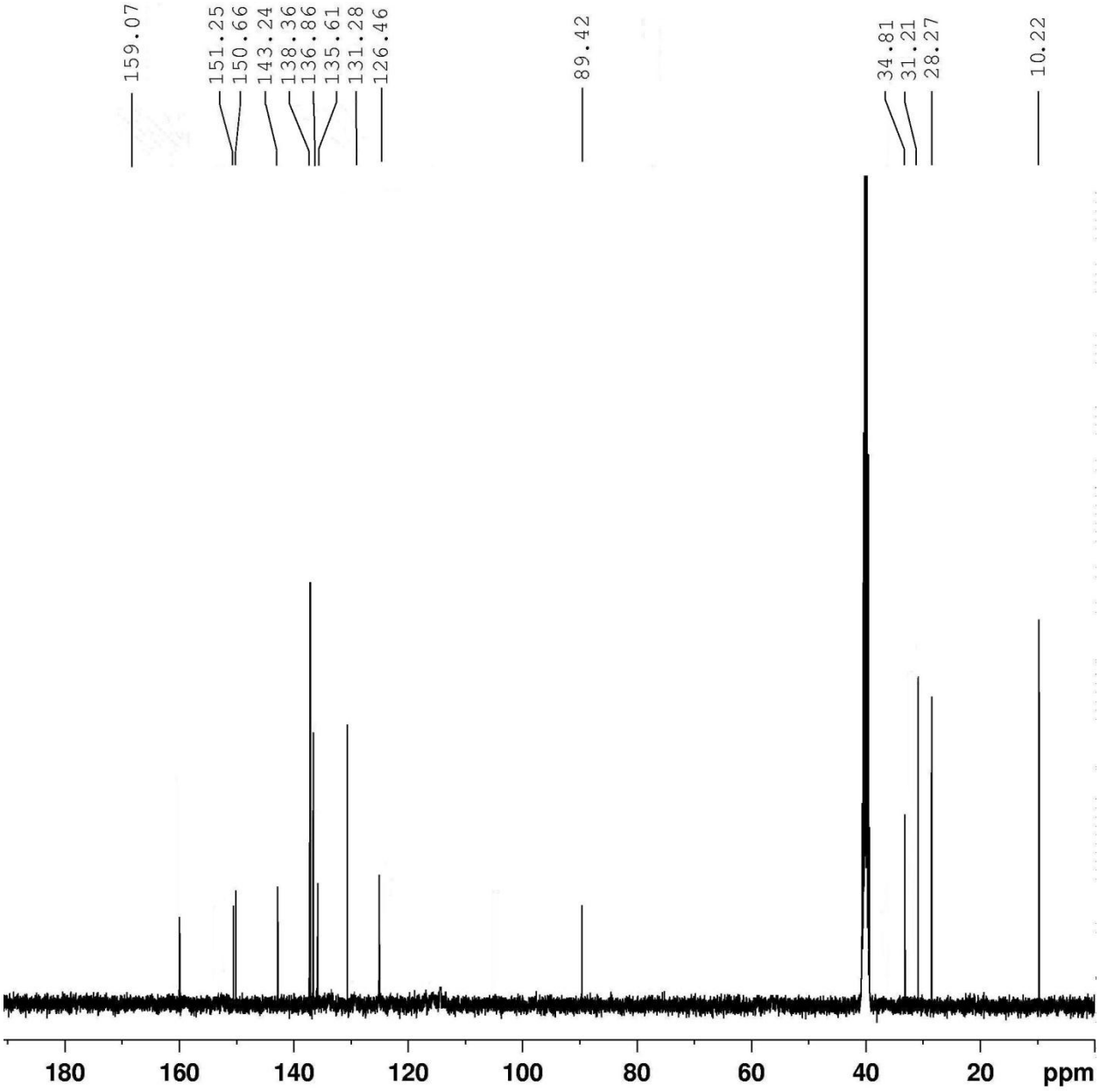
#:1 Ret.Time:Averaged 4.933-4.967(Scan#:297-299)
BG Mode:Calc 4.750<->5.200(286<->313)
Base Peak:358.54(28330) Polarity:Pos Segment1 - Event1



^{1}HNMR of compound **4d**



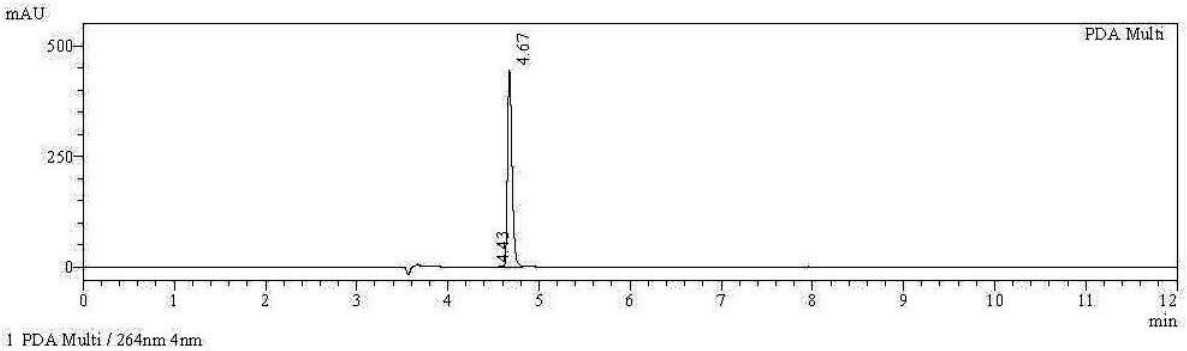
^{13}CNMR of compound **4d**



LC-MS of compound **4e**

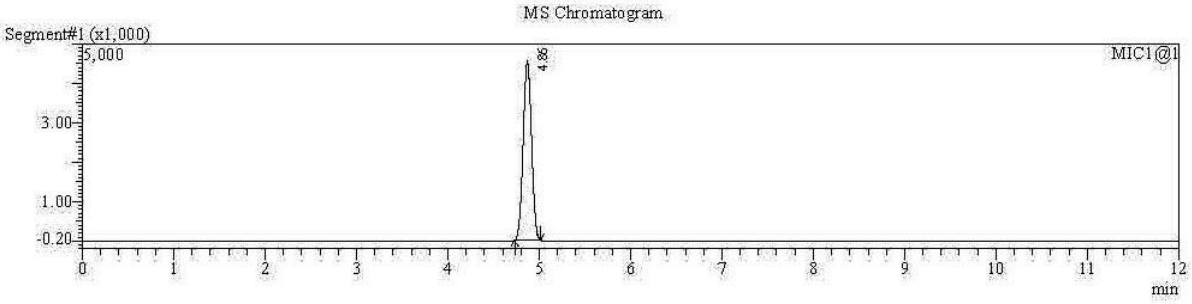
Acquired by	: Admin
Sample ID	: 4
Vial#	: 4
Injection Volume	: 10
Data File	: BDX-615-E-74-FINAL1.1cd
Method file	: LCMS-ES1.lcm

Chromatogram



1 PDA Multi / 264nm 4nm

MS Chromatogram



PeakTable

PDA Ch1 264nm 4nm

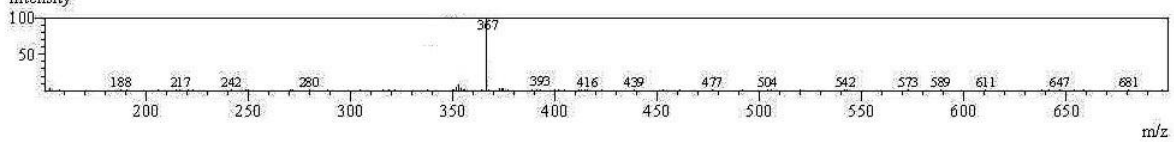
Peak#	Ret. Time	Area	Height	Area %
1	4.43	1638	398	0.11
2	4.67	1508689	445607	99.89
Total		1510327	446005	100.00

MSTABLE MIC1

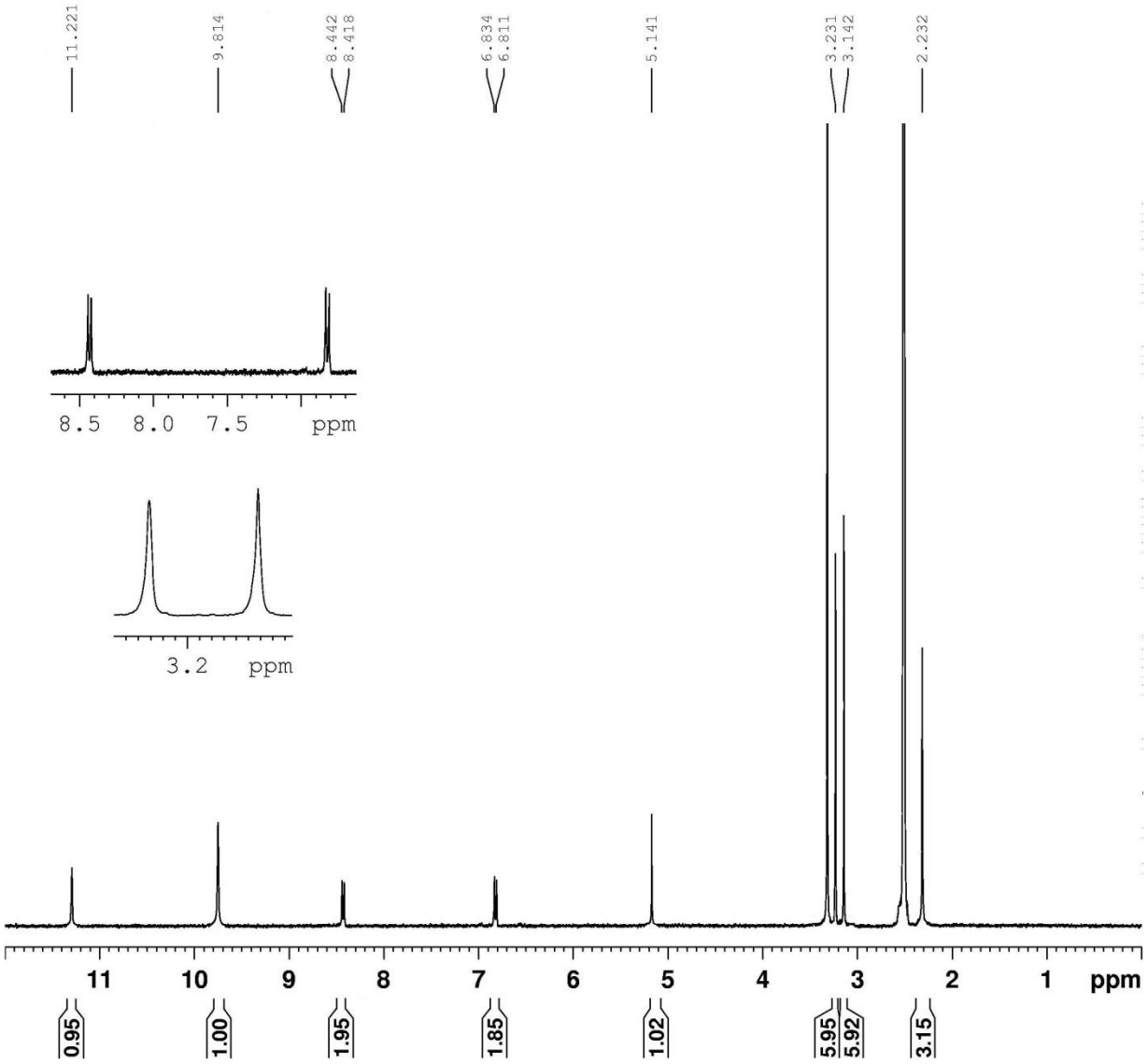
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	4.86	367.10	28856	100.00	1-1
Total			28856	100.00	

MS Spectrum Graph

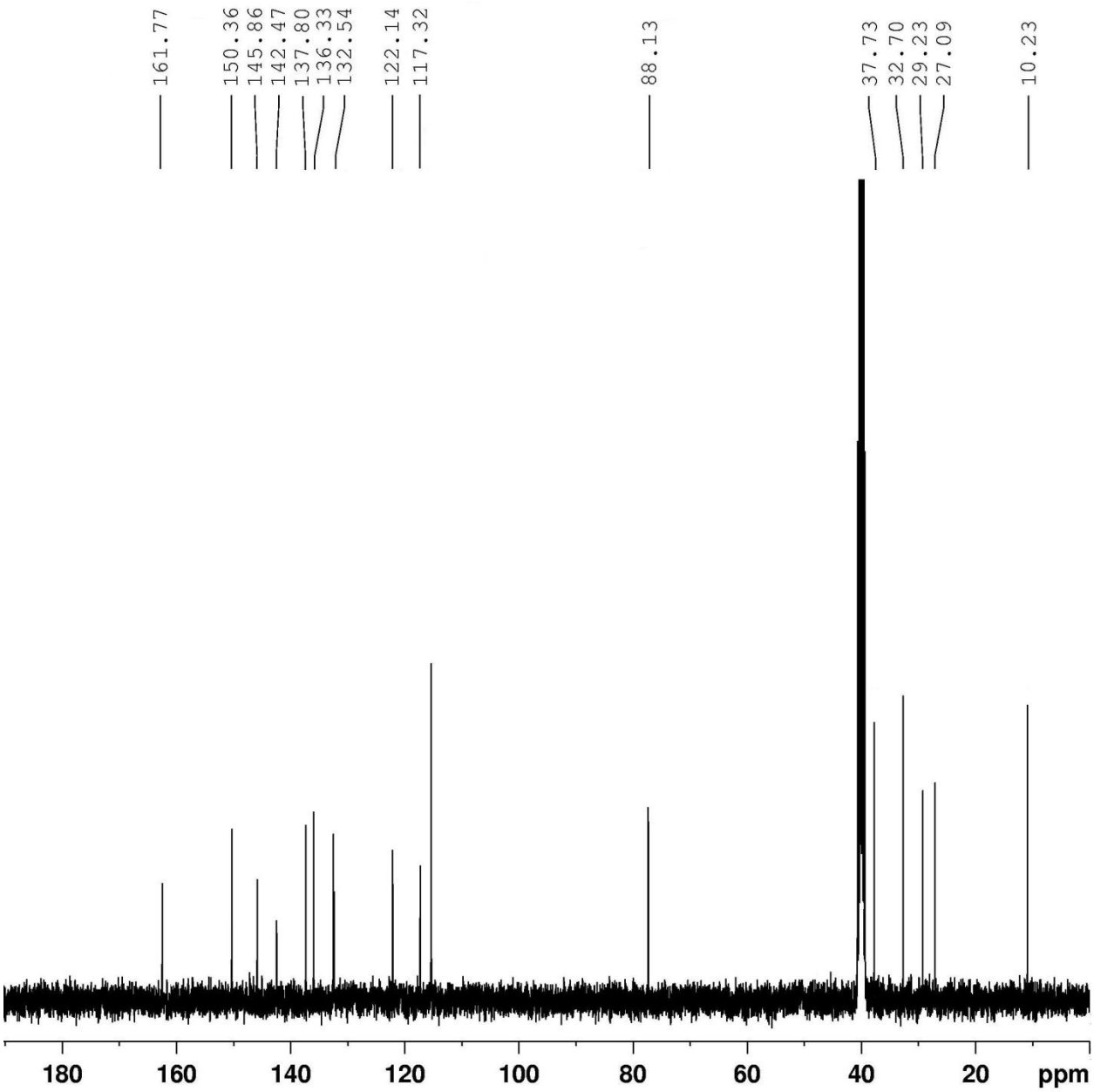
#:1 Ret.Time:Averaged 4.850-4.883(Scan#:292-294)
BG Mode:Calc 4.733<->5.017(285<->302)
Base Peak:367.10 (13276) Polarity:Pos Segment1 - Event1



^{1}HNMR of compound **4e**



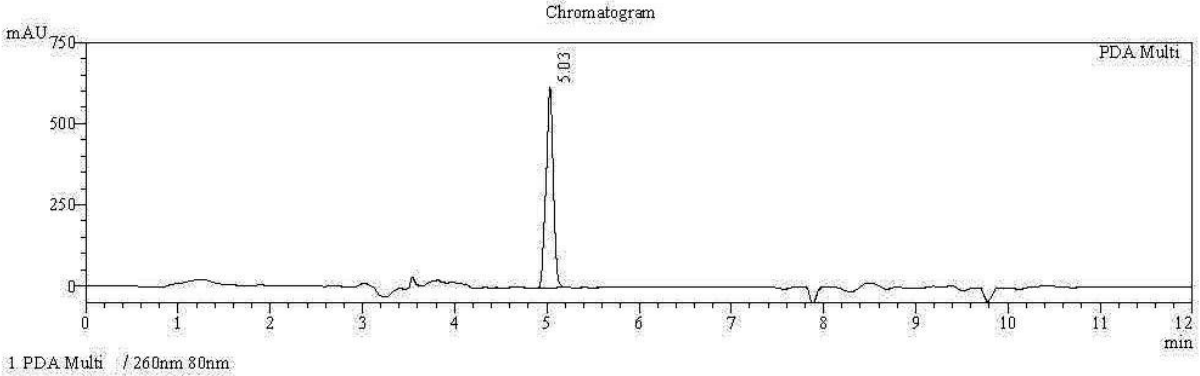
^{13}CNMR of compound **4e**



LC-MS of compound **4f**

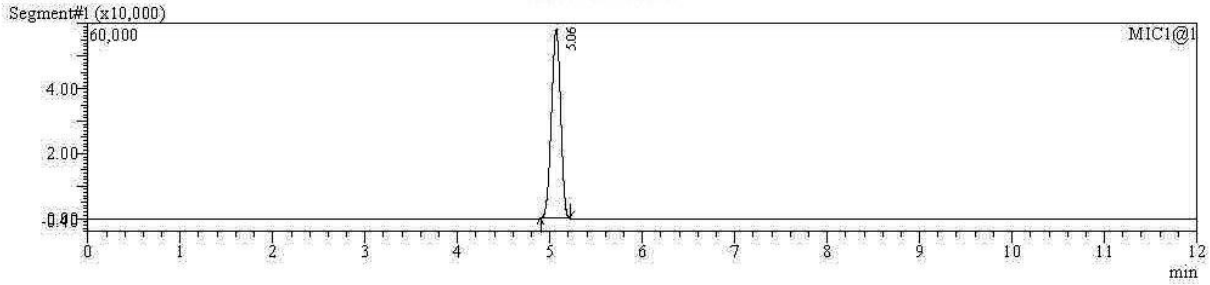
Acquired by	: Admin
Sample ID	: 6
Vial#	: 6
Injection Volume	: 50
Data File	: APX-620-F16-FINAL2.lcd
Method file	: GENESIS-LCMS-ESI-150.lcm

Chromatogram



1 PDA Multi / 260nm 80nm

MS Chromatogram



PeakTable

PDA Ch2 260nm 80nm

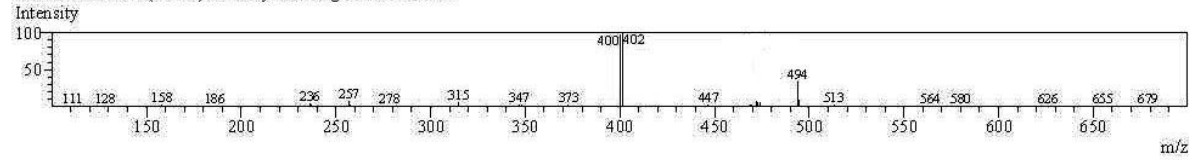
Peak#	Ret. Time	Area	Height	Area %
1	5.03	3356674	615964	100.00
Total		3356674	615964	100.00

MSTABLE MIC1

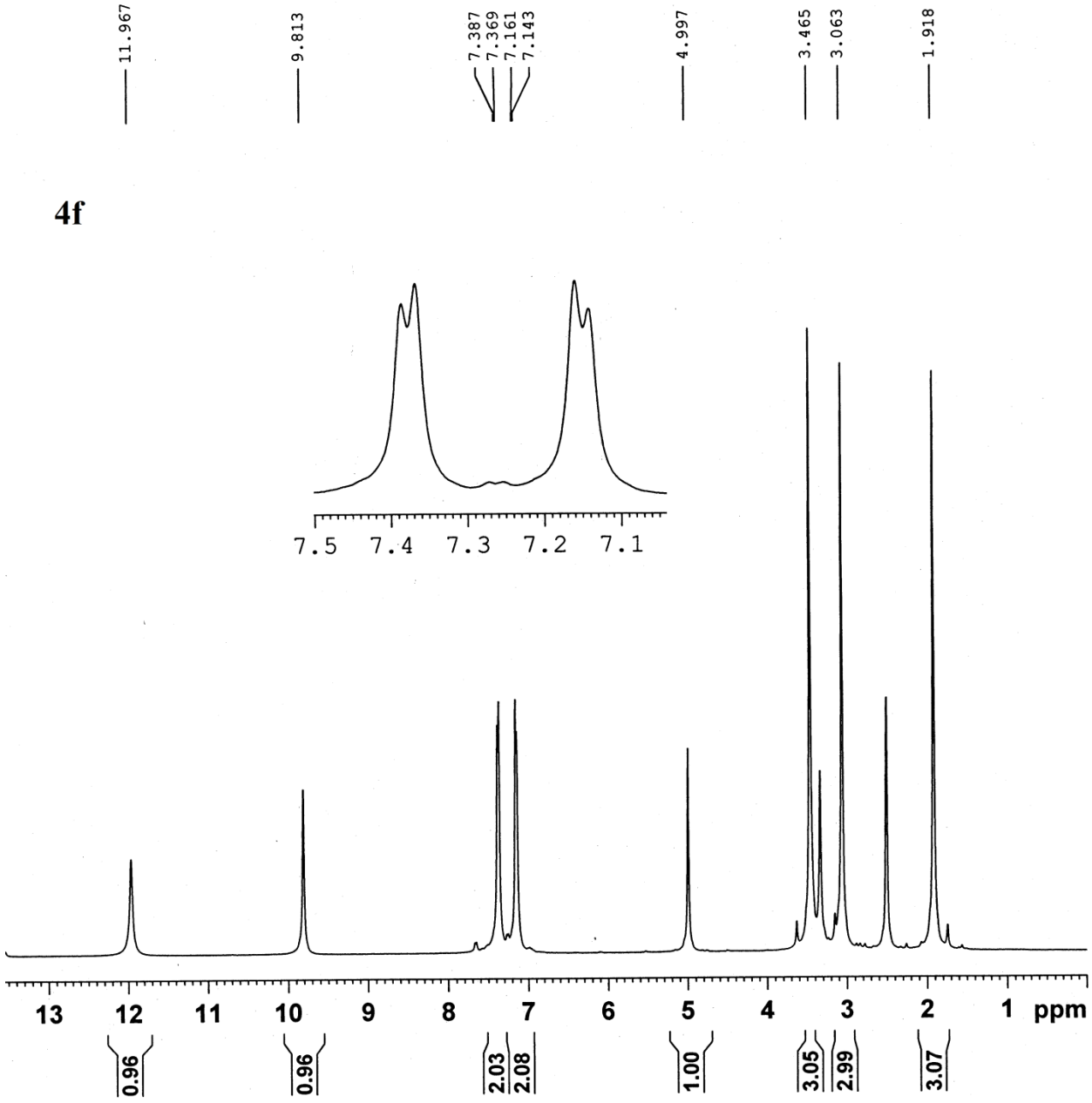
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	5.06	401.85	402452	100.00	1-1
Total			402452	100.00	

MS Spectrum Graph

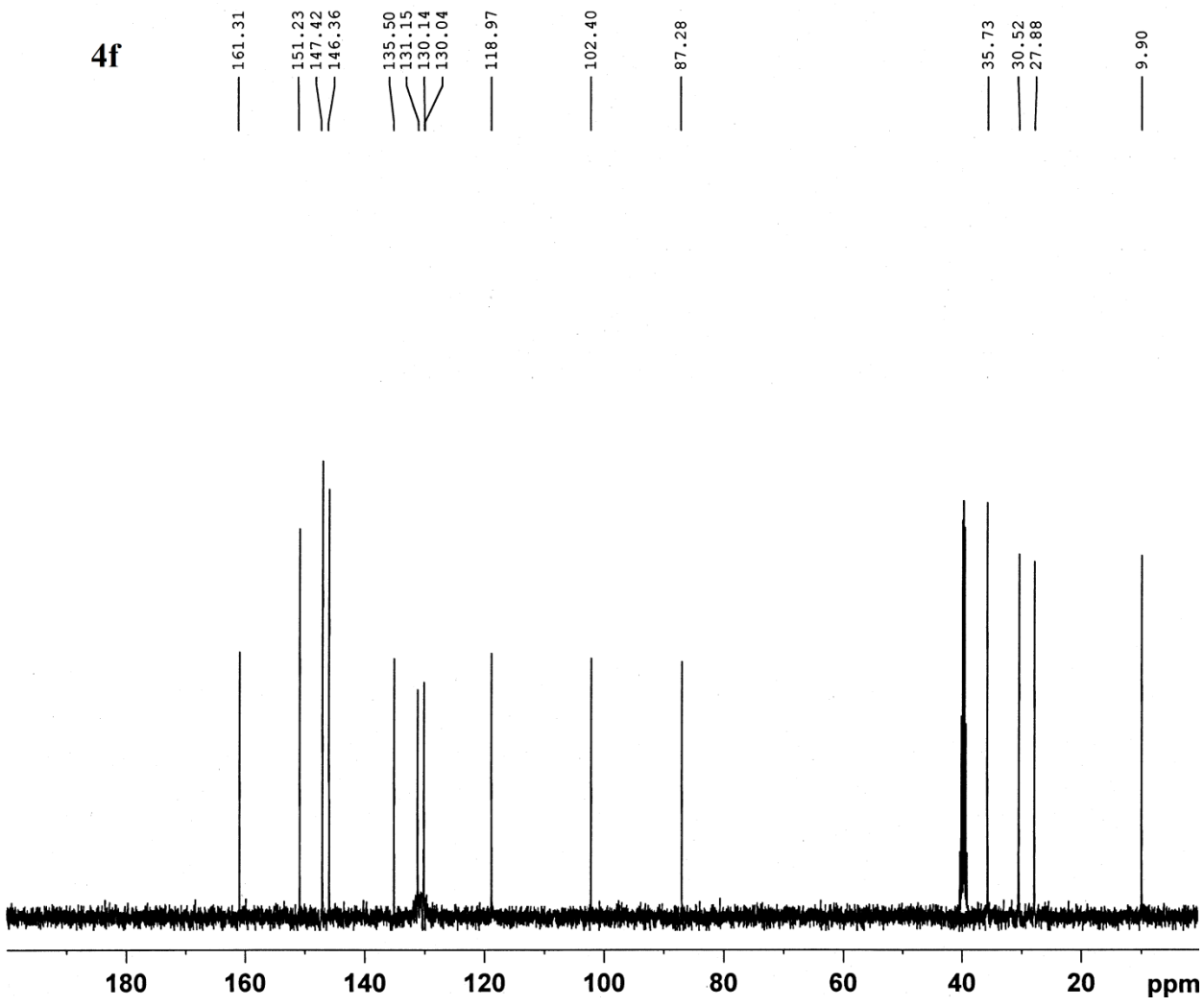
#1 Ret.Time:Averaged 5.050-5.083(Scan# 304-306)
BG Mode:Calc 4.900<->5.217(295<->314)
Base Peak:401.85(70486) Polarity:Pos Segment1 - Event1
Intensity



^{1}HNMR of compound **4f**

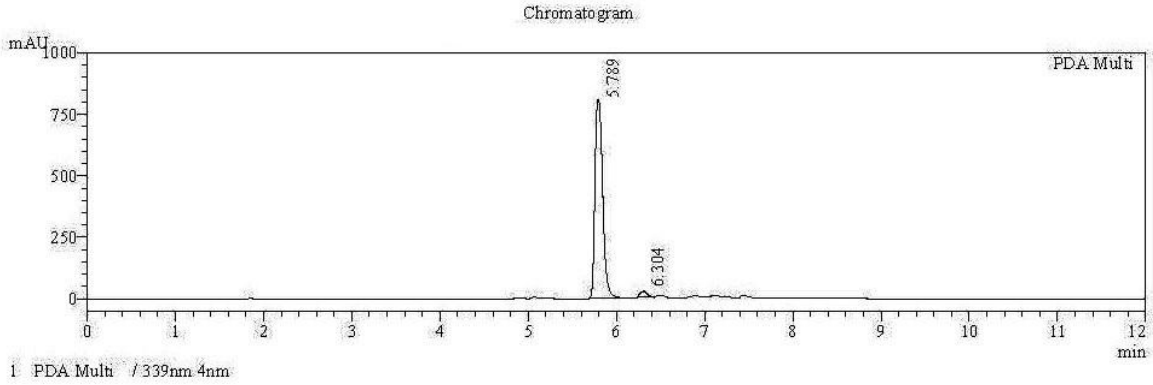


^{13}CNMR of compound **4f**

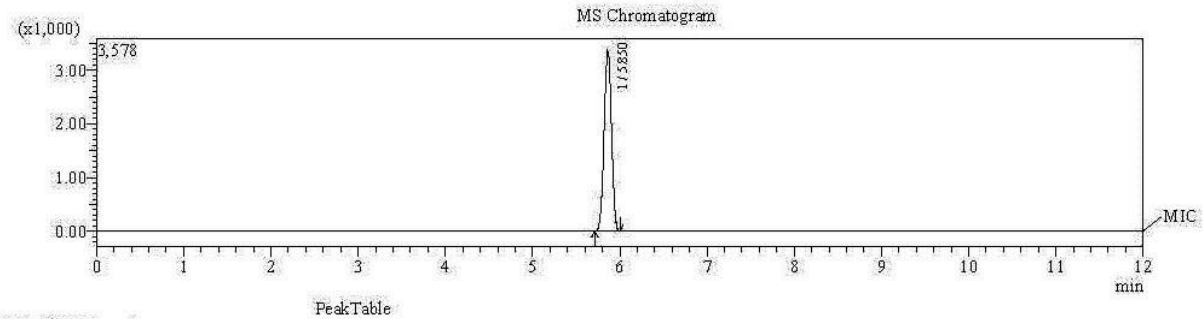


LC-MS of compound **4g**

Acquired by : Admin
Sample ID : 18
Vial# : 18
Injection Volume : 10
Data File : JAG-1-5-3-44.lcd
Method file : X-BRIDGE_150_12min_AA-FA.lcm



1 PDA Multi / 339nm 4nm



PeakTable

PDA Ch1 339nm 4nm

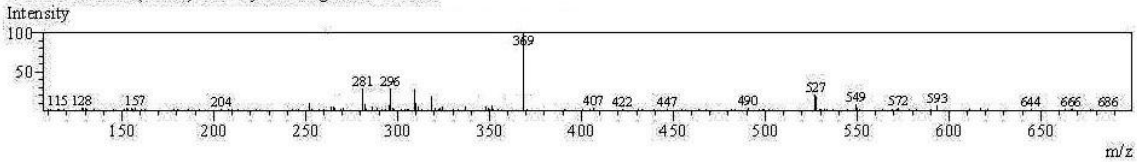
Peak#	Ret. Time	Area	Height	Area %
1	5.789	4875342	809860	97.434
2	6.304	128408	22816	2.566
Total		5003750	832676	100.000

MSTABLE MIC1

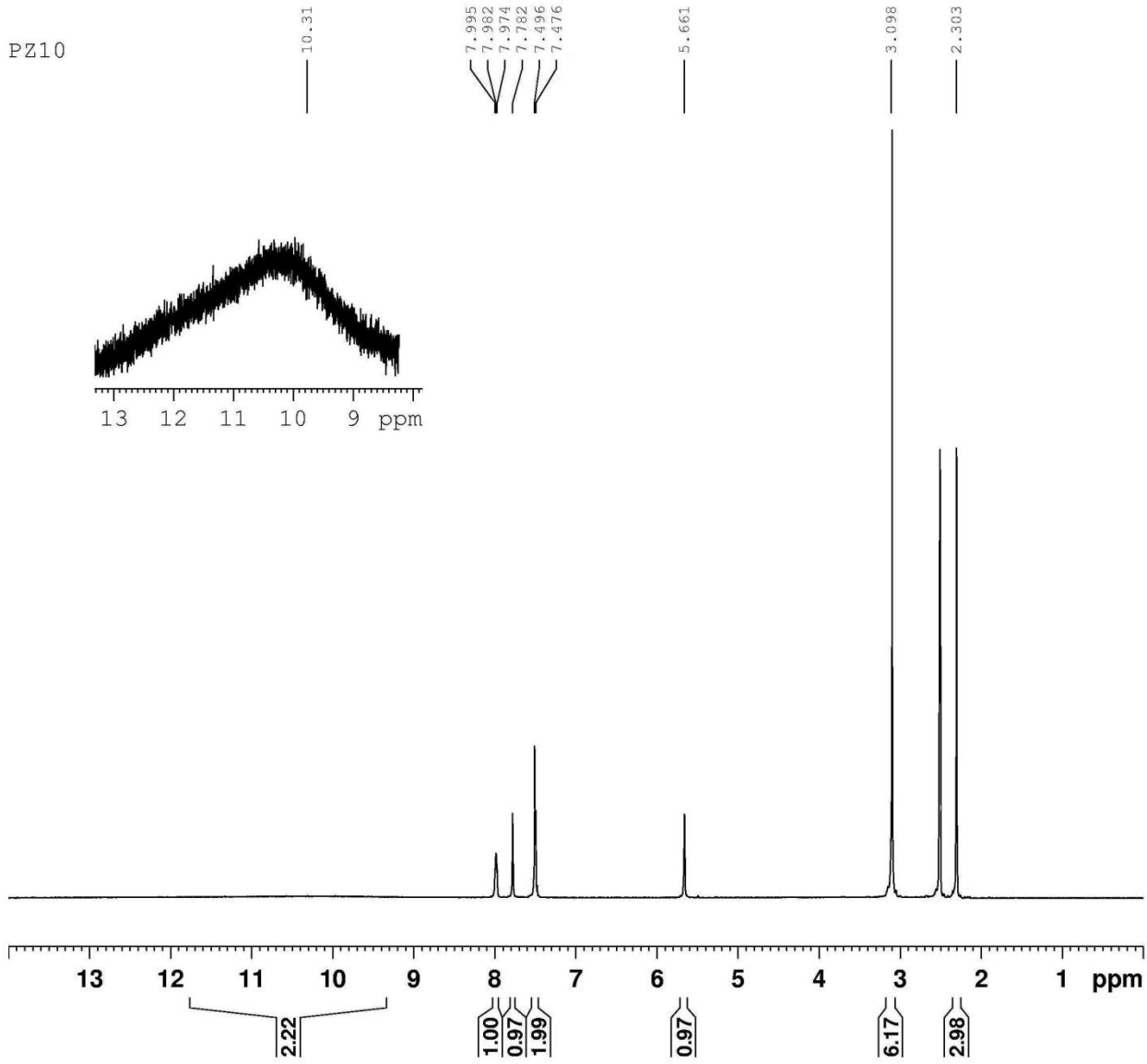
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	5.850	369.15	19898	100.00	1-1
Total			19898	100.00	

MS Spectrum Graph

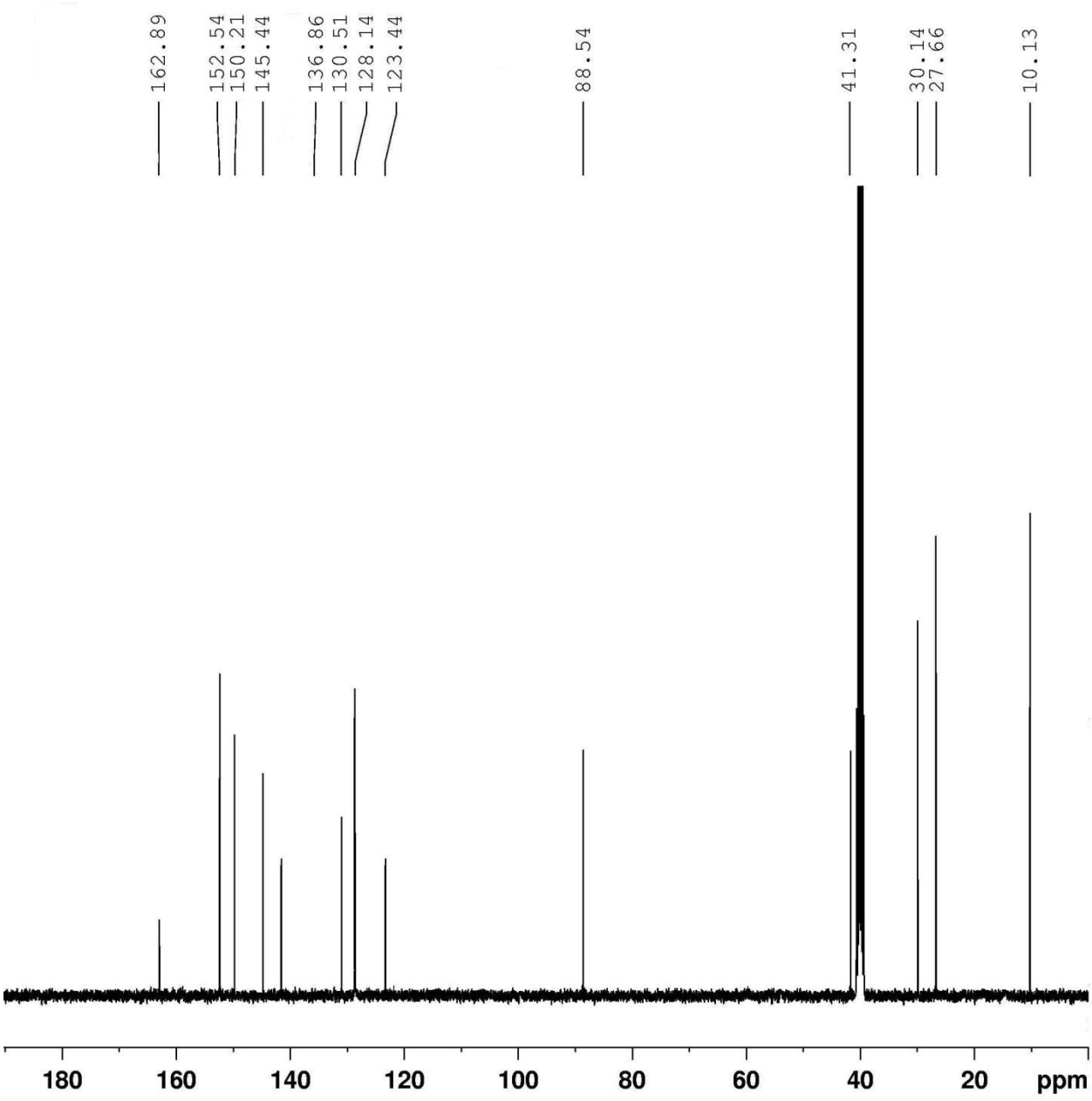
#1 Ret.Time:Averaged 5.833-5.867(Scan# 351-353)
BG Mode:Calc 5.717<->6.000(344<->361)
Base Peak:369.15(27763) Polarity:Pos Segment1 - Event1



^{1}HNMR of compound **4g**

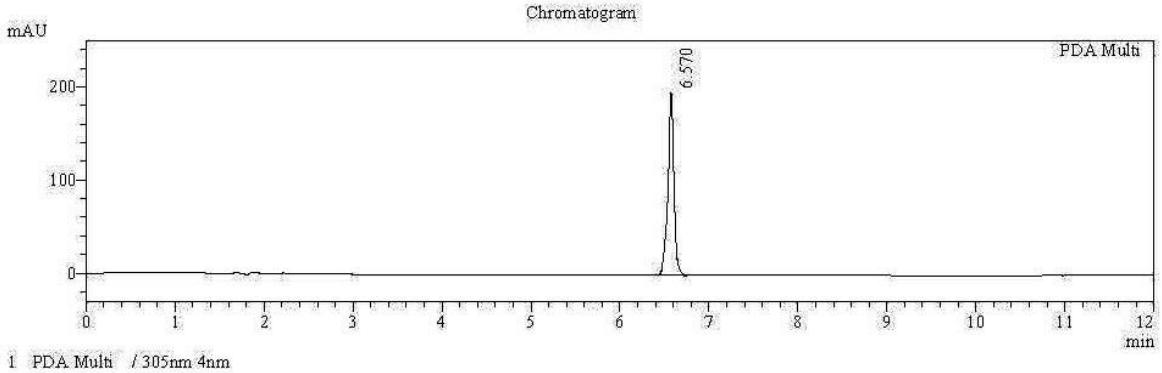


^{13}CNMR of compound **4g**

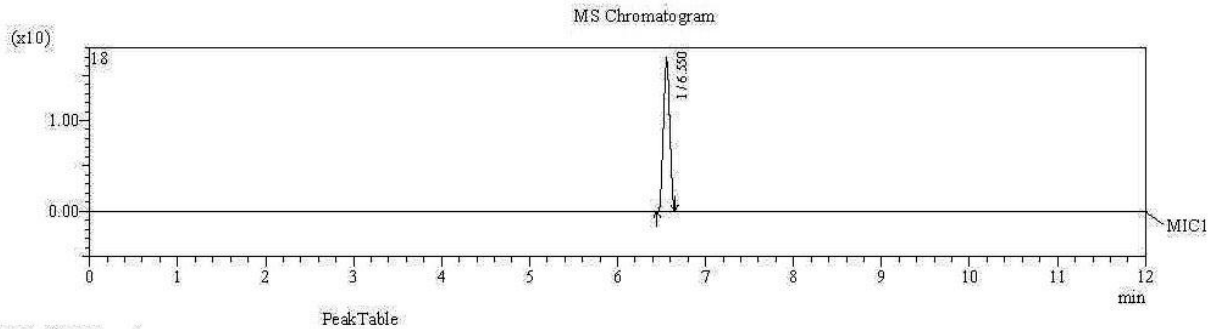


LC-MS of compound **4h**

Acquired by : Admin
Sample ID : O2Hr
Vial# : 19
Injection Volume : 10
Data File : OST-J-13-70-3-59_1.lcd
Method file : X-BRIDGE_150_12min_AA-FA.lcm



1 PDA Multi / 305nm 4nm



PeakTable

PDA Ch1 305nm 4nm

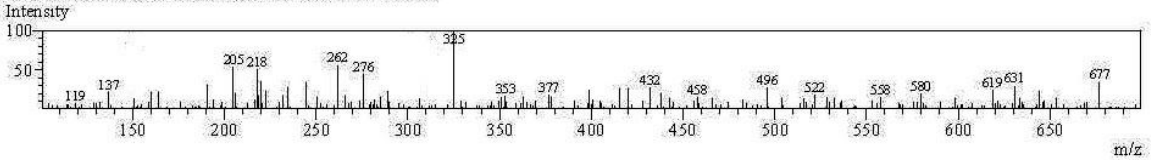
Peak#	Ret. Time	Area	Height	Area%
1	6.570	917581	195821	100.000
Total		917581	195821	100.000

MSTABLE MIC1

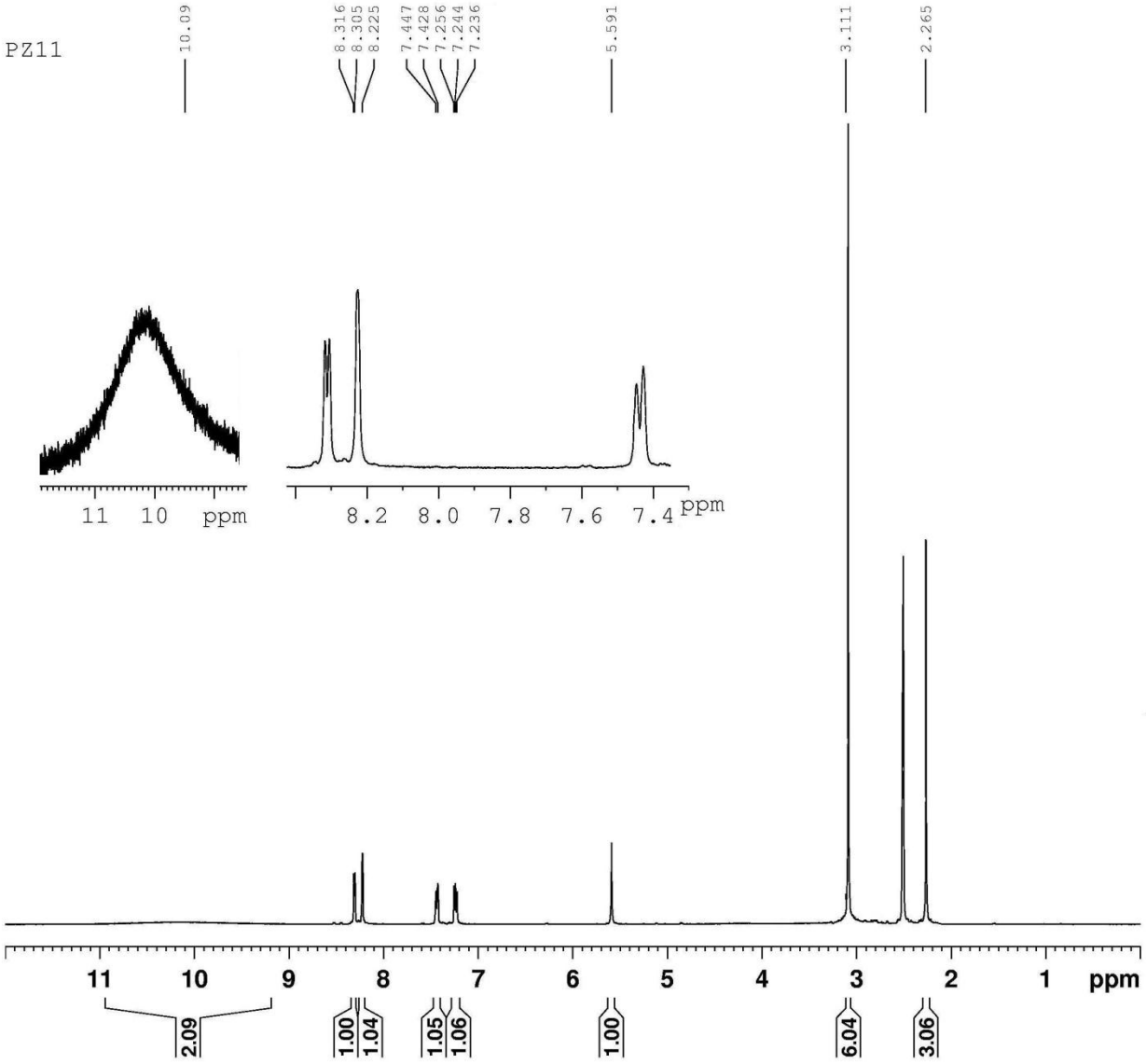
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	6.550	325.65	83	100.00	1-1
Total			83	100.00	

MS Spectrum Graph

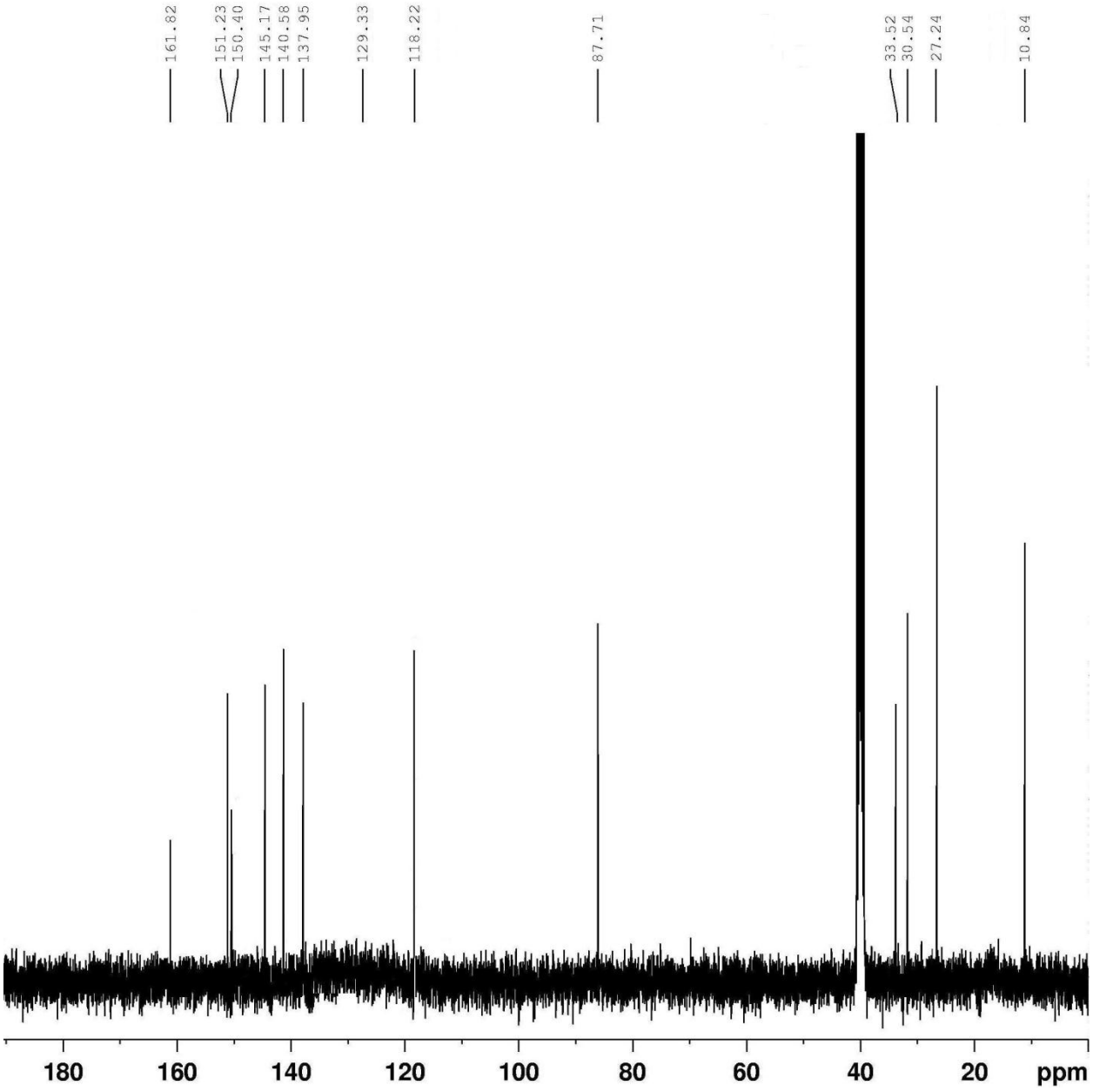
#1 Ret.Time:Averaged 6.533-6.567(Scan#:393-395)
BG Mode:Calc 6.450<->6.650(388<->400)
Base Peak:325.65(1869) Polarity:Neg Segment1 - Event1
Intensity



^{1}HNMR of compound **4h**

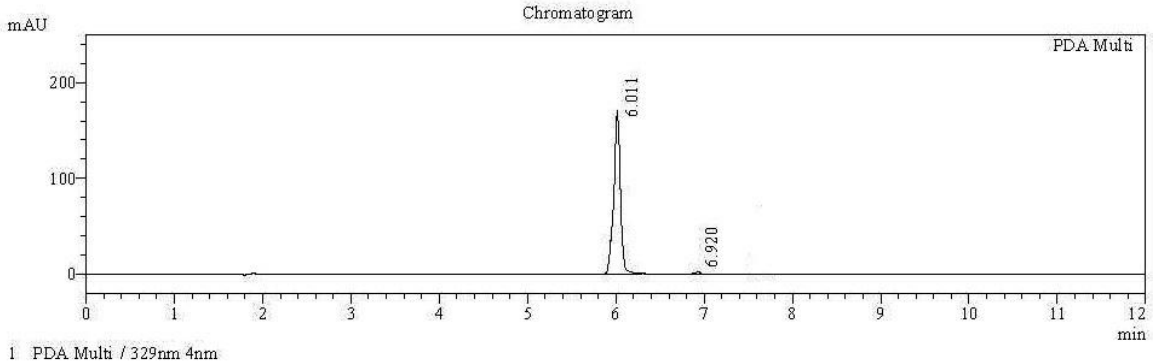


^{13}CNMR of compound **4h**

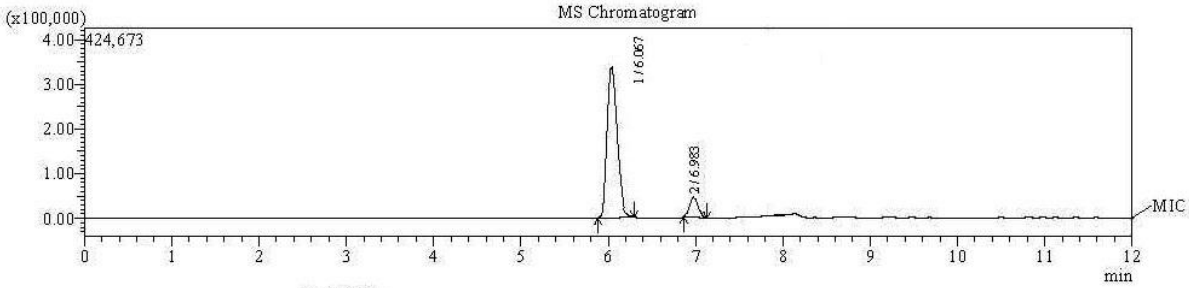


LC-MS of compound **4i**

Acquired by : Admin
Sample ID : 17
Vial# : 17
Injection Volume : 10
Data File : OST-J-14-89-ST-5-147.lcd
Method file : X-BRIDGE_150_12min_AA-FA.lcm



1 PDA Multi / 329nm 4nm



PeakTable

PDA Ch1 329nm 4nm

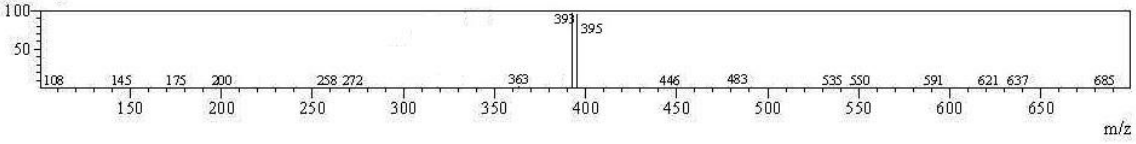
Peak#	Ret. Time	Area	Height	Area %
1	6.011	895599	192939	99.095
2	6.920	9143	2193	0.905
Total		1010833	195132	100.000

MSTABLE MIC1

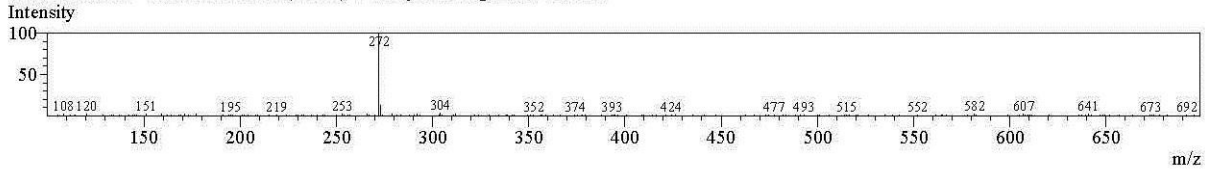
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	6.067	393.40	5300803	94.50	1-1
2	6.983	272.20	308269	5.50	1-1
Total			5609072	100.00	

MS Spectrum Graph

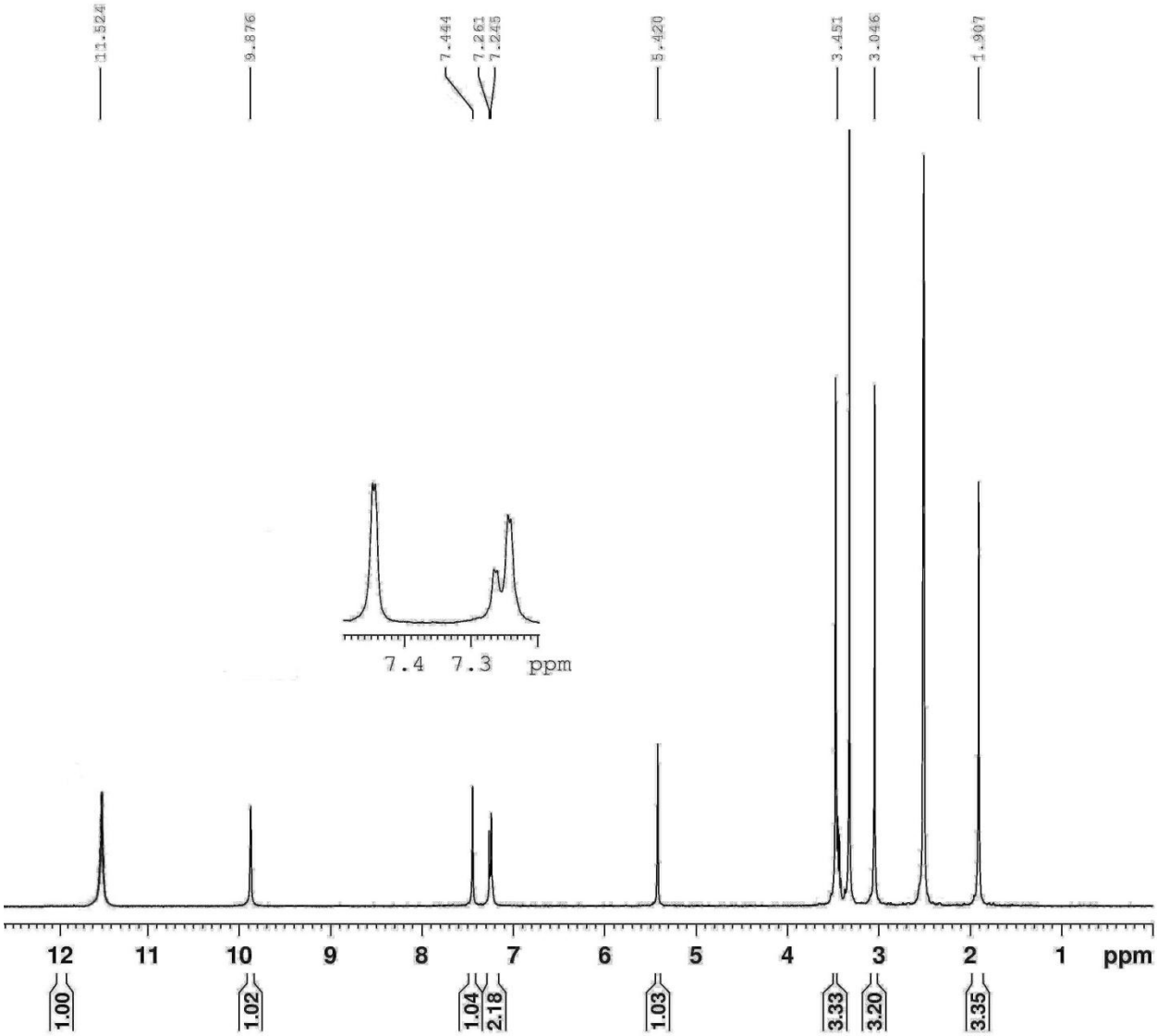
#:1 Ret.Time:Averaged 6.050-6.083(Scan#:364-366)
BG Mode:Calc 5.883<->6.300(354<->379)
Base Peak:393.40(205644) Polarity:Pos Segment1 - Event1
Intensity



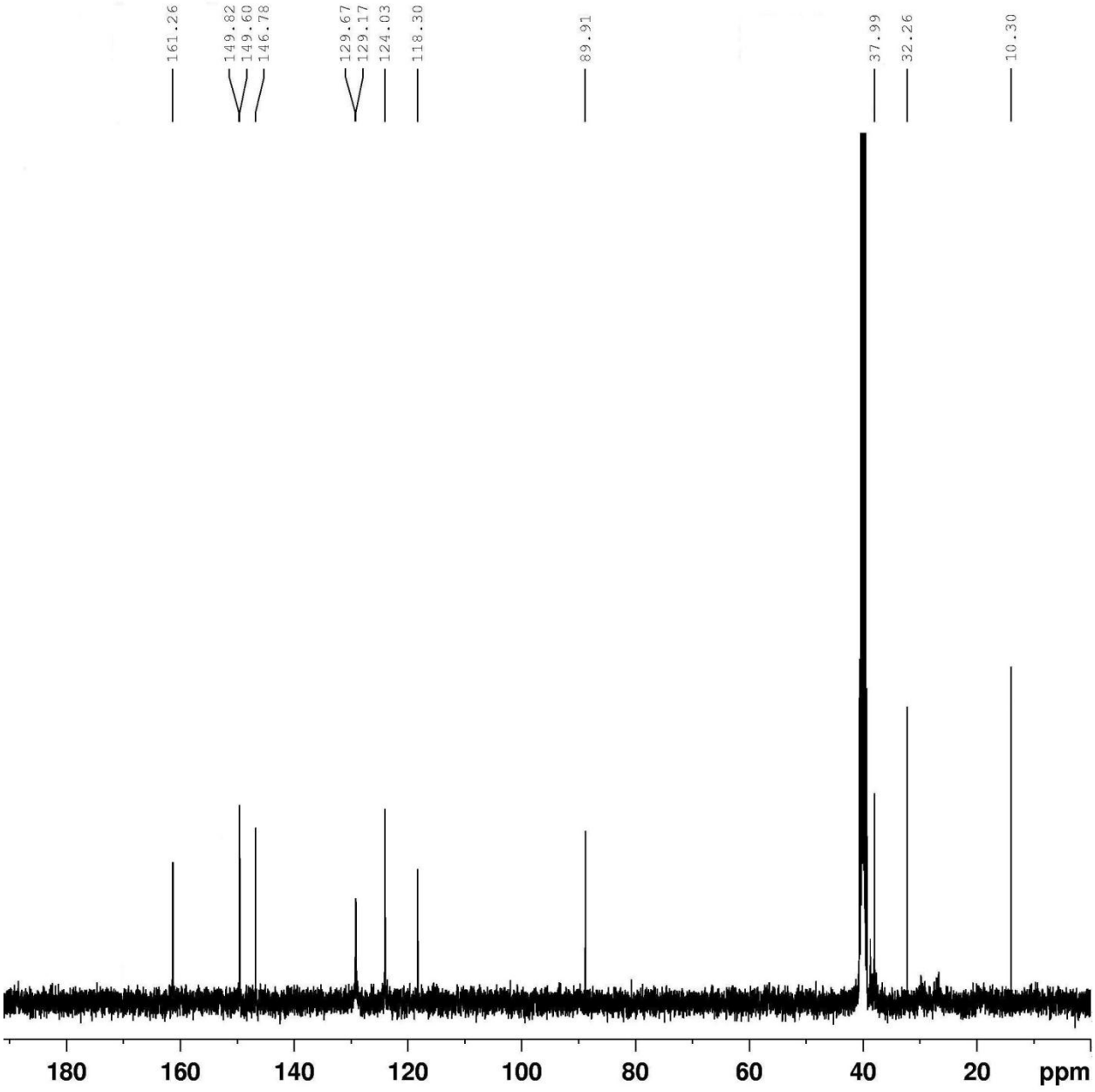
#:2 Ret.Time:Averaged 6.967-7.000(Scan#:419-421)
BG Mode:Calc 6.867<->7.133(413<->429)
Mass Peaks:282 Base Peak:272.20(62307) Polarity:Pos Segment1 - Event1
Intensity



^{1}HNMR of compound **4i**

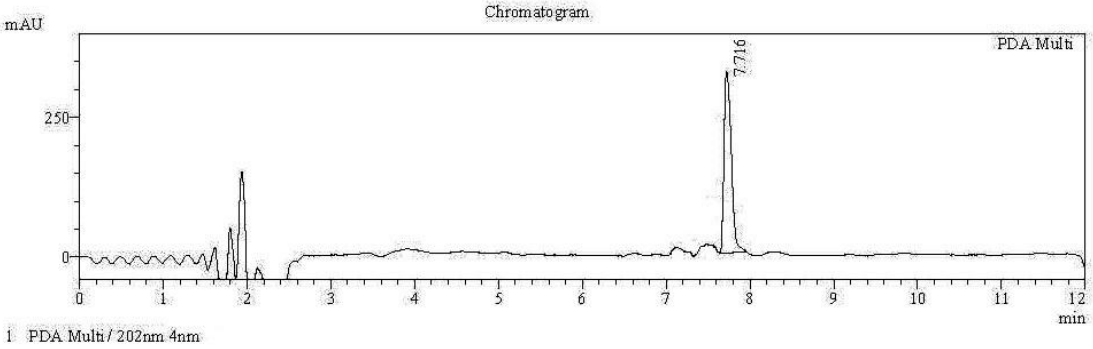


^{13}CNMR of compound **4i**

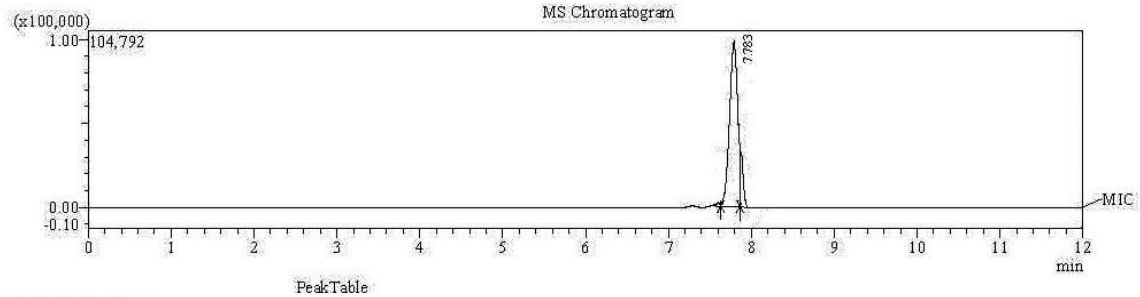


LC-MS of compound **4j**

Acquired by	: Admin
Sample ID	: 20
Vial#	: 20
Injection Volume	: 30
Data File	: OST-J-13-70-3-87_1cd
Method file	: X-BRIDGE_150_12min_AA-FA.lcm



1 PDA Multi / 202nm 4nm



PeakTable

PDA Ch1 202nm 4nm

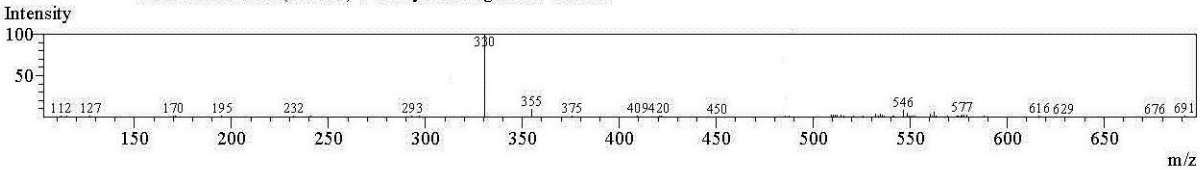
Peak#	Ret. Time	Area	Height	Area%
1	7.716	2052318	342235	100.000
Total		2052318	342235	100.000

MSTABLE MIC1

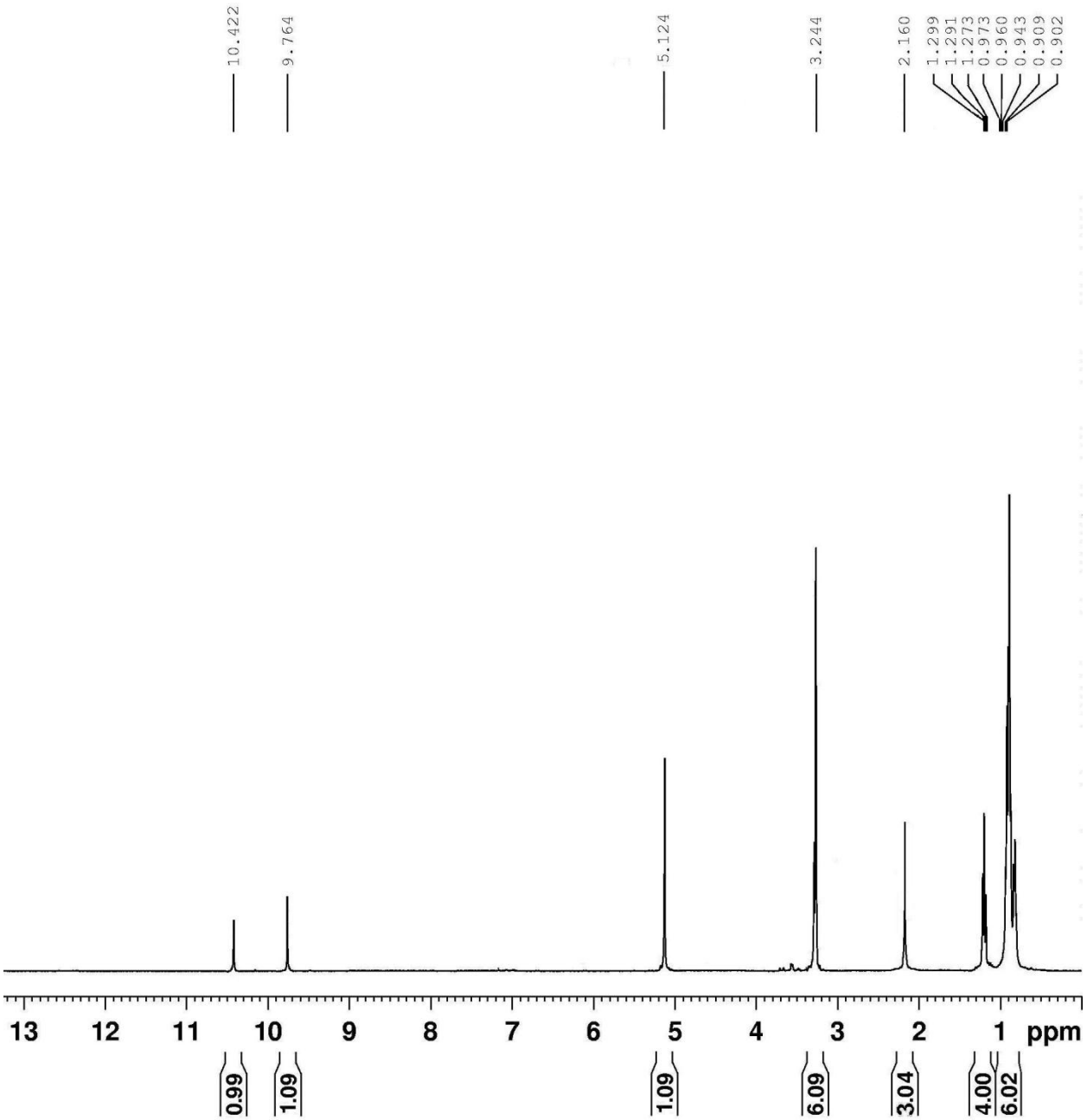
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	7.783	330.43	933076	100.00	1-1
Total			933076	100.00	1-1

MS Spectrum Graph

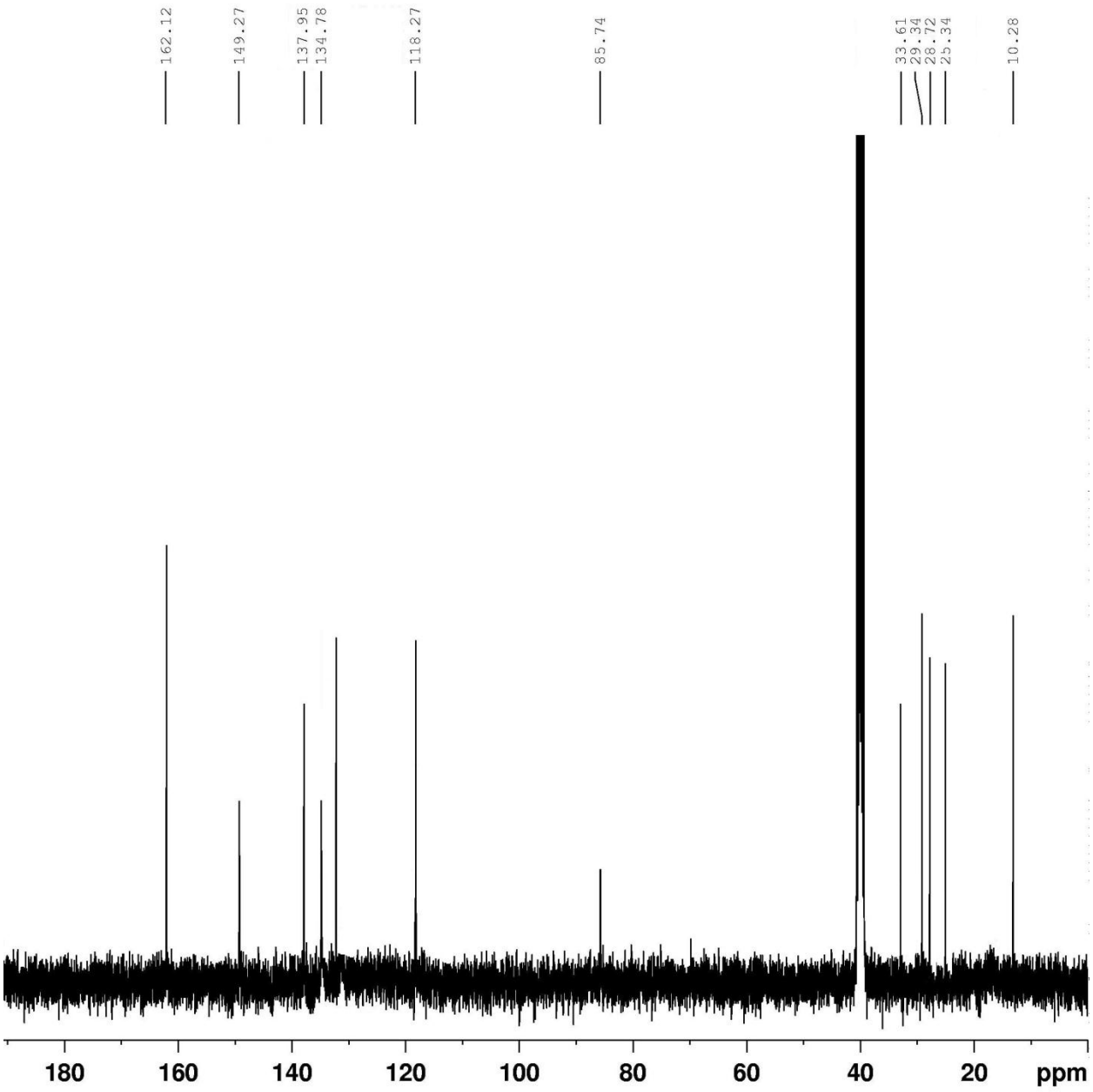
#:1 Ret.Time:Averaged 7.767-7.800(Scan#:467-469)
BG Mode:Calc 7.633<->7.867(459<->473)
Base Peak:330.43(159294) Polarity:Pos Segment1 - Event1



^{1}HNMR of compound **4j**

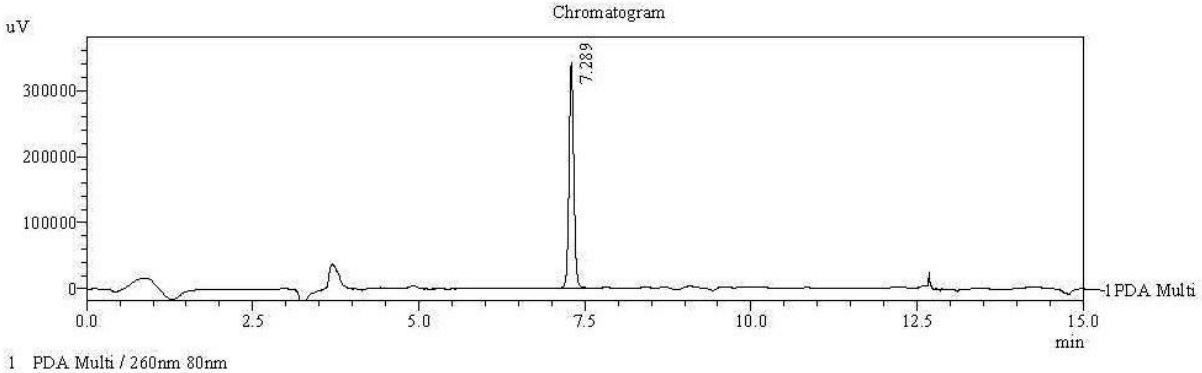


^{13}CNMR of compound **4j**

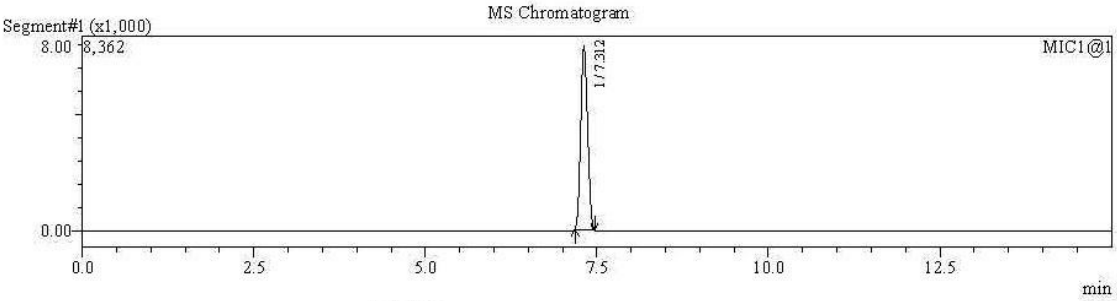


LC-MS of compound **4k**

Acquired by : Admin
Sample ID : 1
Vial# : 16
Injection Volume : 5
Data File : TPX-621-J3-FINAL.lcd
Method file : GENESIS-LCMS-ESI-150.lcm



1 PDA Multi / 260nm 80nm



PeakTable

PDA Ch1 260nm 80nm

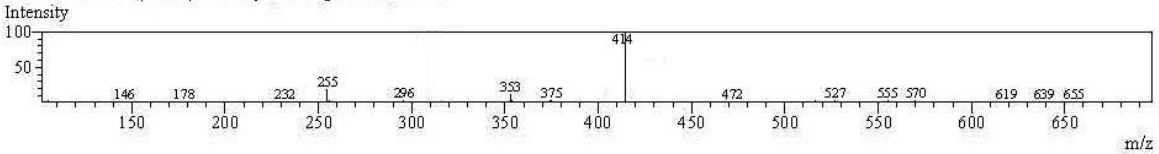
Peak#	Ret. Time	Area	Height	Area %
1	7.289	1685969	340291	100.000
Total		1685969	340291	100.000

MSTABLE MIC1

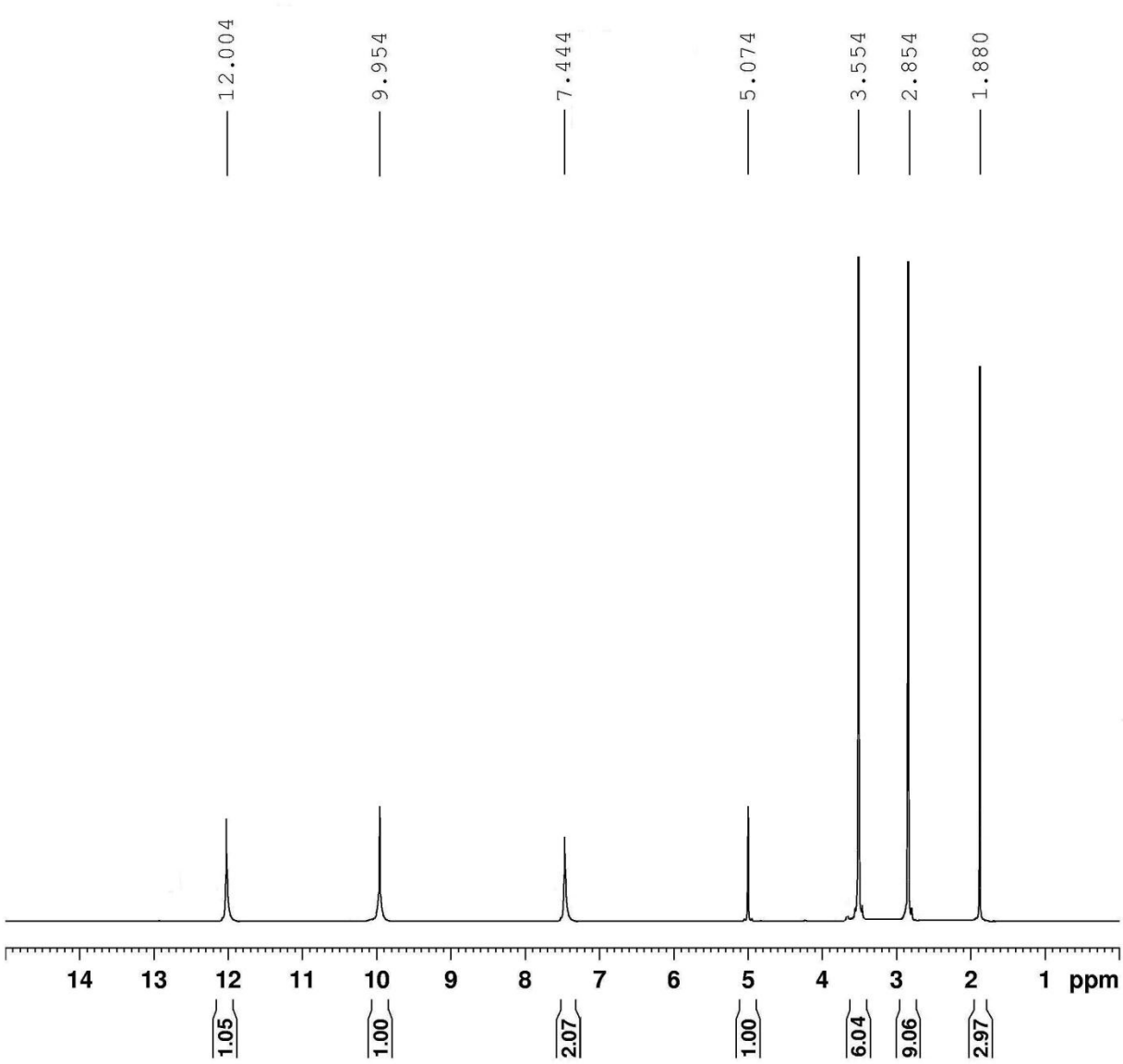
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	7.312	414.60	55043	100.00	1-1
Total			55043	100.00	

MS Spectrum Graph

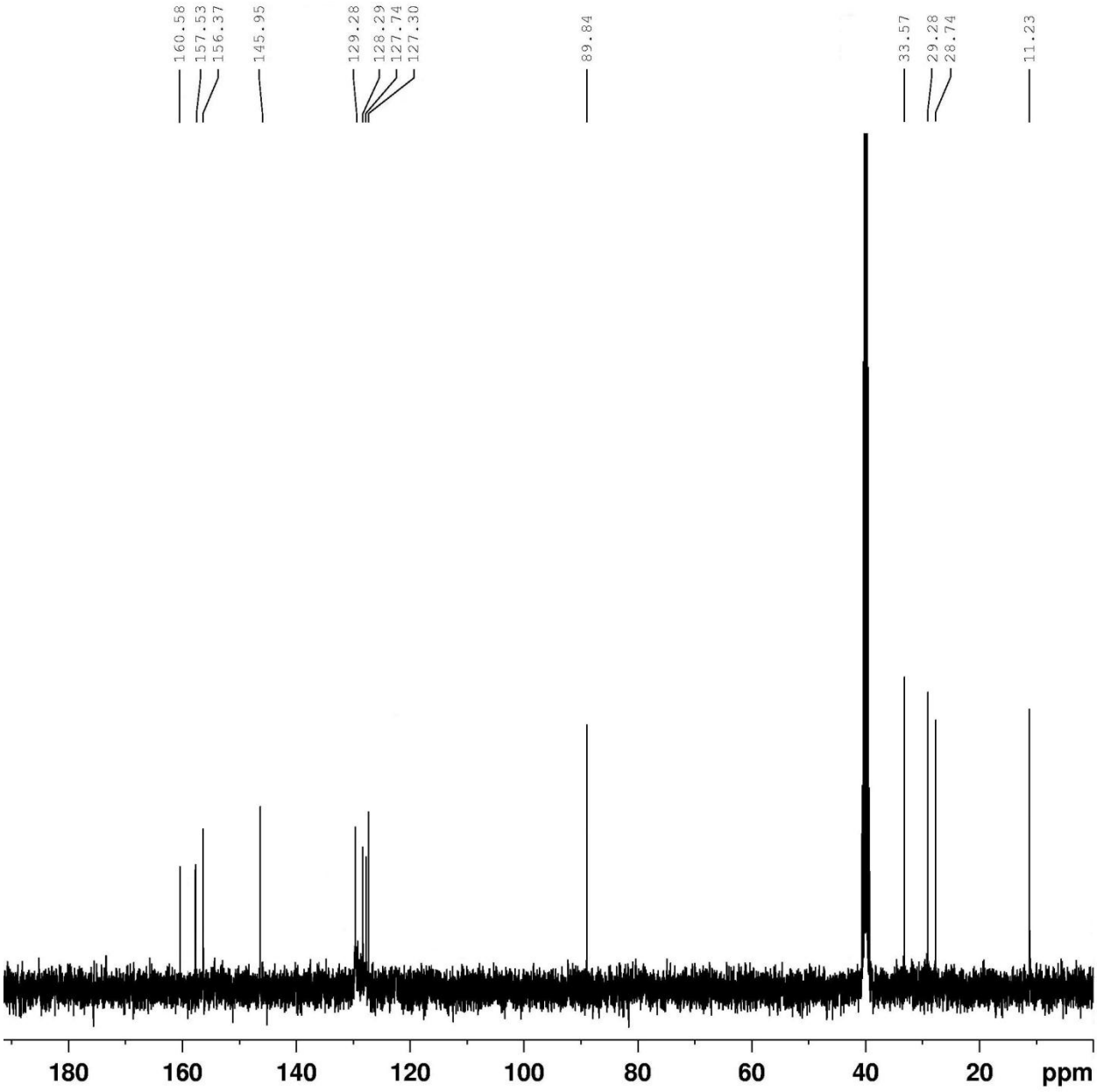
#:1 Ret.Time:Averaged 7.300-7.333(Scan#:439-441)
BG Mode:Calc 7.183<->7.467(432<->449)
Base Peak:414.60(17991) Polarity:Pos Segment1 - Event1



^{1}HNMR of compound **4k**

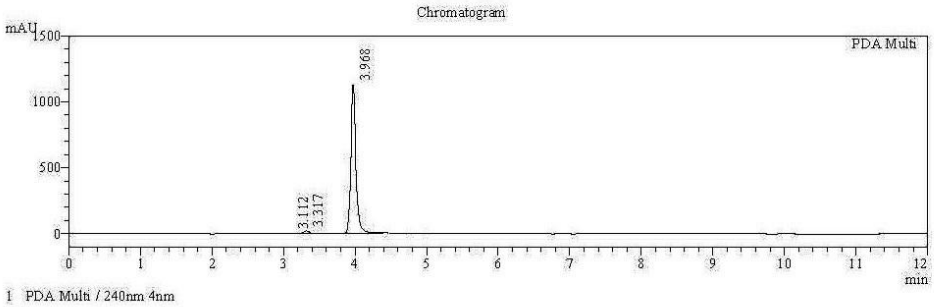


^{13}CNMR of compound **4k**

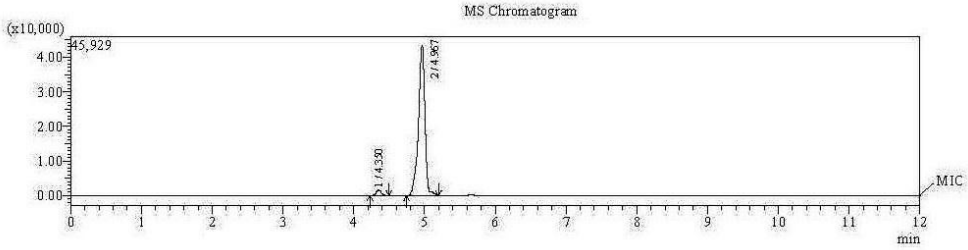


LC-MS of compound **4l**

Acquired by : Admin
Sample ID : 21
Vial# : 21
Injection Volume : 10
Data File : JAG-13-1-4-US.lcd
Method file : X-BRIDGE_150_12min_AA-FA.lcm



1 PDA Multi / 240nm 4nm



PDA Ch2 240nm 4nm

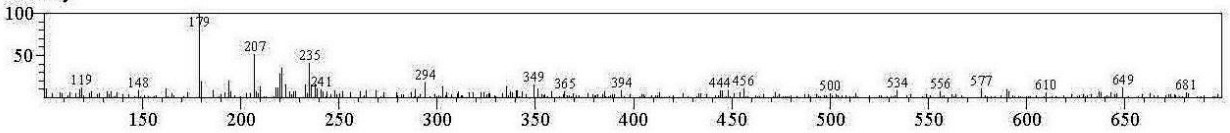
Peak#	Ret. Time	Area	Height	Area %
1	3.112	18292	3944	0.310
2	3.317	91974	19111	1.560
3	3.968	5786190	1185506	98.130
Total		5896455	1208561	100.000

MSTABLE MIC1

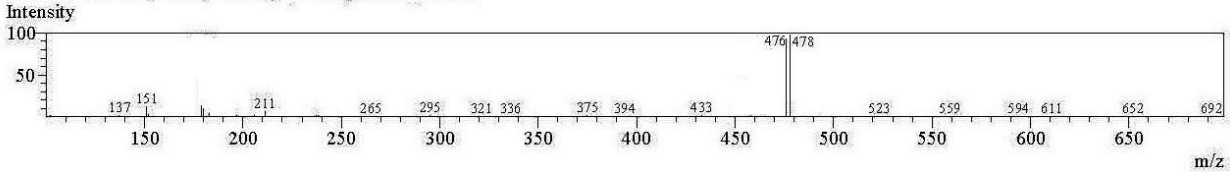
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	4.350	179.05	8304	2.91	1-1
2	4.967	478.10	277527	97.09	1-1
Total			285831	100.00	

MS Spectrum Graph

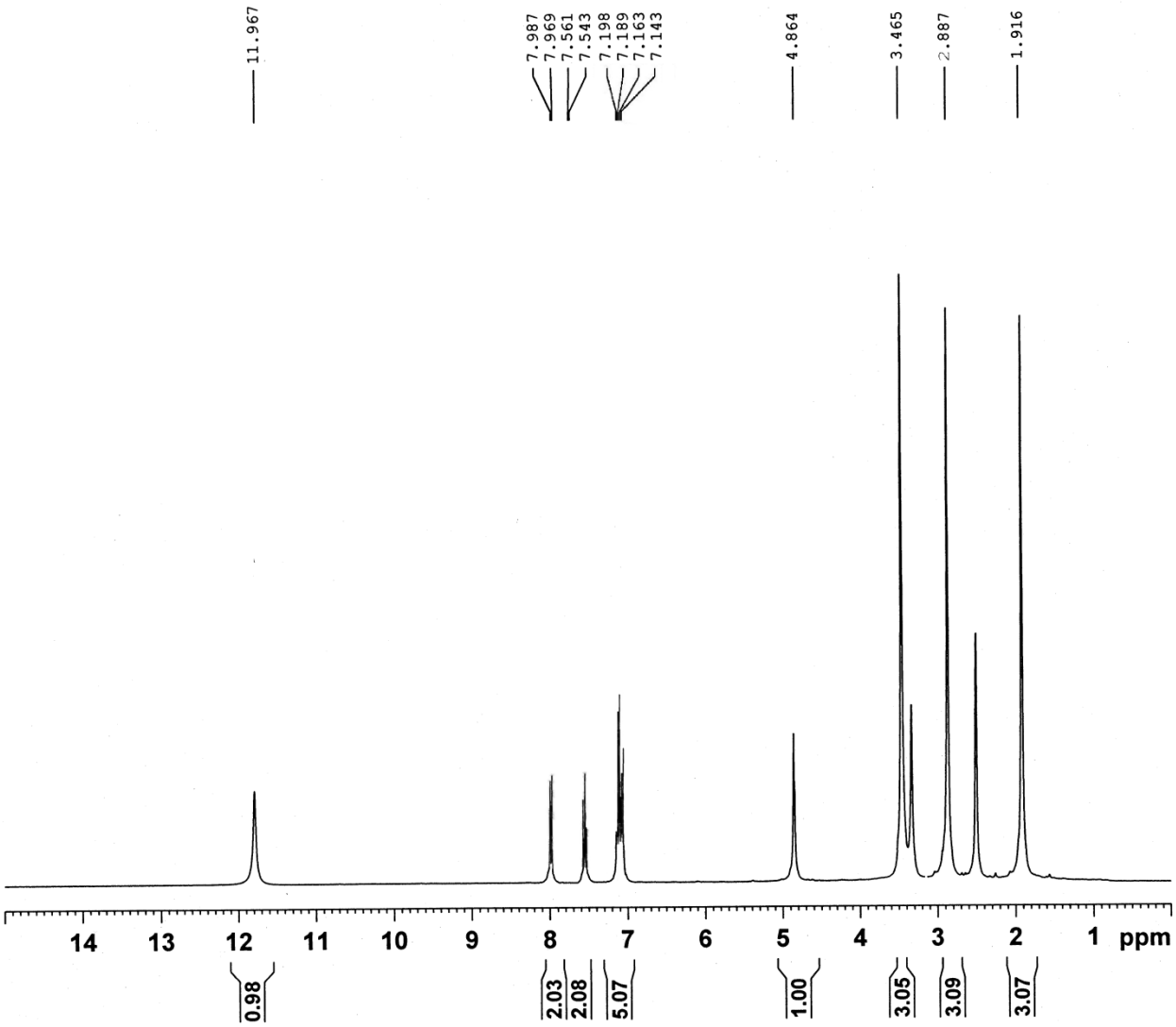
#:1 Ret.Time:Averaged 4.333-4.367(Scan#:261-263)
BG Mode:Calc 4.233<->4.500(255<->271)
Base Peak:179.05(5403) Polarity:Pos Segment1 - Event1
Intensity



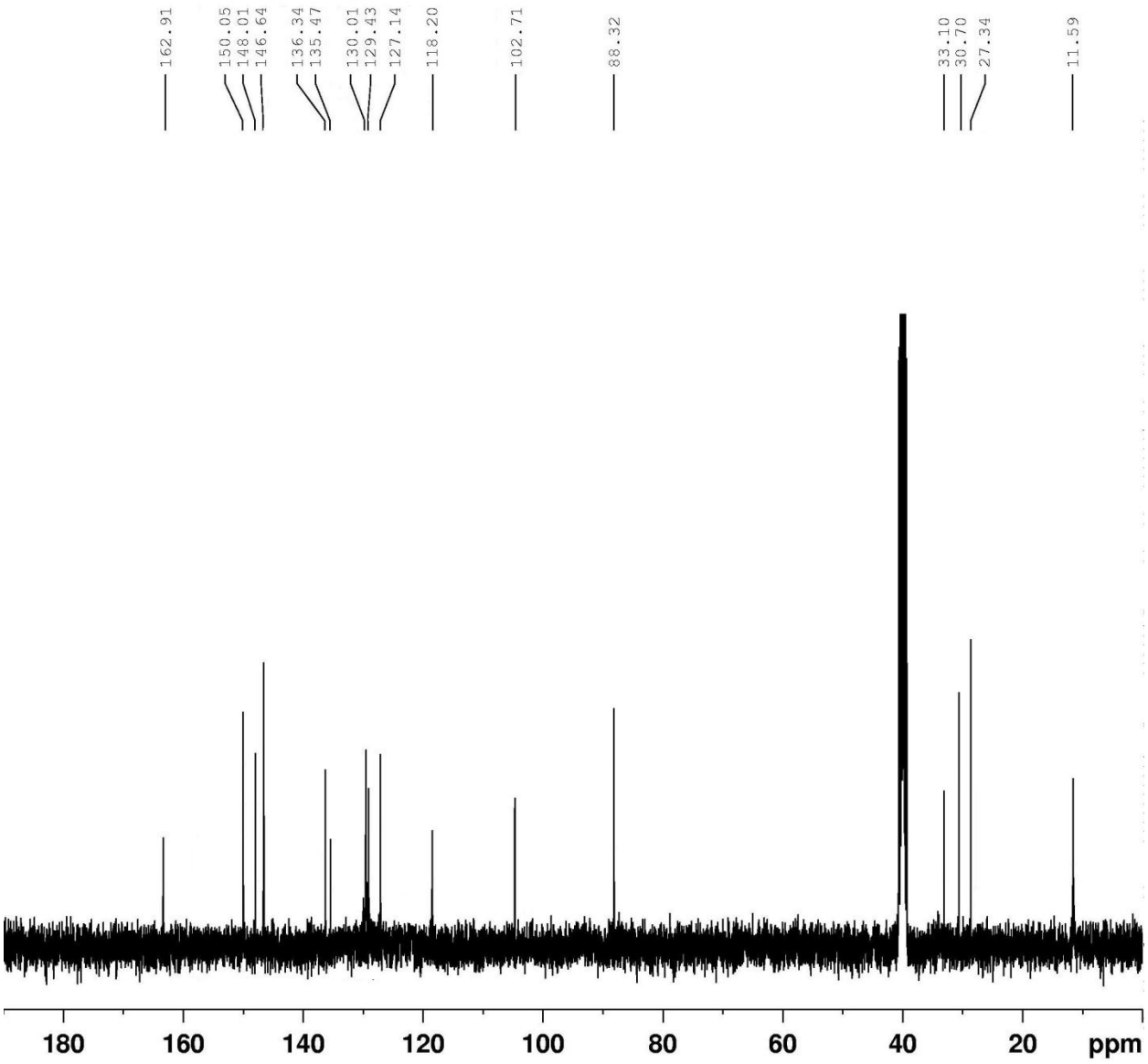
#:2 Ret.Time:Averaged 4.950-4.983(Scan#:298-300)
BG Mode:Calc 4.750<->5.200(286<->313)
Base Peak:478.10(72312) Polarity:Pos Segment1 - Event1
Intensity



^{1}HNMR of compound **4l**

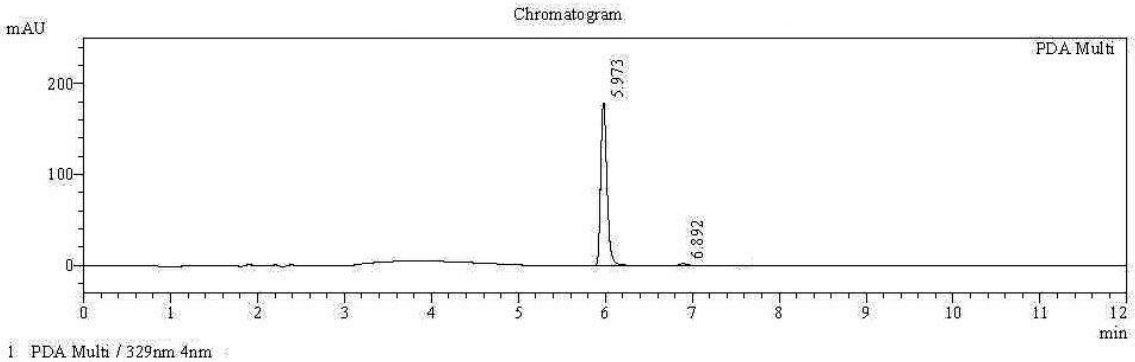


^{13}CNMR of compound **4l**

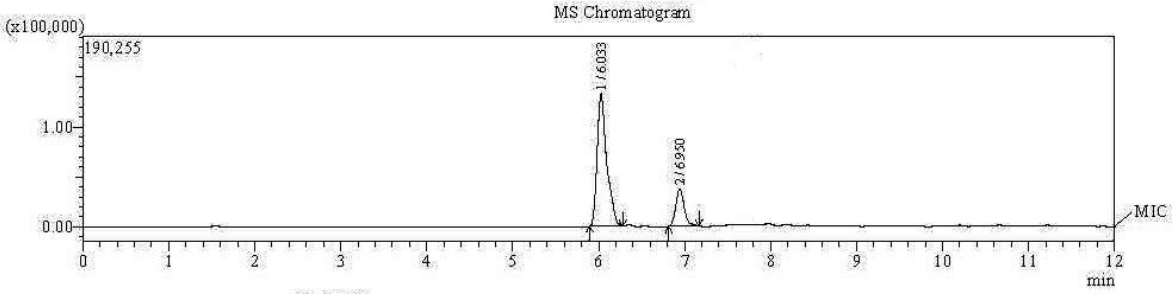


LC-MS of compound **4m**

Acquired by : Admin
Sample ID : 3
Vial# : 19
Injection Volume : 12
Data File : OST-J-14-89-ST-5-147_1cd
Method file : X-BRIDGE_150_12min_AA-FA.lcm



1 PDA Multi / 329nm 4nm



PeakTable

PDA Ch1 329nm 4nm

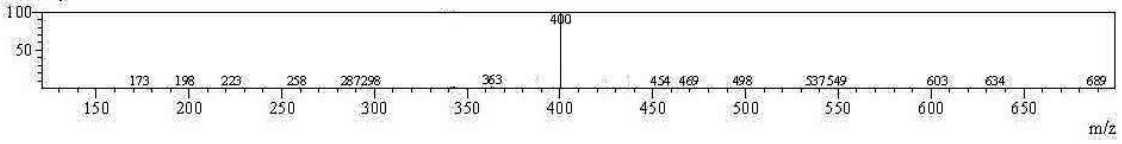
Peak#	Ret. Time	Area	Height	Area%
1	5.973	908203	183033	98.914
2	6.892	9966	1939	1.085
Total		918169	184972	100.000

MSTABLE MIC1

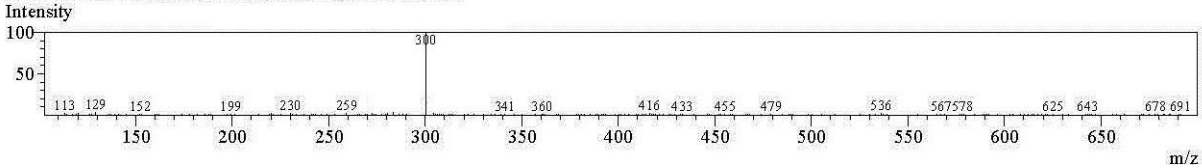
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	6.033	400.25	2576798	91.03	1-1
2	6.950	302.20	253881	8.97	1-1
Total			2830679	100.00	

MS Spectrum Graph

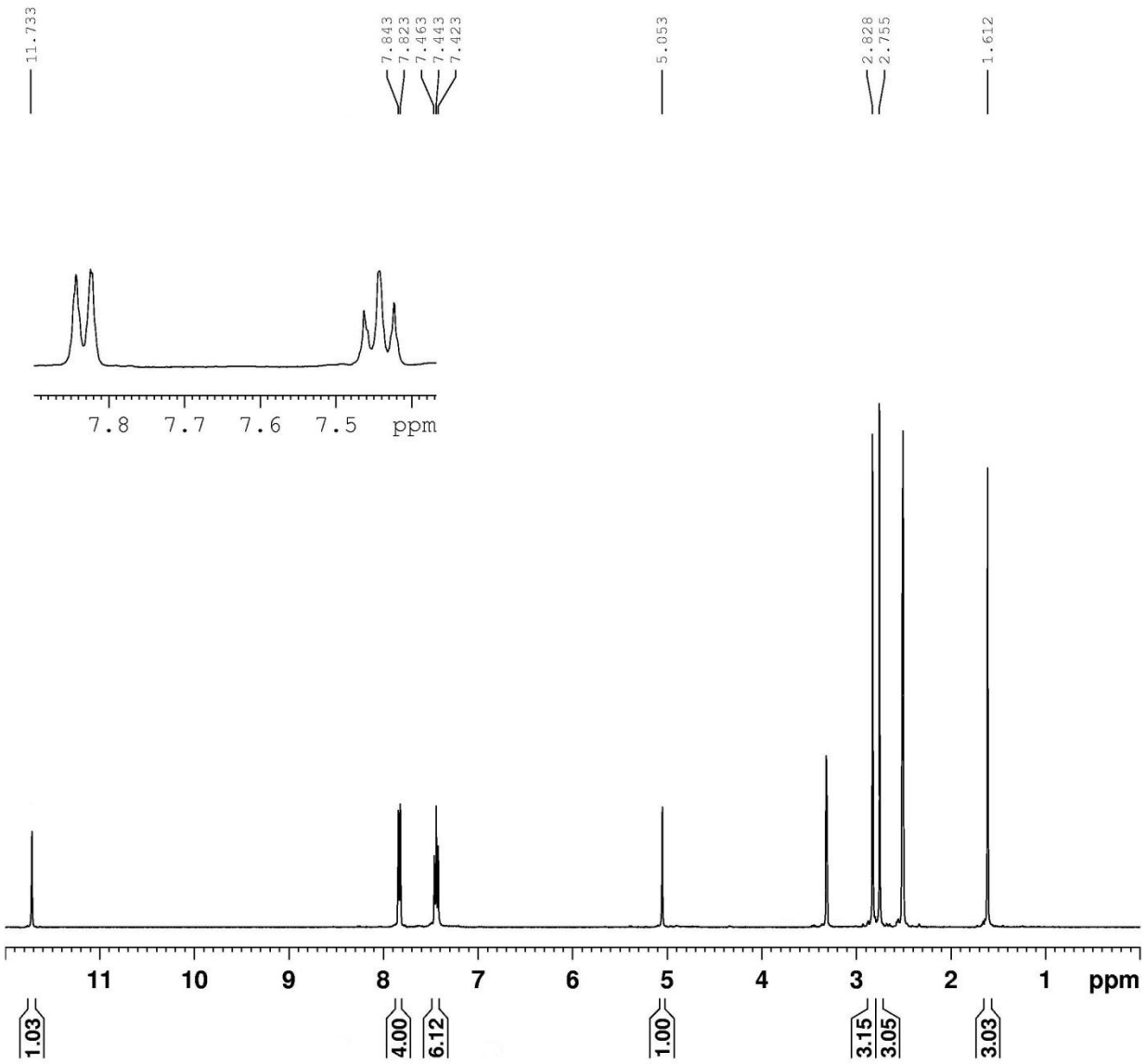
#:1 Ret.Time:Averaged 6.017-6.050(Scan#:362-364)
BG Mode:Calc 5.900<->6.283(355<->378)
Base Peak:400.25(206089) Polarity:Pos Segment1 - Event1
Intensity



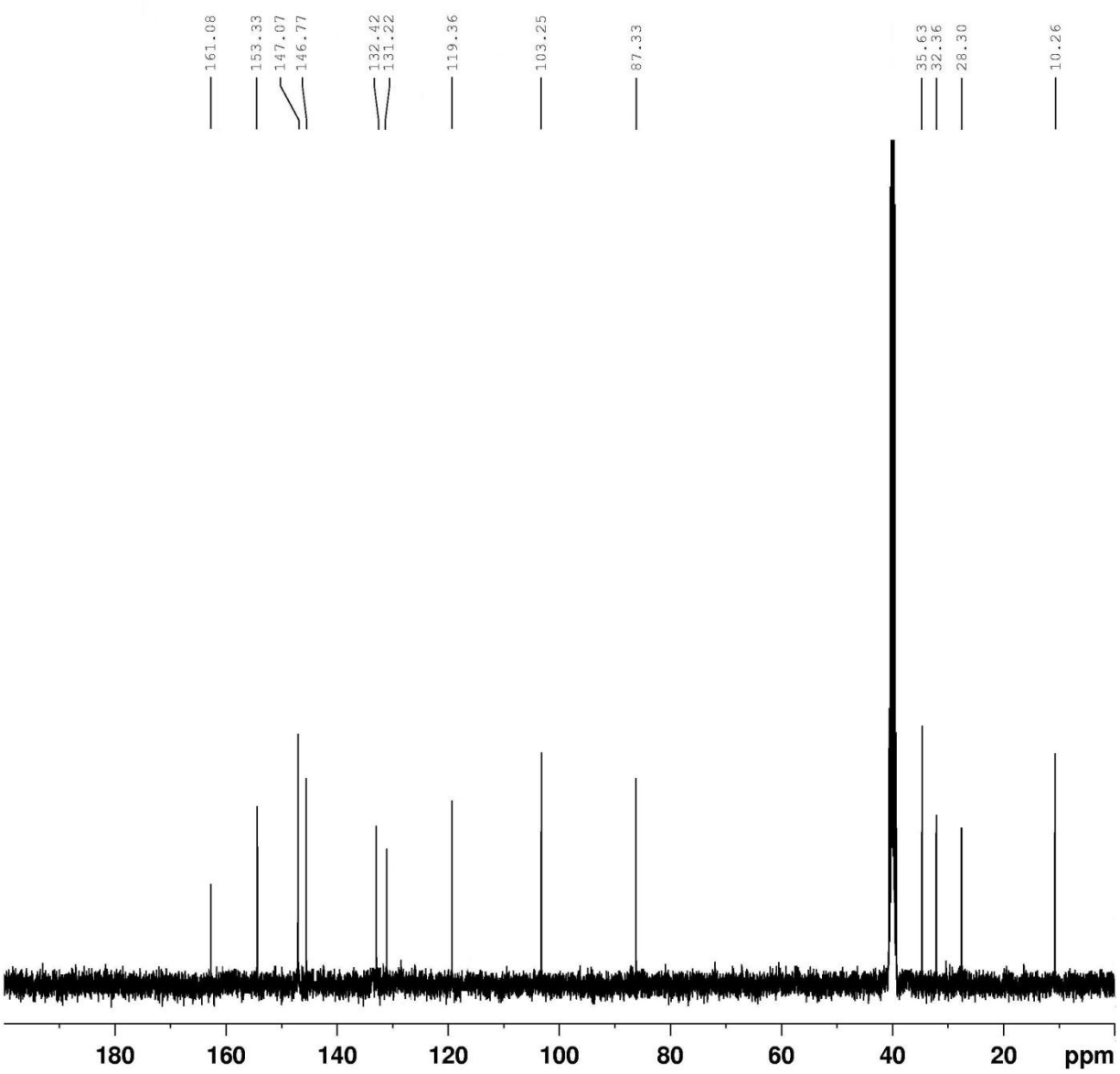
#:2 Ret.Time:Averaged 6.933-6.967(Scan#:417-419)
BG Mode:Calc 6.817<->7.167(410<->431)
Base Peak:300.20(59166) Polarity:Pos Segment1 - Event1
Intensity



^{1}HNMR of compound **4m**



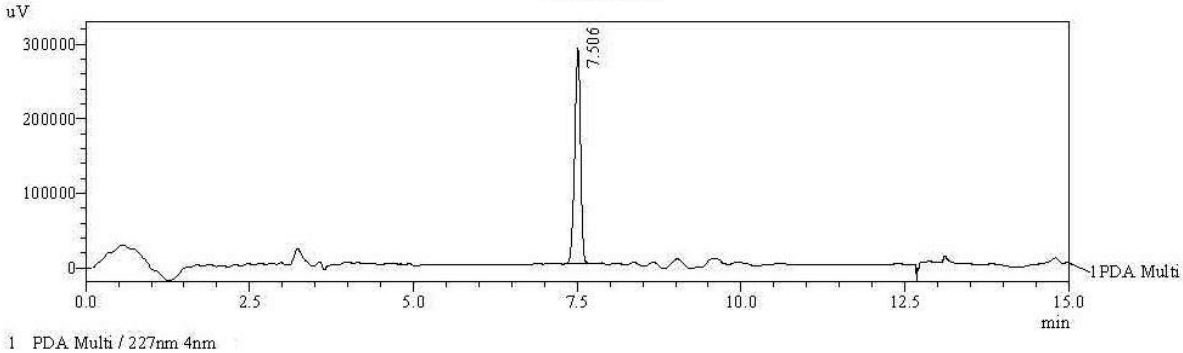
^{13}CNMR of compound **4m**



LC-MS of compound **4n**

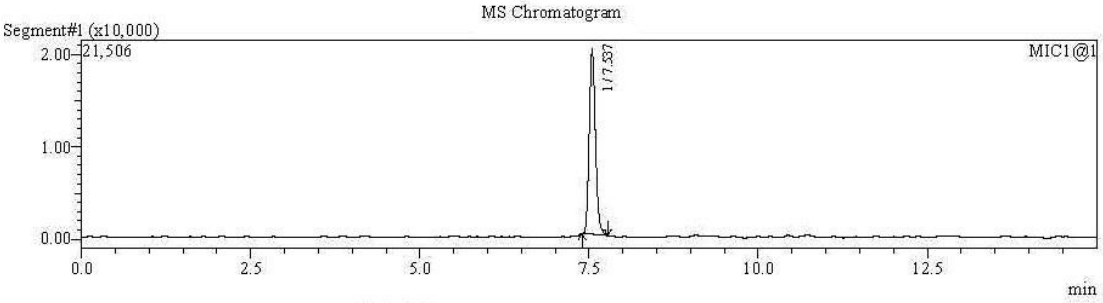
Acquired by : Admin
Sample ID : 3
Vial# : 3
Injection Volume : 5
Data File : VTX-622-D-30-FINAL-AP21cd
Method file : GENESIS-LCMS-ESI-150.lcm

Chromatogram



1 PDA Multi / 227nm 4nm

MS Chromatogram



PeakTable

PDA Ch1 227nm 4nm

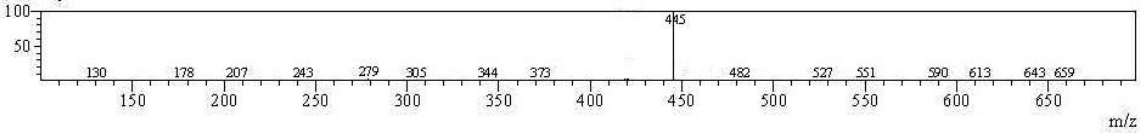
Peak#	Ret. Time	Area	Height	Area %
1	7.506	1796660	289211	100.000
Total		1796660	289211	100.000

MSTABLE MIC1

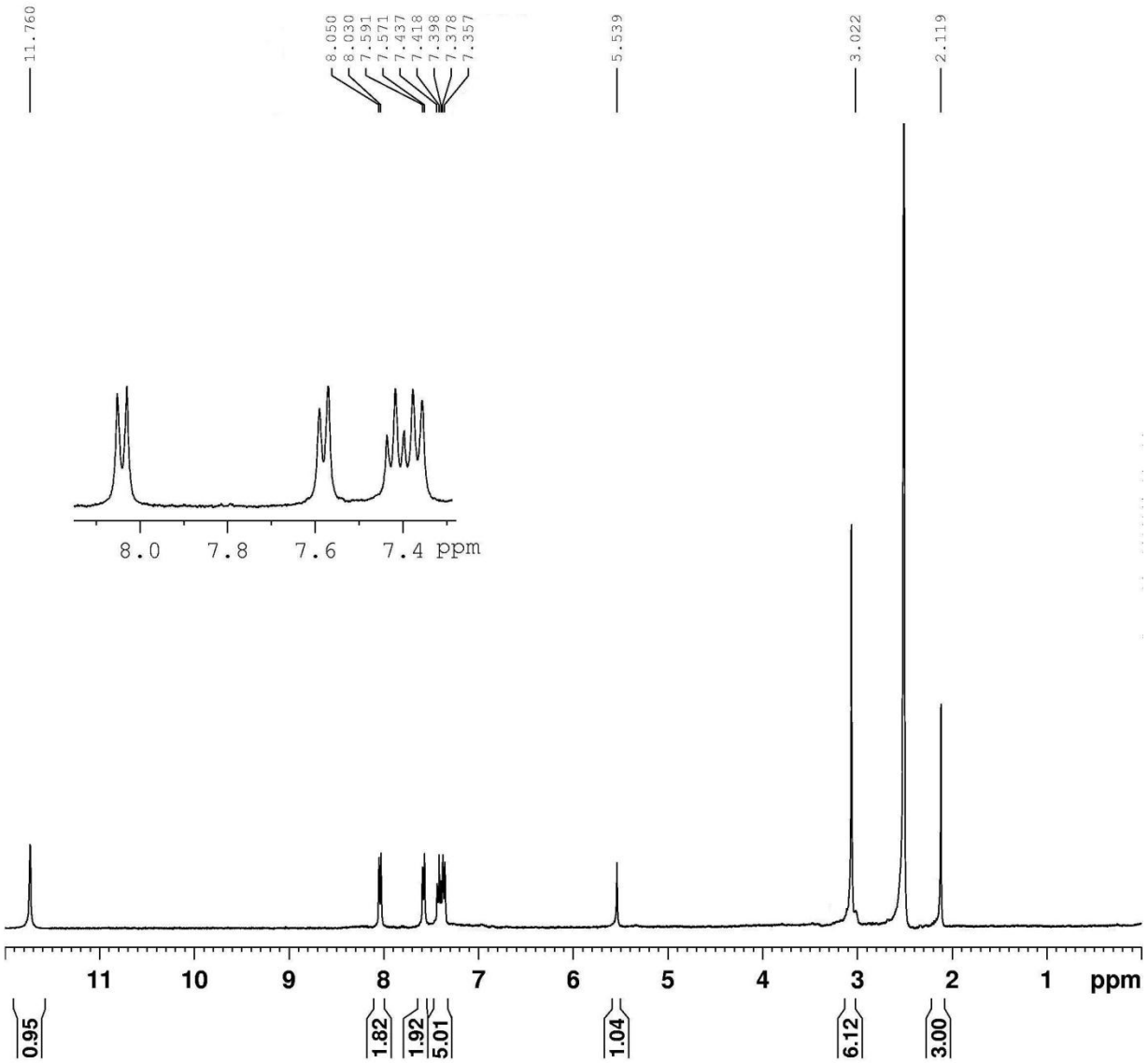
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	7.537	445.10	127246	100.00	1-1
Total			127246	100.00	

MS Spectrum Graph

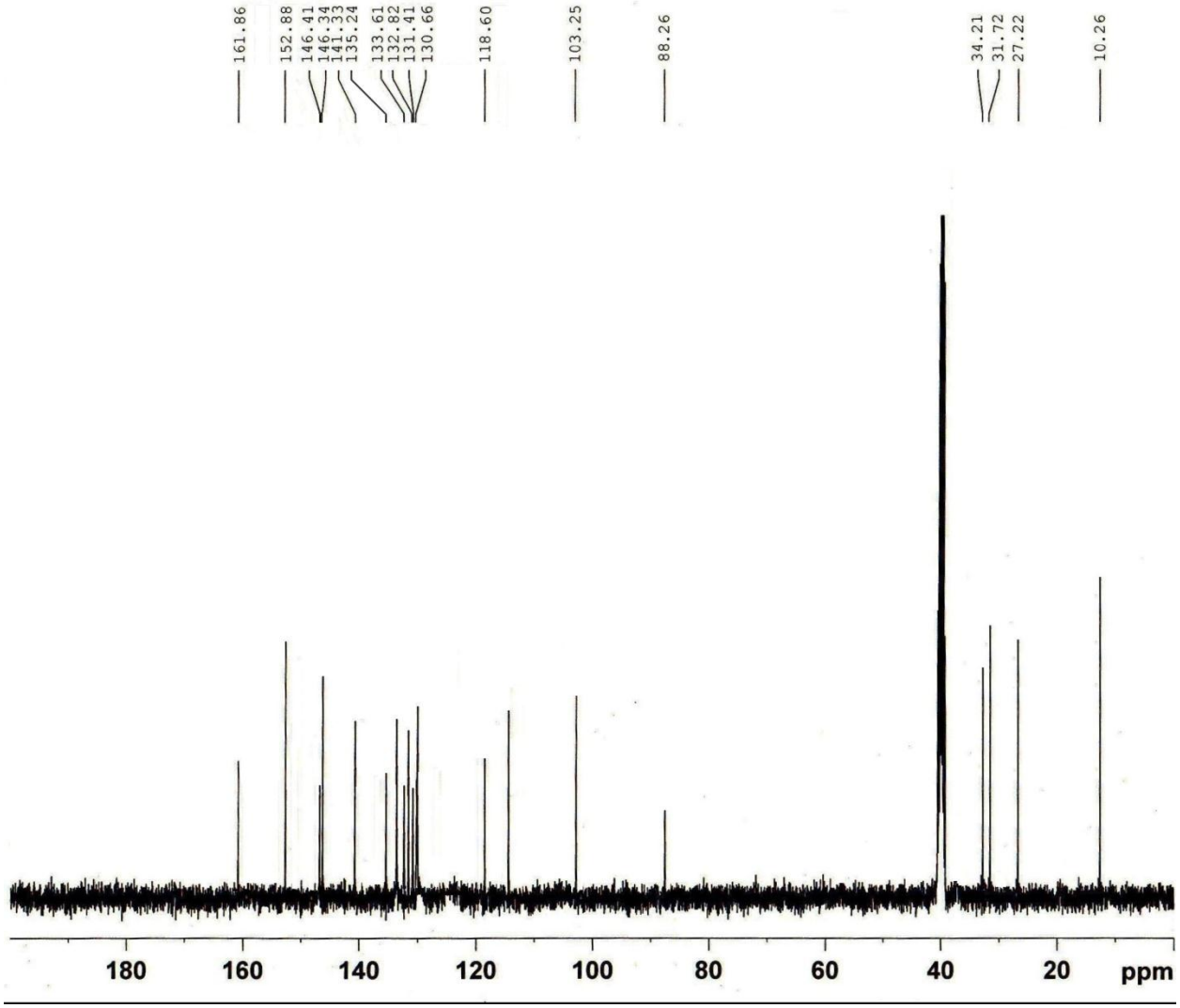
#:1 Ret.Time:Averaged 7.517-7.550(Scan#:452-454)
BG Mode:Calc 7.400<->7.783(445<->468)
Base Peak:445.10(35696) Polarity:Pos Segment1 - Event1



^{1}HNMR of compound **4n**



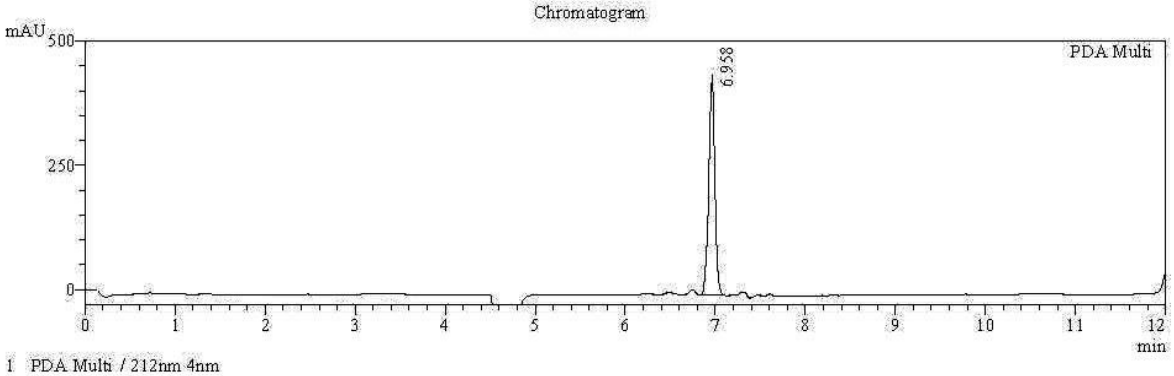
^{13}CNMR of compound **4n**



LC-MS of compound **4o**

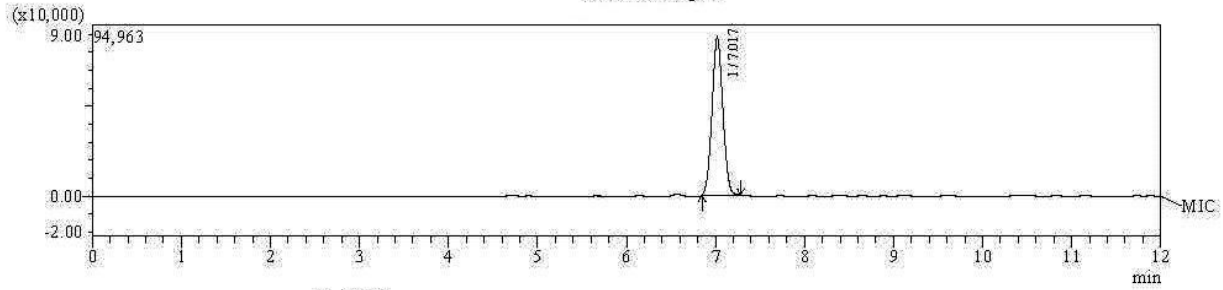
Acquired by : Admin
Sample ID : 22
Vial# : 22
Injection Volume : 10
Data File : OST-J-12-113.lcd
Method file : X-BRIDGE_150_12min_AA-FA.lcm

Chromatogram



1 PDA Multi / 212nm 4nm

MS Chromatogram



PeakTable

PDA Ch1 212nm 4nm

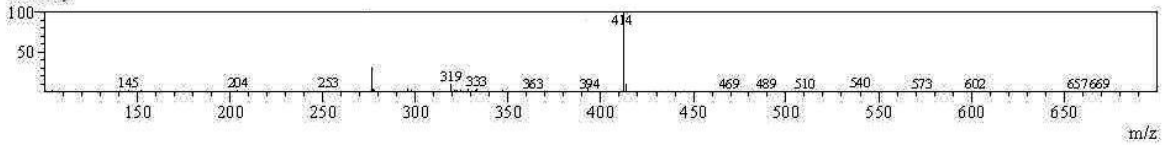
Peak#	Ret. Time	Area	Height	Area%
1	6.958	3161673	654343	100.000
Total		3161673	654343	100.000

MSTABLE MIC1

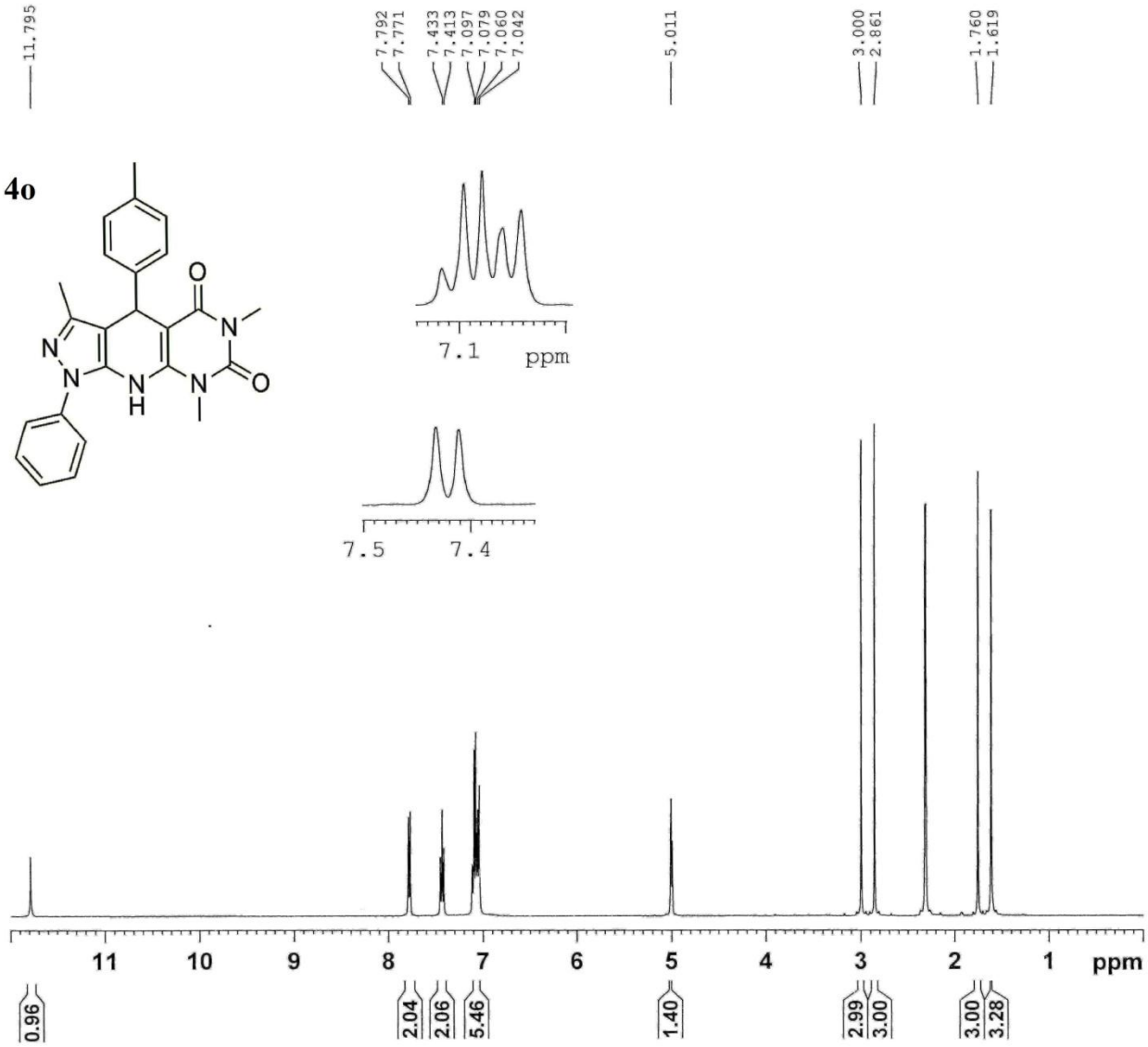
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	7.017	414.35	740099	100.00	1-1
Total			740099	100.00	

MS Spectrum Graph

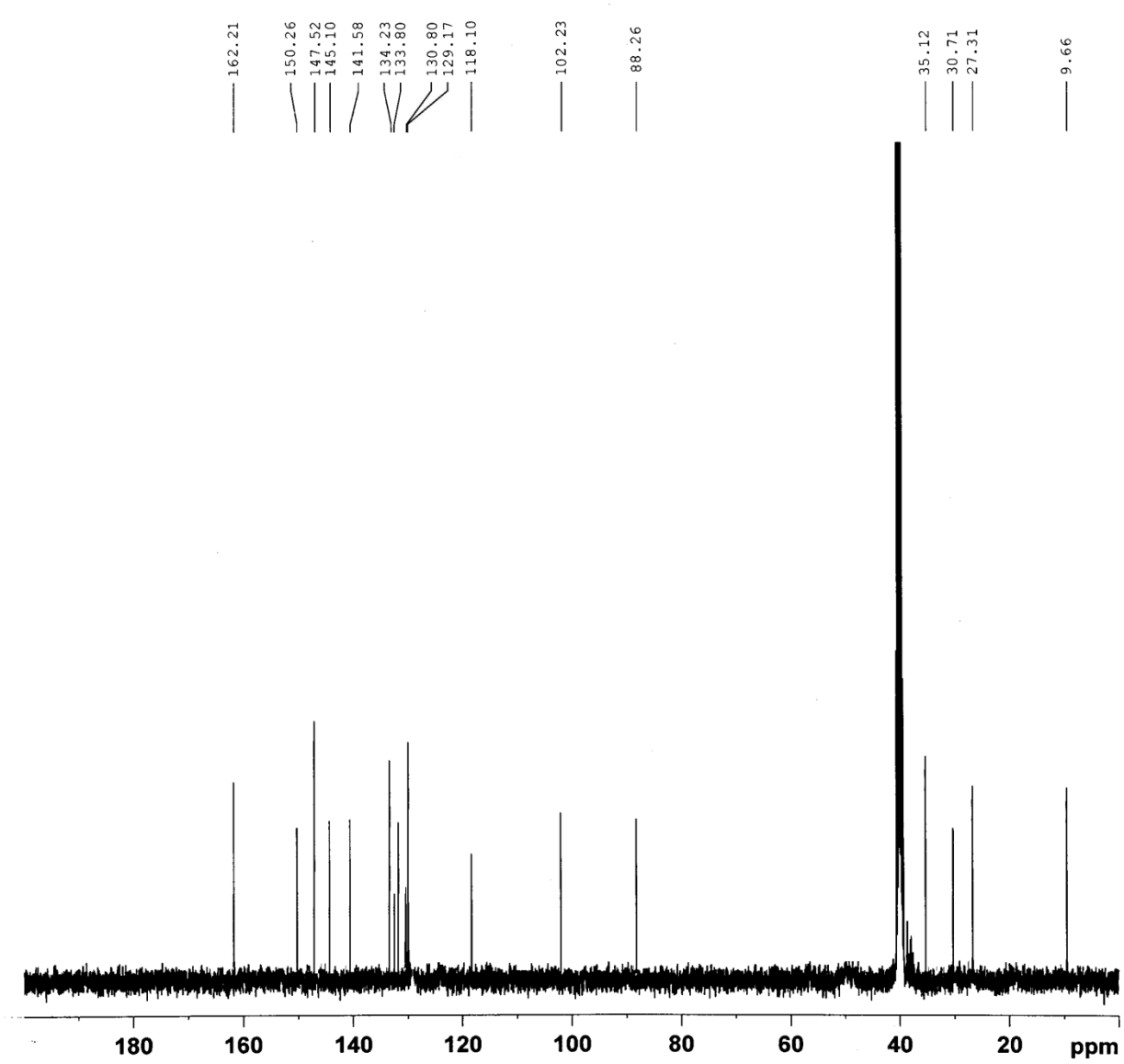
#:1 Ret.Time:Averaged 7.000-7.033(Scan#:421-423)
BG Mode:Calc 6.850<->7.283(412<->438)
Base Peak:414.35(111018) Polarity:Pos Segment1 - Event1
Intensity



^{1}HNMR of compound **4o**



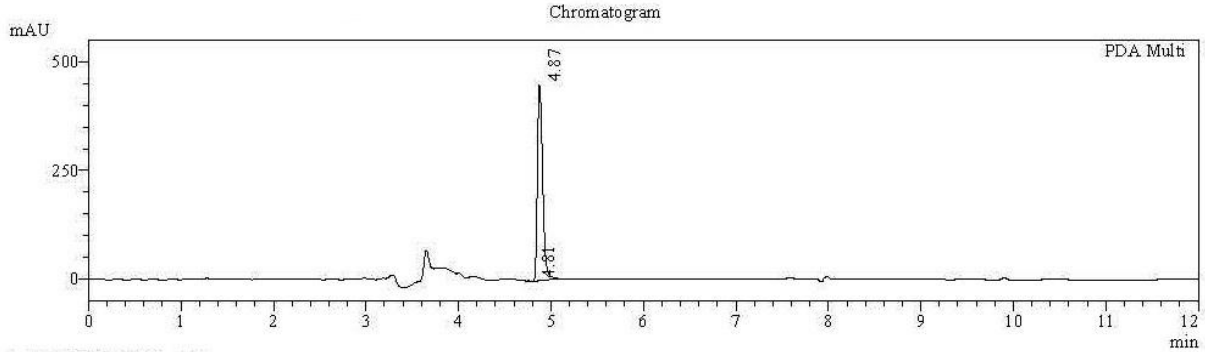
^{13}CNMR of compound **4o**



LC-MS of compound **4p**

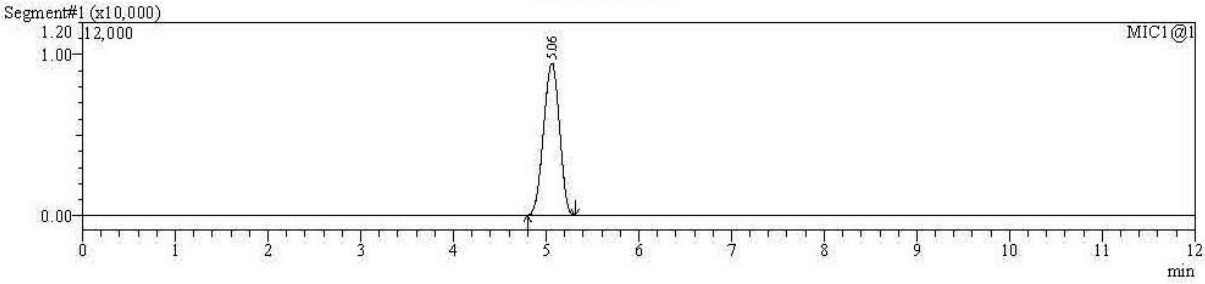
Acquired by	: Admin
Sample ID	: 3
Vial#	: 3
Injection Volume	: 10
Data File	: BDX-615-E-44-FINAL11cd
Method file	: LCMS-ESI1cm

Chromatogram



1 PDA Multi / 260nm 80nm

MS Chromatogram



PeakTable

PDA Ch2 260nm 80nm

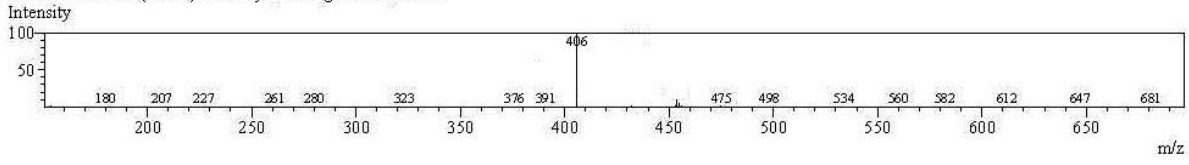
Peak#	Ret. Time	Area	Height	Area %
1	4.81	1001	362	0.06
2	4.87	1767146	449903	99.94
Total		1768147	450265	100.00

MSTABLE MIC1

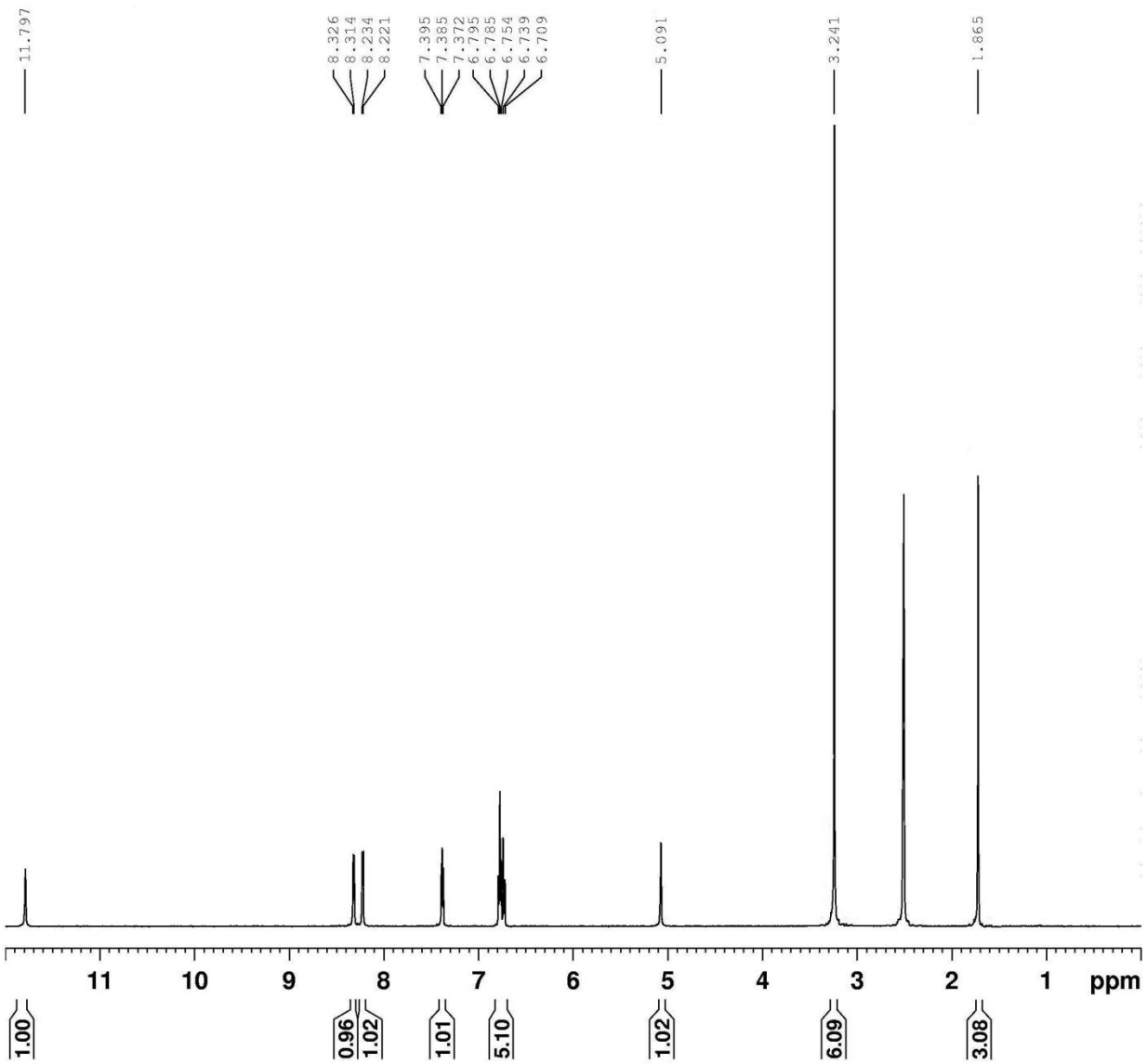
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	5.06	406.21	112778	100.00	1-1
Total			112778	100.00	

MS Spectrum Graph

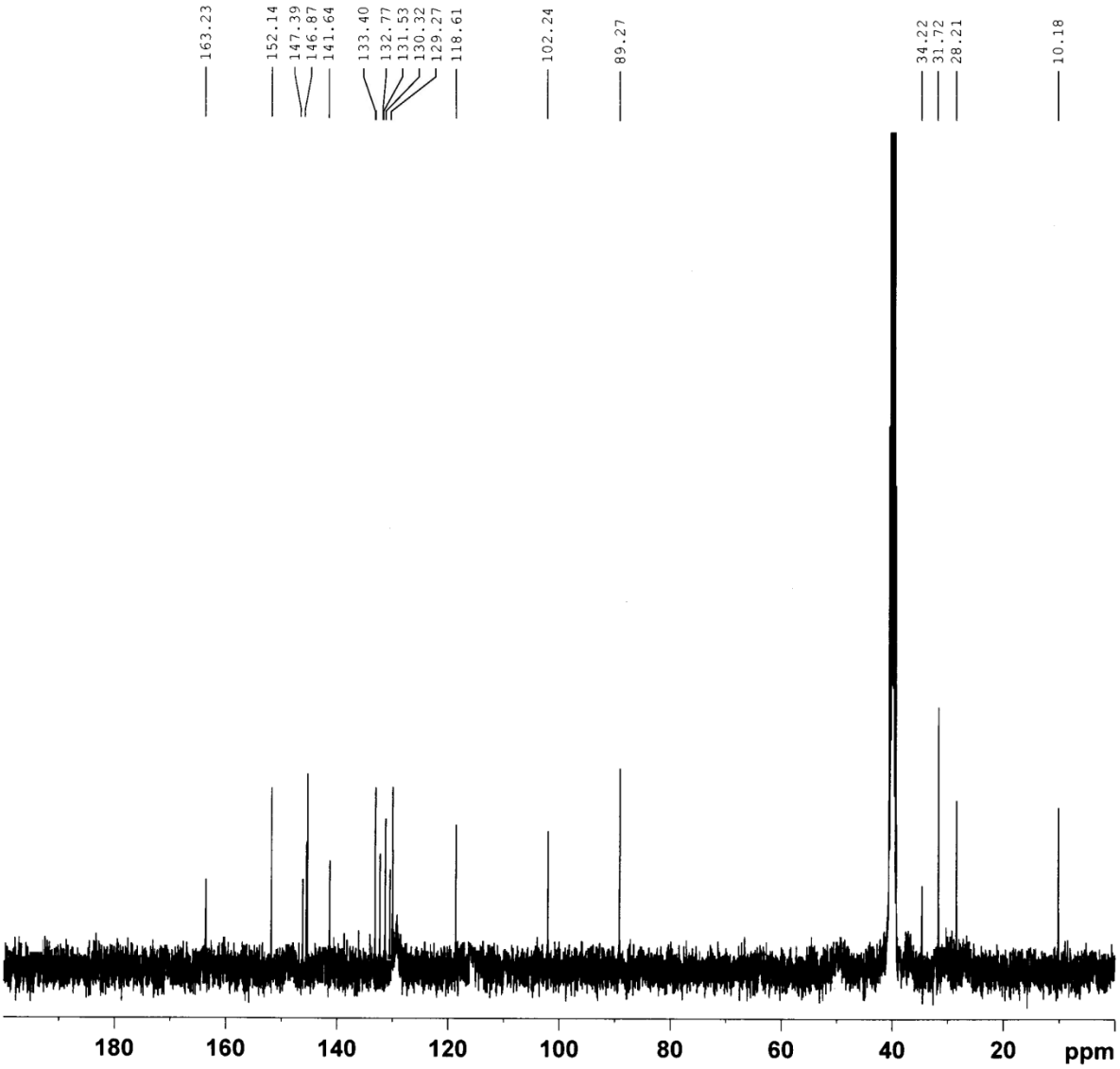
#:1 Ret.Time:Averaged 5.033-5.067(Scan#:303-305)
BG Mode:Calc 4.800<->5.317(289<->320)
Base Peak:406.21(34162) Polarity:Pos Segment1 - Event1



^{1}HNMR of compound **4p**



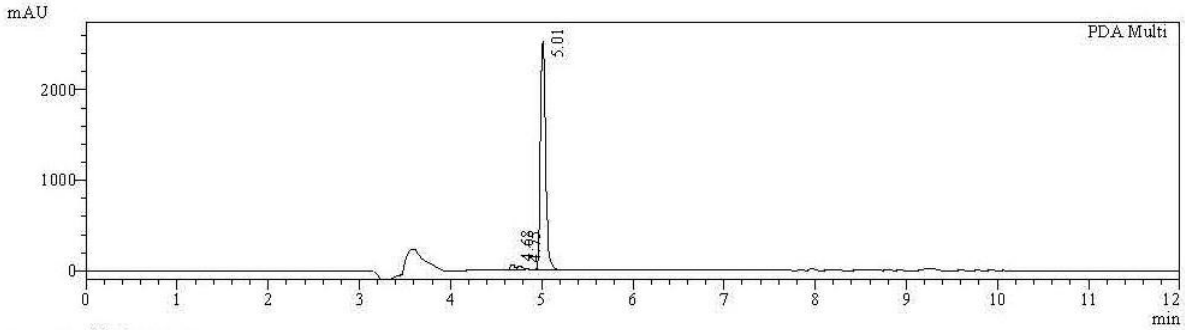
^{13}CNMR of compound **4p**



LC-MS of compound **4q**

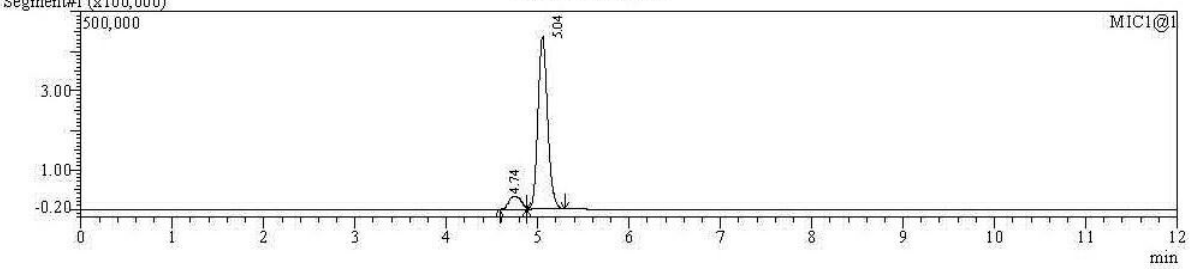
Acquired by : Admin
Sample ID : 2
Vial# : 2
Injection Volume : 6
Data File : O2H-236-HCL
Method file : LCMS-ESI1cm

Chromatogram



1 PDA Multi / 211nm 4nm

MS Chromatogram



PeakTable

PDA Ch1 211nm 4nm

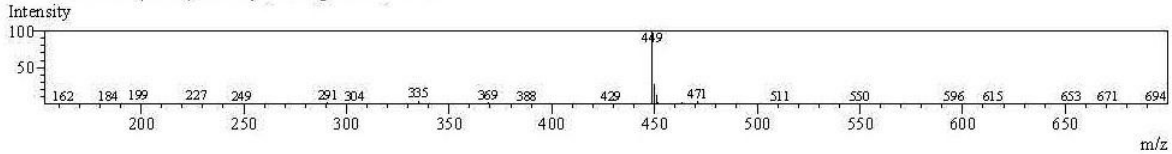
Peak#	Ret. Time	Area	Height	Area %
1	4.68	177109	55267	1.74
2	4.75	125912	39077	1.24
3	5.01	9865967	2524600	97.02
Total		10168988	2618944	100.00

MSTABLE MIC1

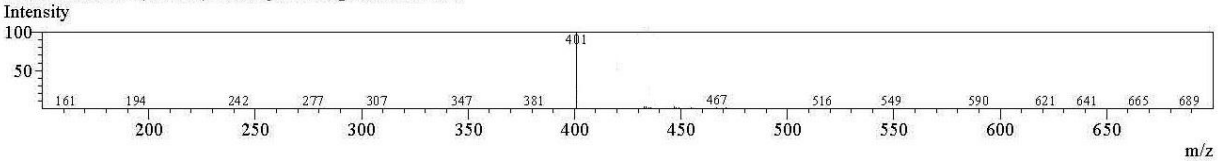
Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	4.74	448.80	304225	8.67	1-1
2	5.04	401.34	3203356	91.33	1-1
Total			3507581	100.00	

MS Spectrum Graph

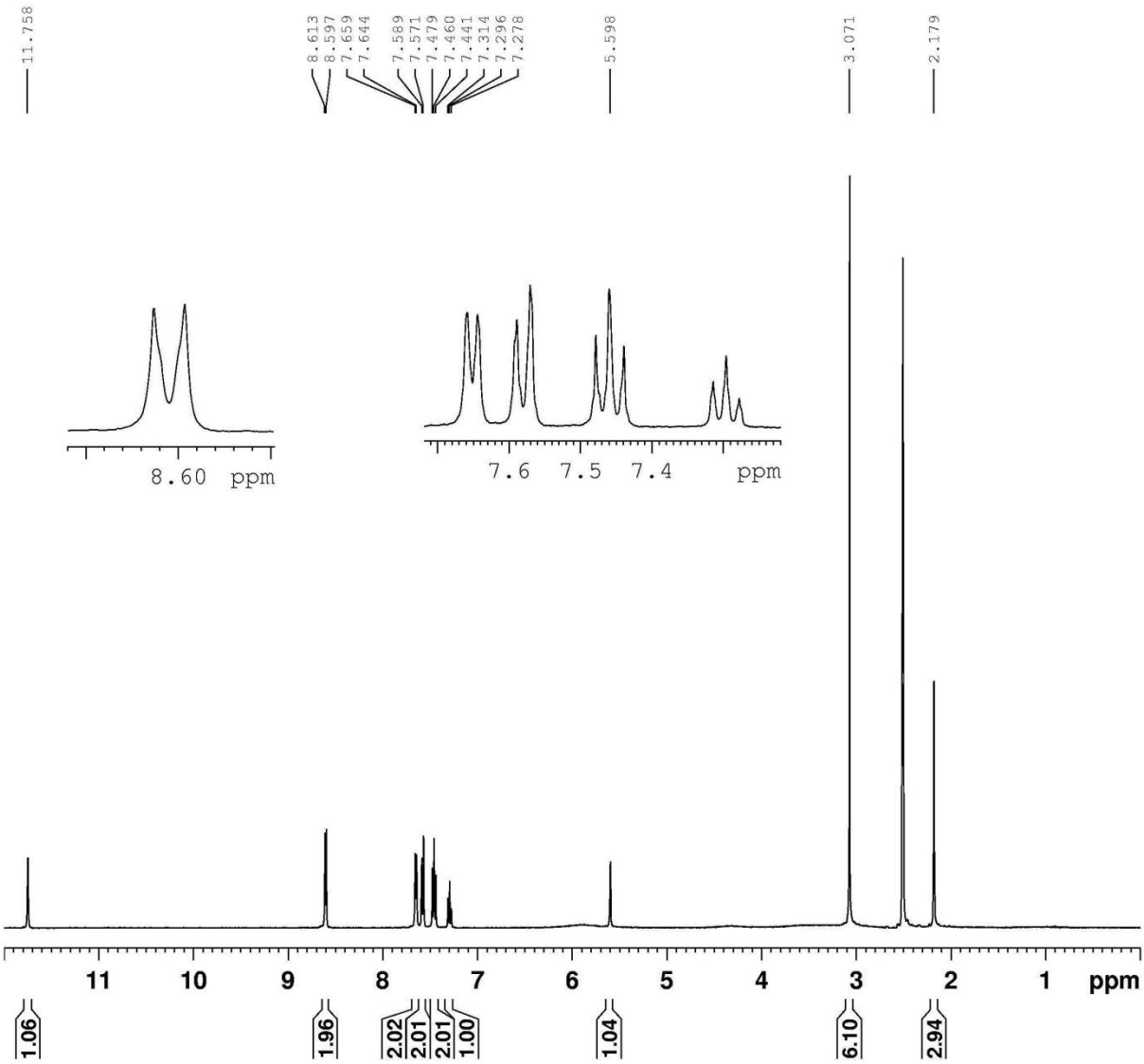
#:1 Ret.Time:Averaged 4.717-4.750(Scan#:284-286)
BG Mode:Calc 4.583<->4.883(276<->294)
Base Peak:448.80(32998) Polarity:Pos Segment1 - Event1
Intensity



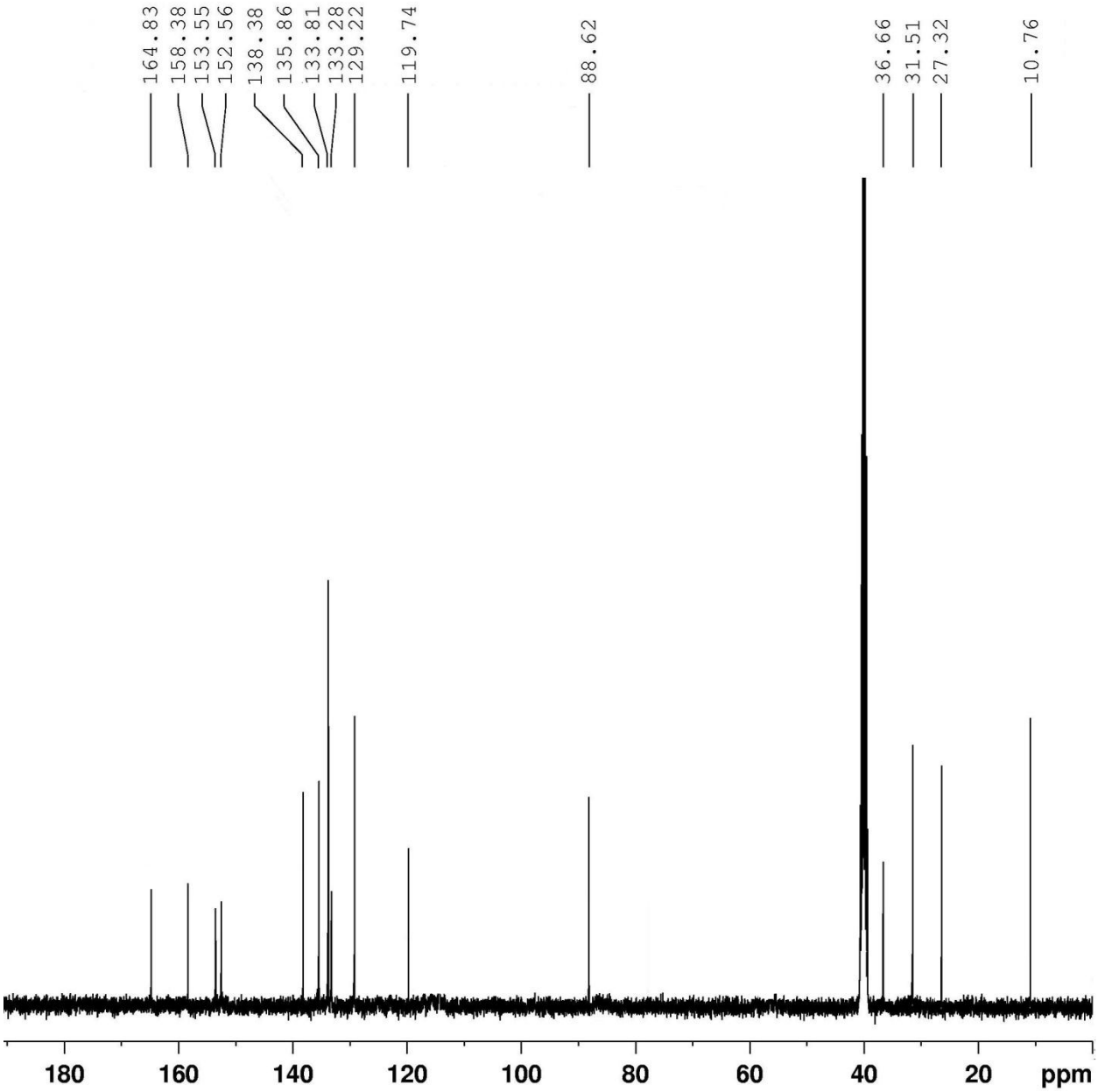
#:2 Ret.Time:Averaged 5.033-5.067(Scan#:303-305)
BG Mode:Calc 4.883<->5.300(294<->319)
Base Peak:401.34(507790) Polarity:Pos Segment1 - Event1
Intensity



^{1}HNMR of compound **4q**



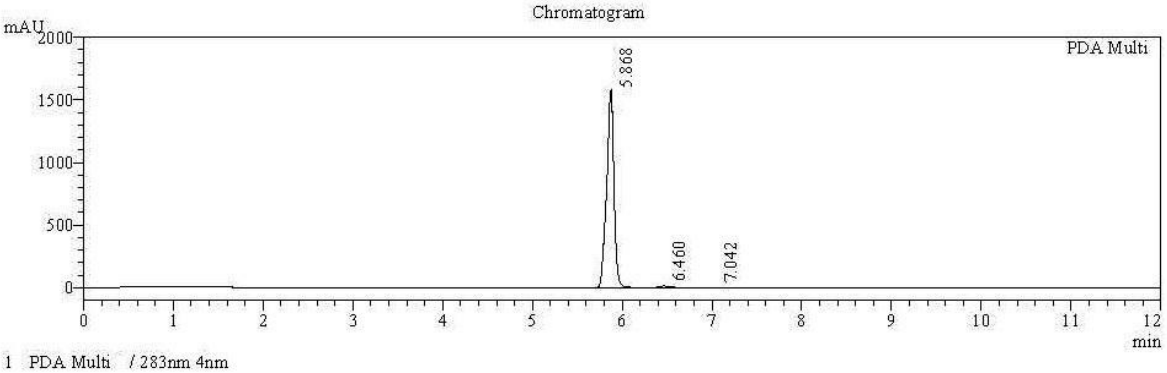
^{13}CNMR of compound **4q**



LC-MS of compound **4r**

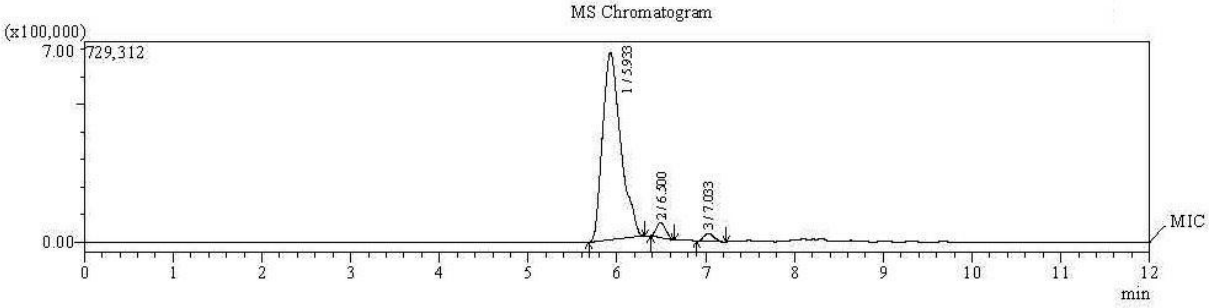
Acquired by	: Admin
Sample ID	: O2Hr
Vial#	: 25
Injection Volume	: 10
Data File	: OST-J-11-84-01-127 .lcd
Method file	: X-BRIDGE_150_12min_AA-FA.lcm

Chromatogram



1 PDA Multi / 283nm 4nm

MS Chromatogram



PeakTable

PDA Ch1 283nm 4nm

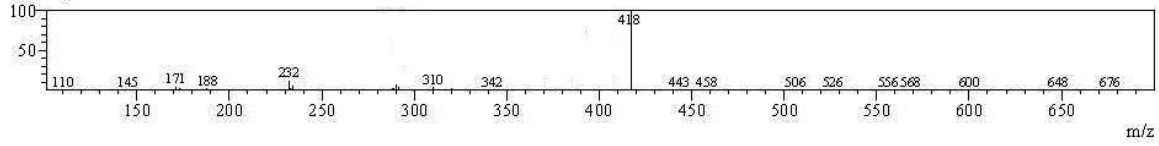
Peak#	Ret. Time	Area	Height	Area %
1	5.868	8841872	1575526	99.049
2	6.460	82816	11704	0.928
3	7.042	2064	740	0.023
Total		8926752	1587970	100.000

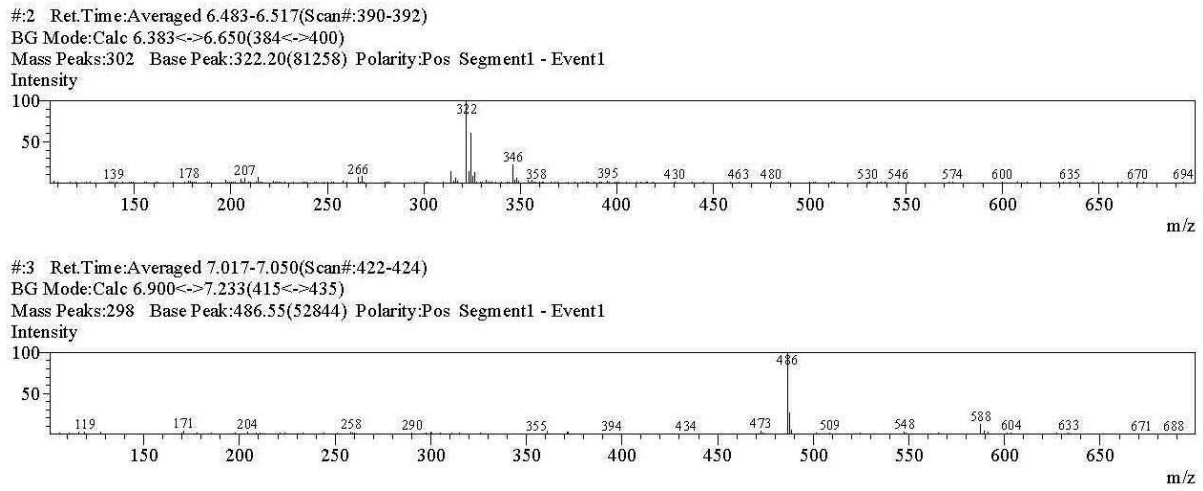
MSTABLE MIC1

Peak#	Ret.Time	Base Peak m/z	Area	Area%	Event#
1	5.933	418.20	9641423	93.92	1-1
2	6.500	322.20	397415	3.87	1-1
3	7.033	486.55	227350	2.21	1-1
Total			10266188	100.00	

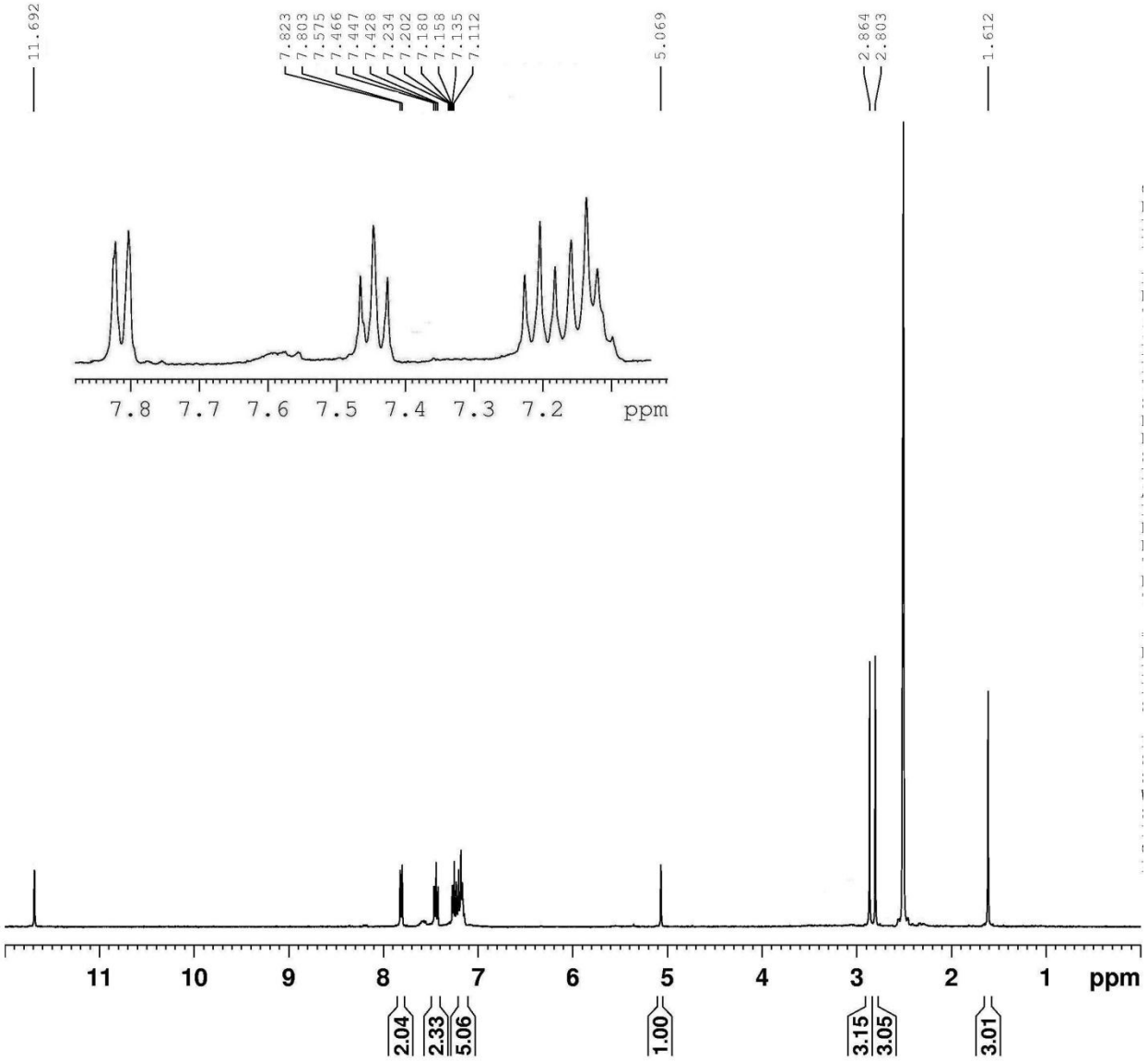
MS Spectrum Graph

#1 Ret.Time:Averaged 5.917-5.950(Scan#:356-358)
BG Mode:Calc 5.683<->6.317(342<->380)
Base Peak:418.20(751691) Polarity:Pos Segment1 - Event1
Intensity

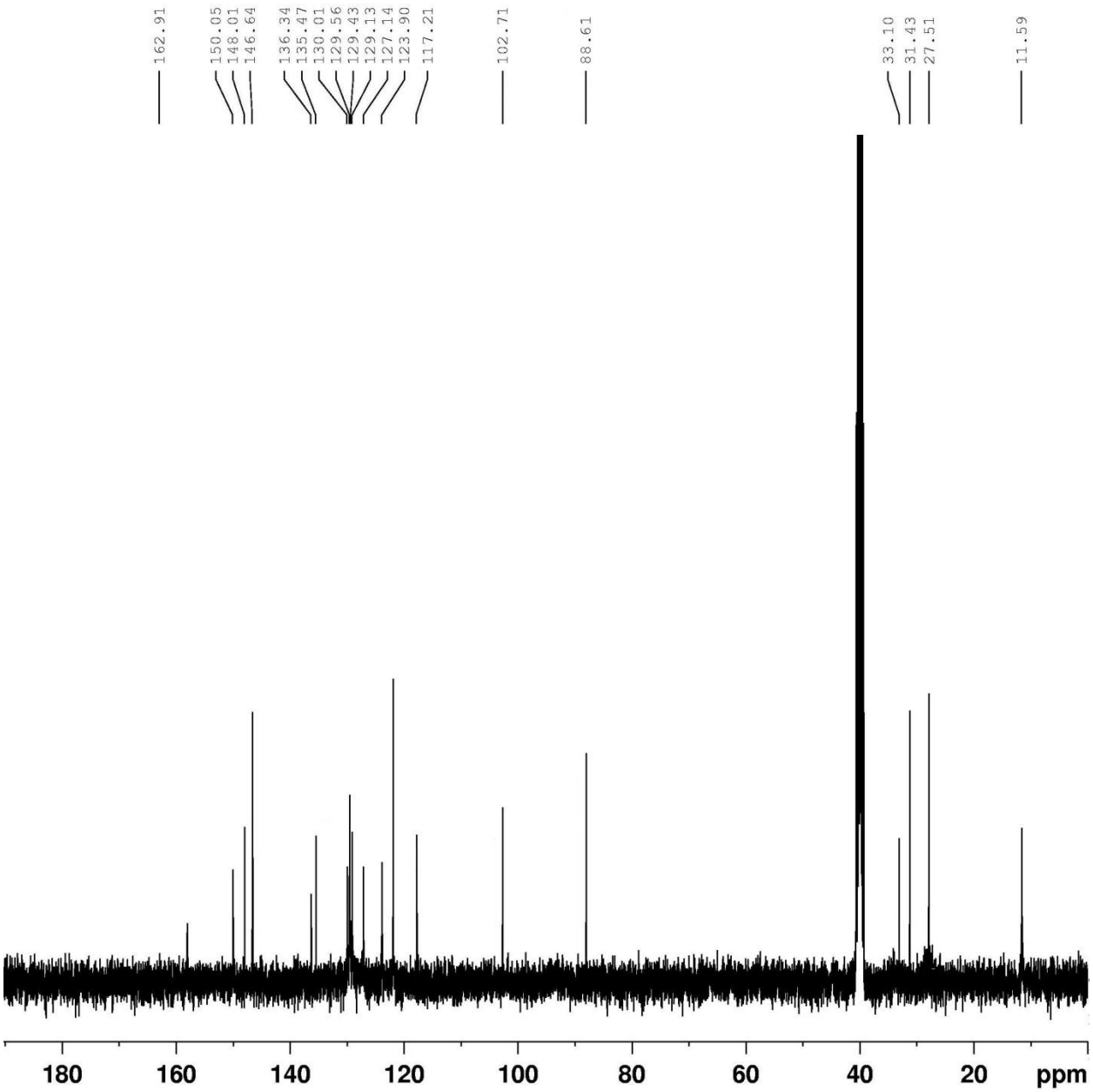




^{1}HNMR of compound **4r**



^{13}CNMR of compound **4r**



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[1] A. Rattan, *in: B. I. Churchill, Antimicrobials in Laboratory Medicine. Livingstone, New Delhi*, 2000, 85.

[2] W. Trager, J.B. Jensen, *Science*, 1976, **193,** 673.

[3] L.H. Carvalho, A.U. Krettli, *Mem. Inst. Oswaldo. Cruz. (Suppl. II)* **86,** 181.

[4] C. Lambros, J.P. van der Berg, J. Parasitol. 1979, **65,** 418.