

Supplementary Material

**Microwave-Assisted synthesis of 2,8-di(alkyl/ aryl)-4,6-dichloro-2,8-dihydropyrano[3,2-g]
chromene-3,7-dicarbaldehydes and their antimicrobial activity**

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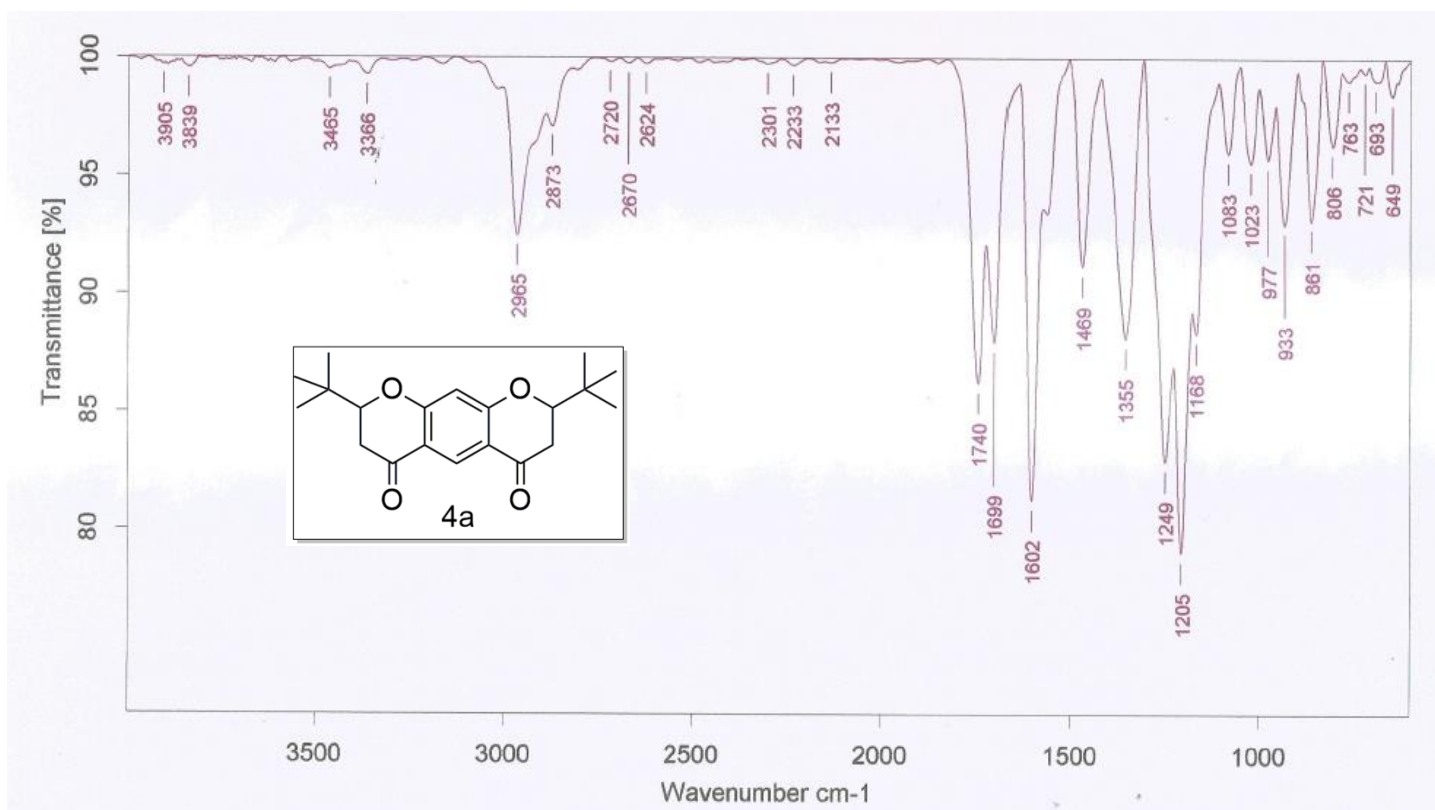


Figure 1: IR data of 2,8-di-tert-butyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione (4a)

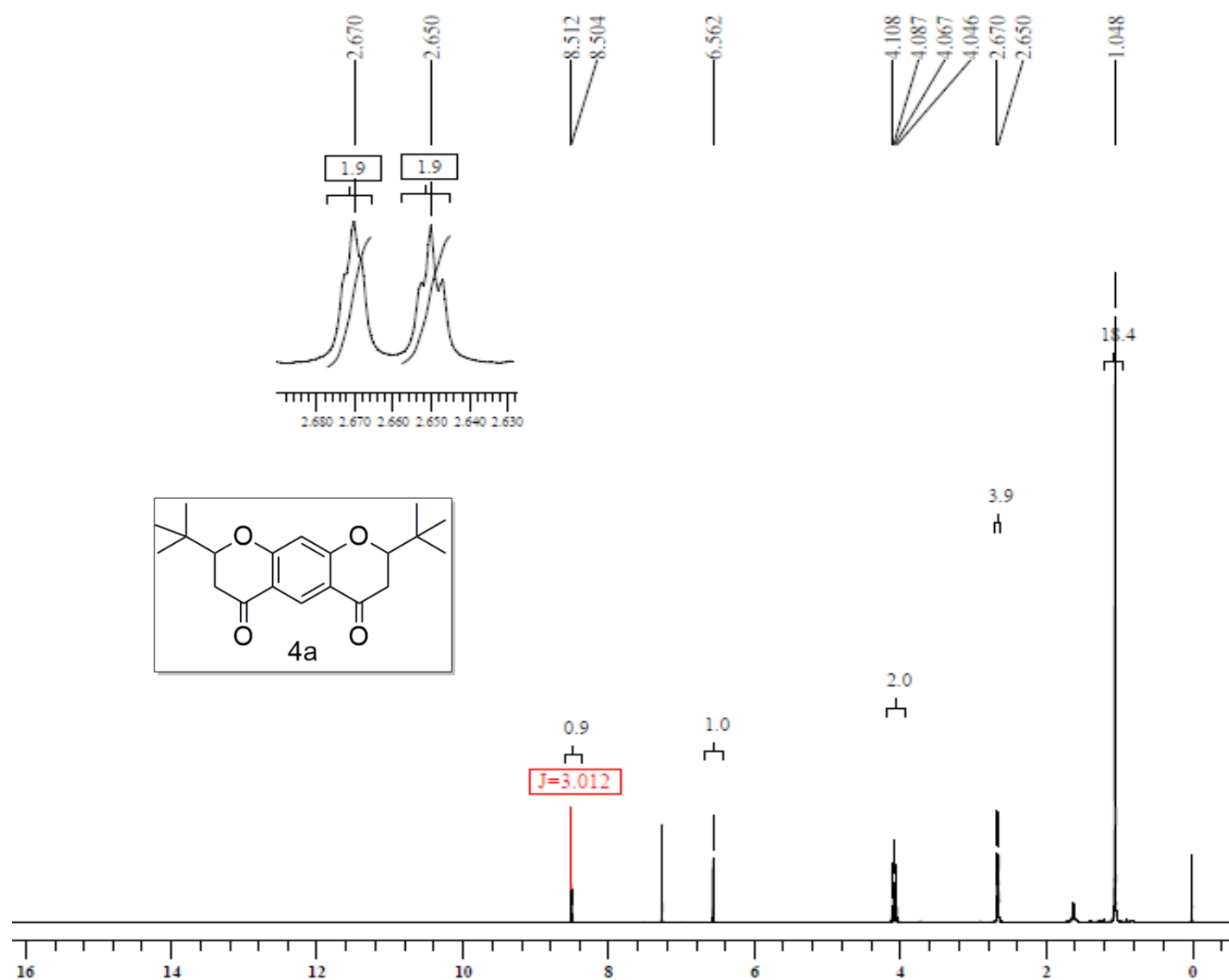


Figure -2: ^1H NMR of 2,8-di-tert-butyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione (4a).

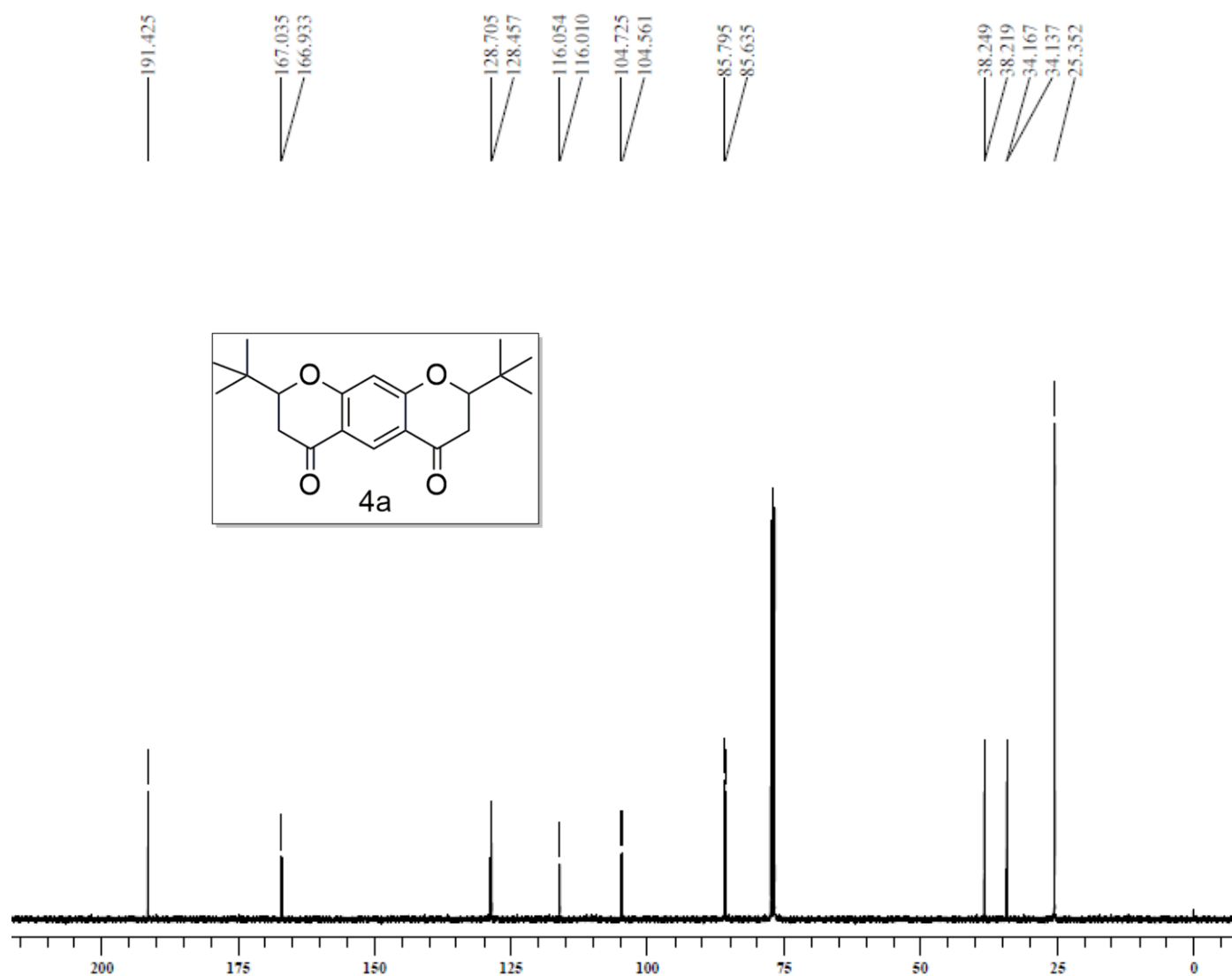
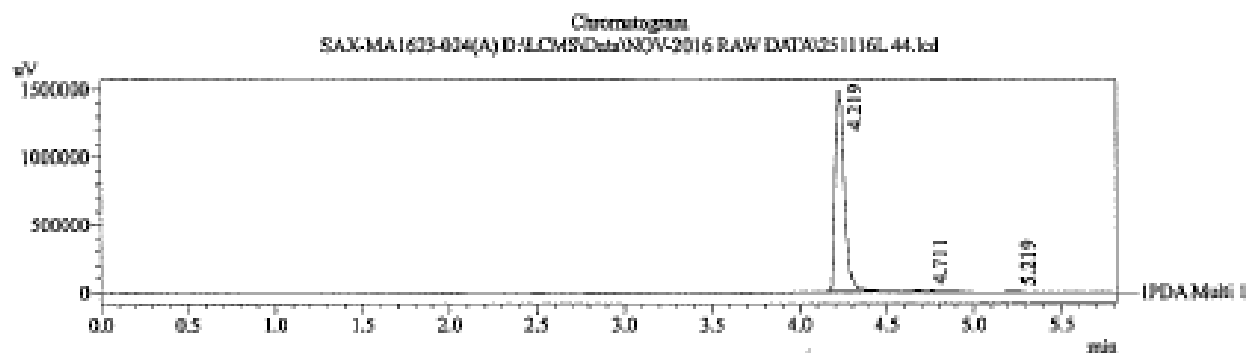
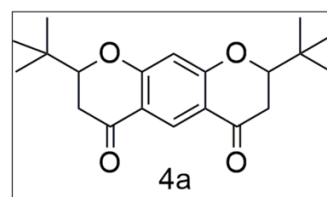


Figure 3: ^{13}C NMR of 2,8-di-tert-butyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione (4a).

Tuning File :D:\LCMS\Tuning Files\Tuning-ESI-15092014.lct
Vial No :25
Description :Kinetex EVO C-18 (50 X3.0mm,2.6um)
Mobile Phase : A :2.5mM NH₄OOCH in water+5%ACN,
B:ACN+ 5% 2.5mM NH₄OOCH in Water
T/B% :0.01/5,4/95,5.5/95
Flow : 0.8ml / min(Gradient).

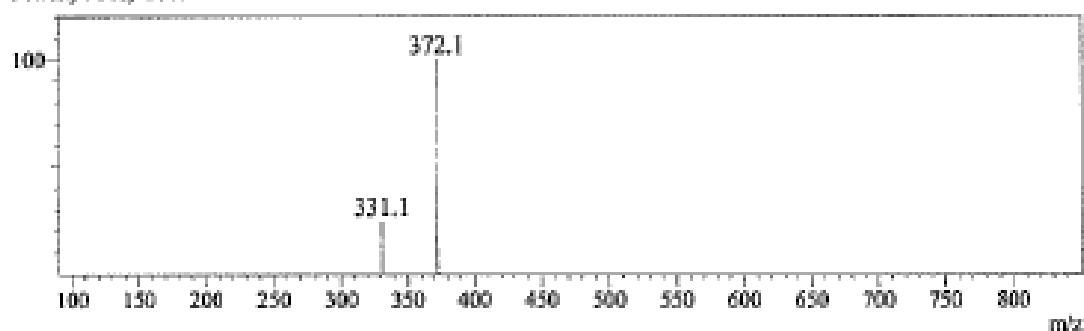


1 PDA, Multi I / 250nm 4nm

MS Spectrum Graph

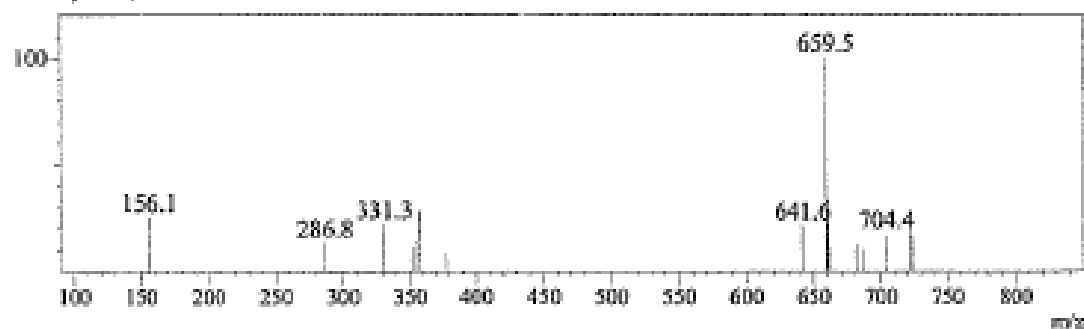
Ret. Time:4.200-4.267

Polarity : Pos, Base Peak m/z : 372.1



Ret. Time:4.700-4.767

Polarity : Pos, Base Peak m/z : 659.5



PeakTable

PDA, Ch1 250nm 4nm

Peak#	Ret. Time	Area	Area %
1	4.219	5312277	97.294
2	4.711	125241	2.294
3	5.219	22483	0.412
Total		5460000	100.000

Figure 4: LCMS of 2,8-di-tert-butyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione (4a)



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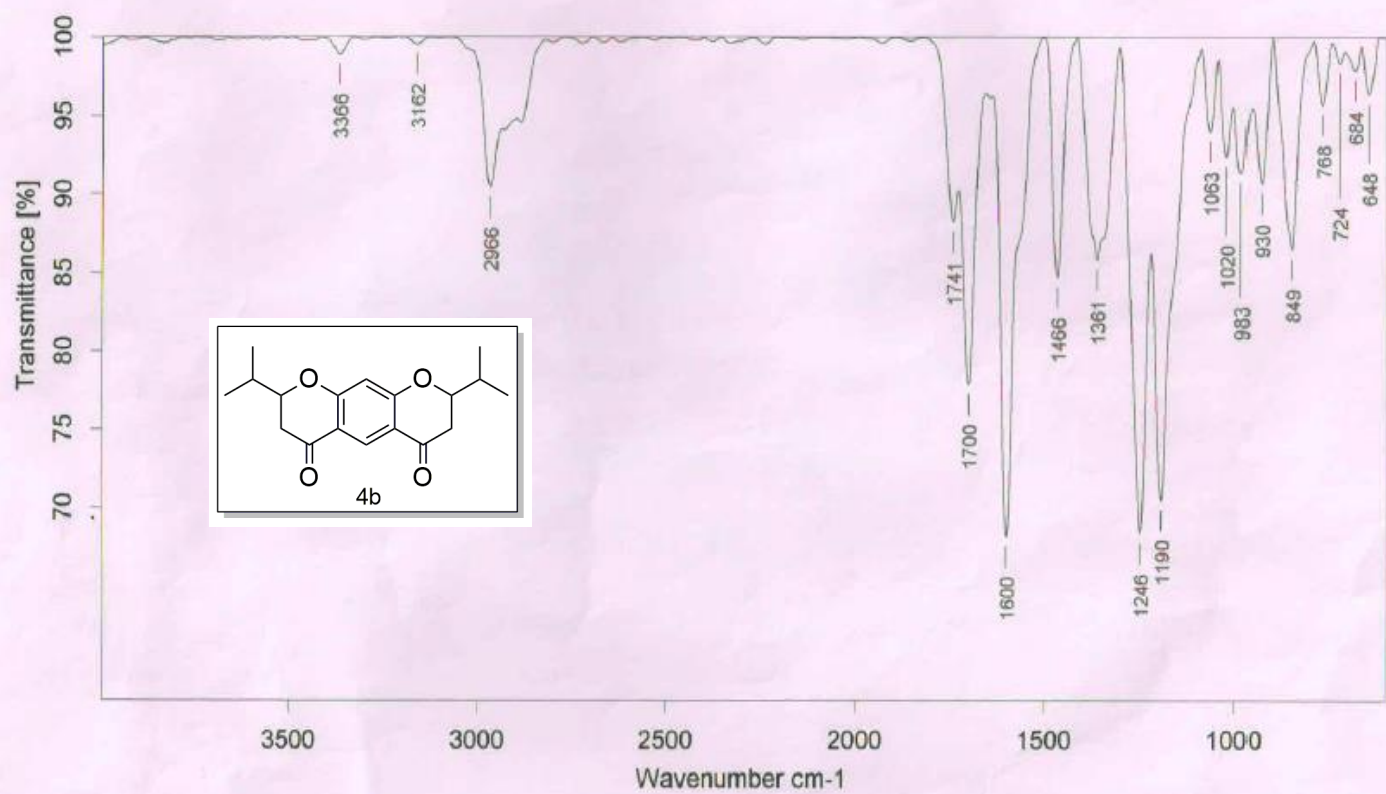


Figure 5: IR data of 2,8-diisopropyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione(4b)

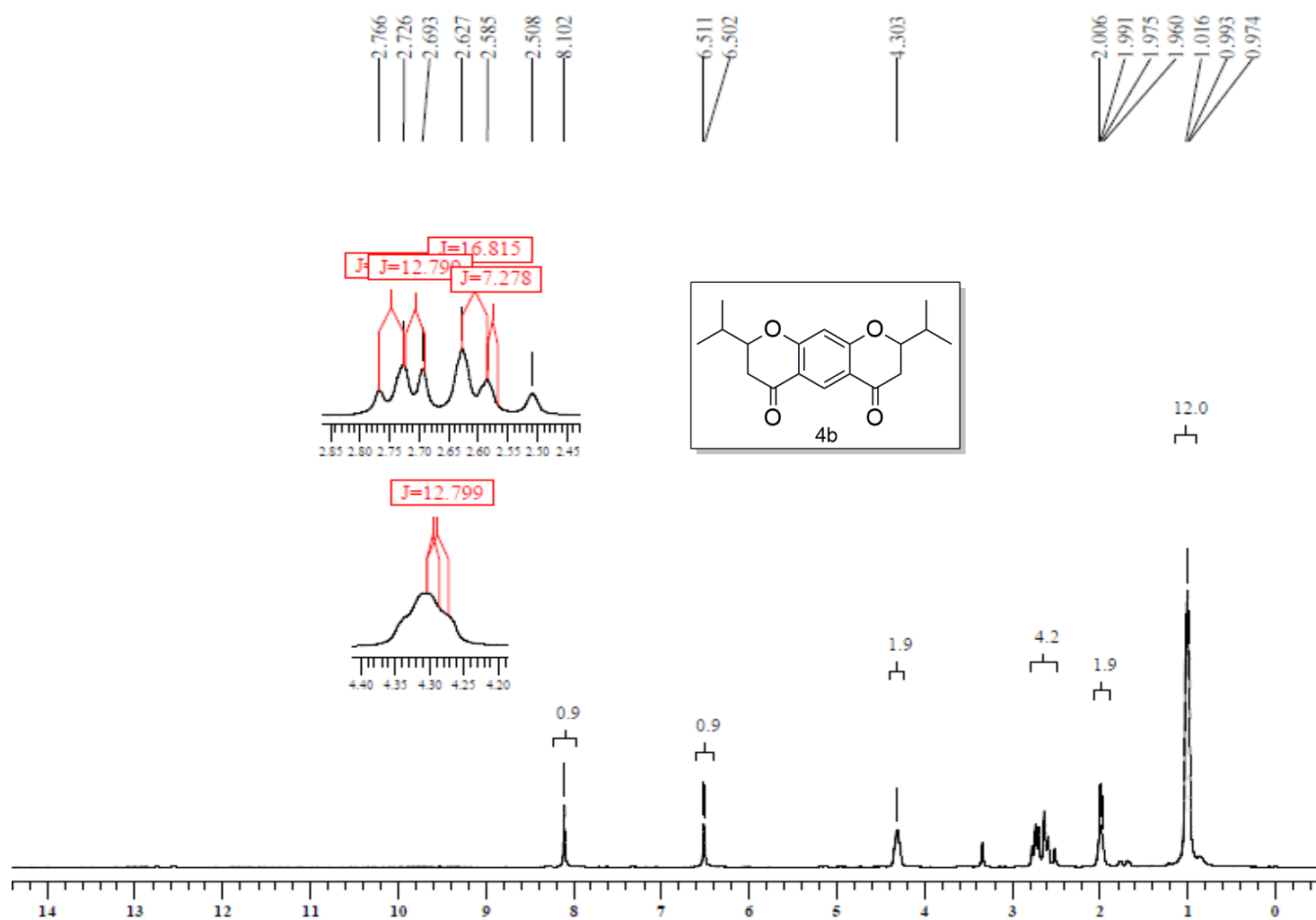


Figure 6: ^1H NMR of 2,8-diisopropyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione(4b)

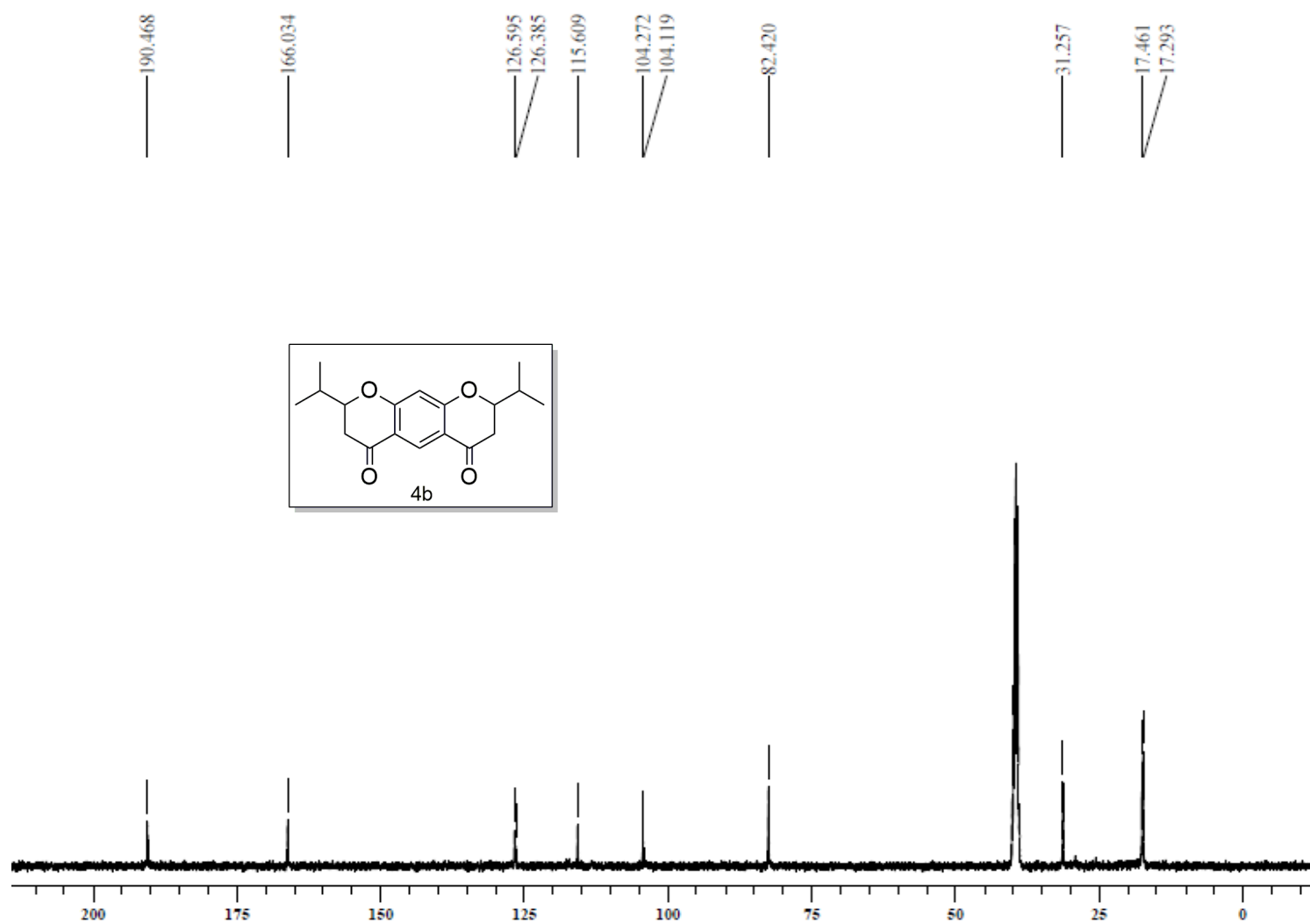


Figure 7: ¹³C NMR of 2,8-diisopropyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione(4b)

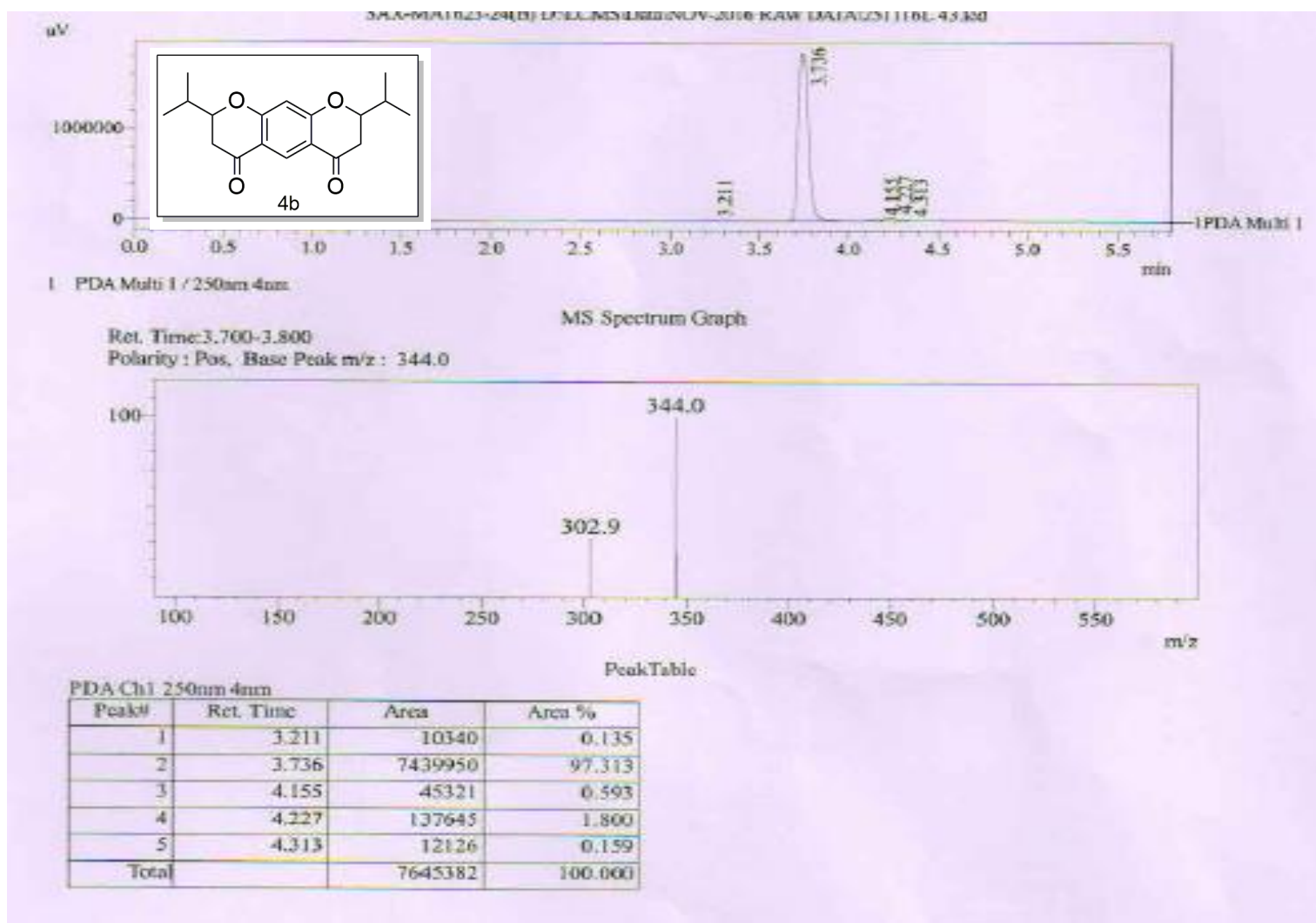


Figure 8: LCMS of 2,8-diisopropyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione (4b)

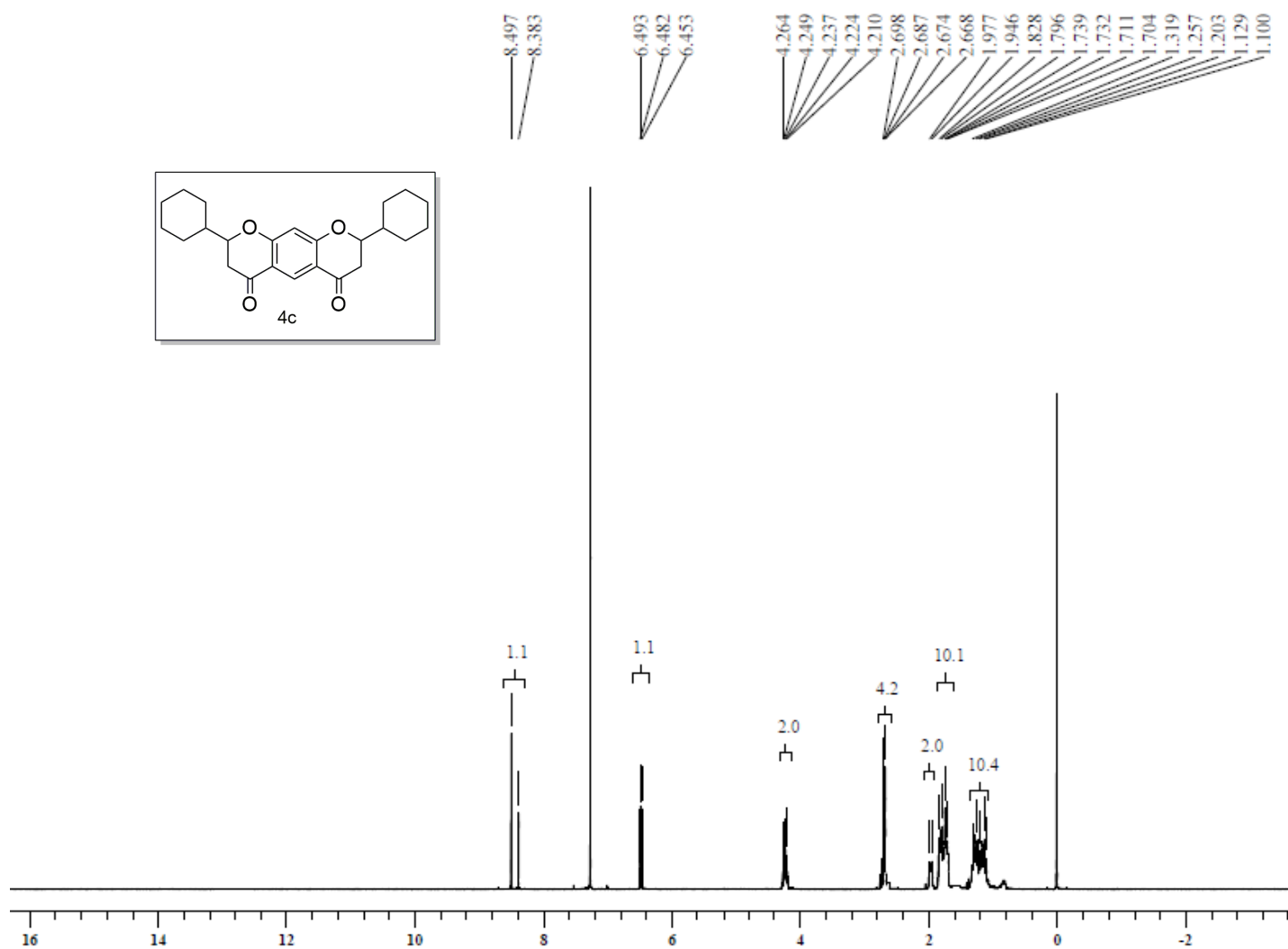


Figure 9: ^1H NMR of 2,8-dicyclohexyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione(4c)

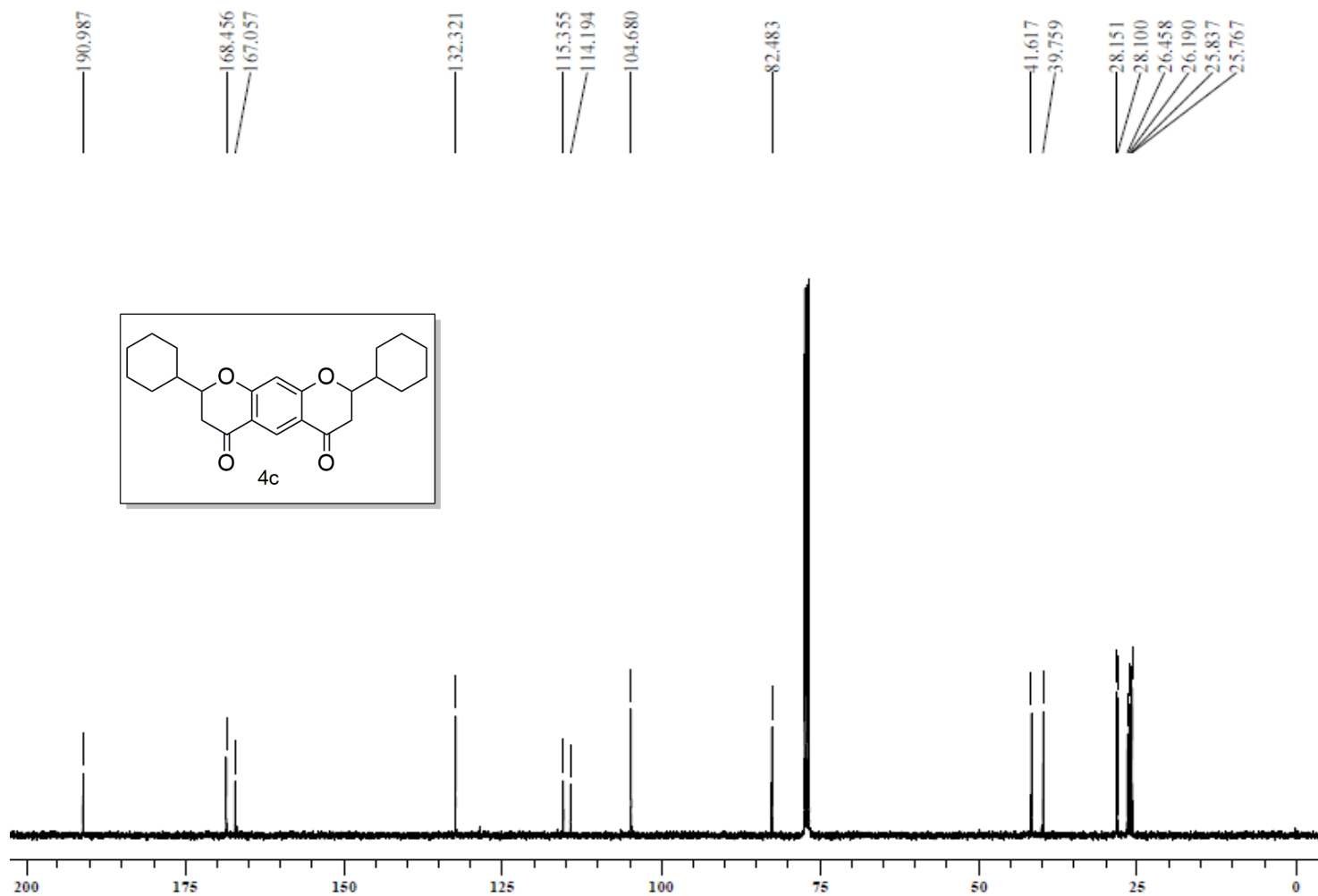


Figure 10: ¹³C NMR of 2,8-dicyclohexyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione(4c)

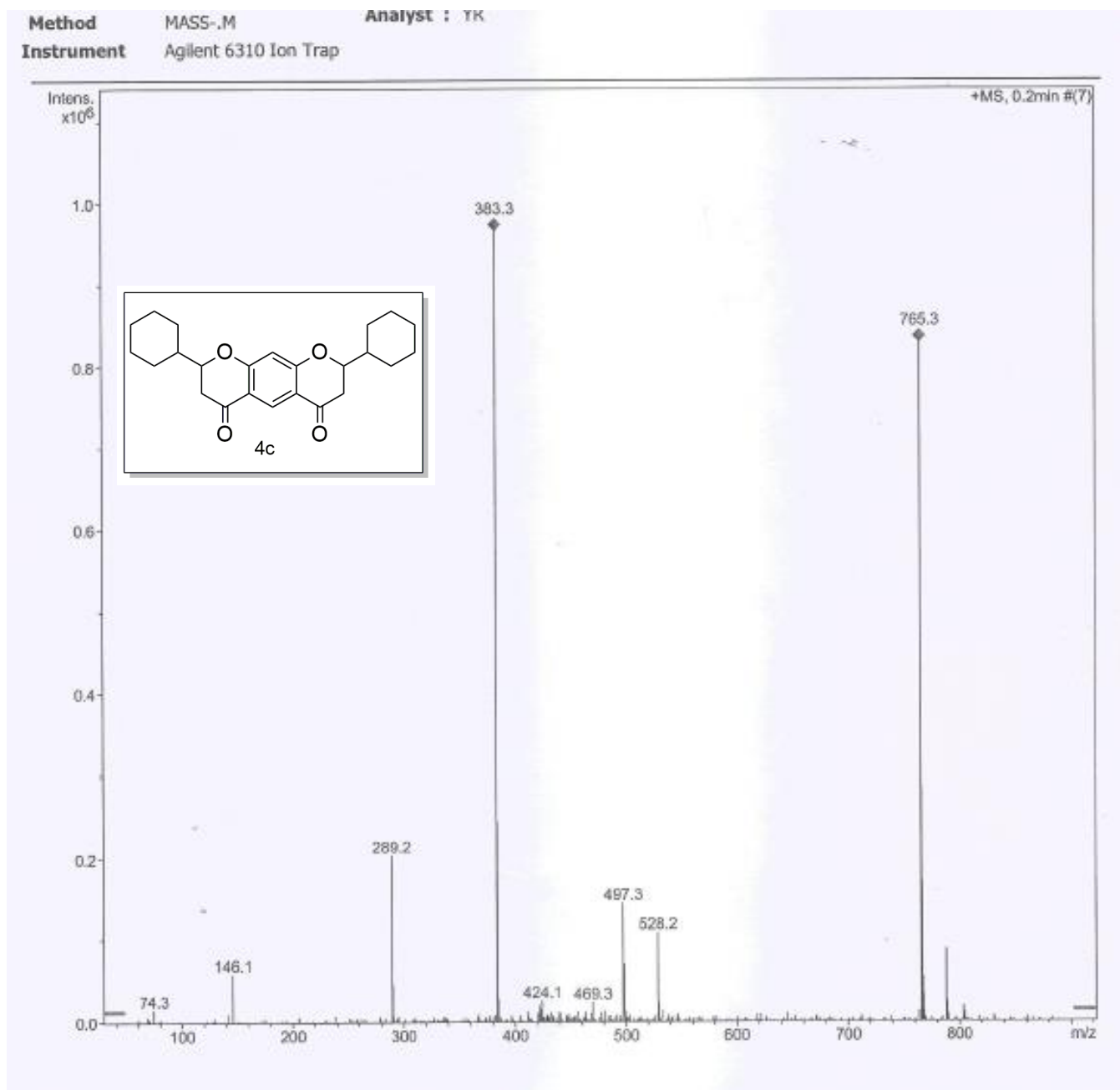


Figure 11: Mass data of 2,8-dicyclohexyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione(4c)



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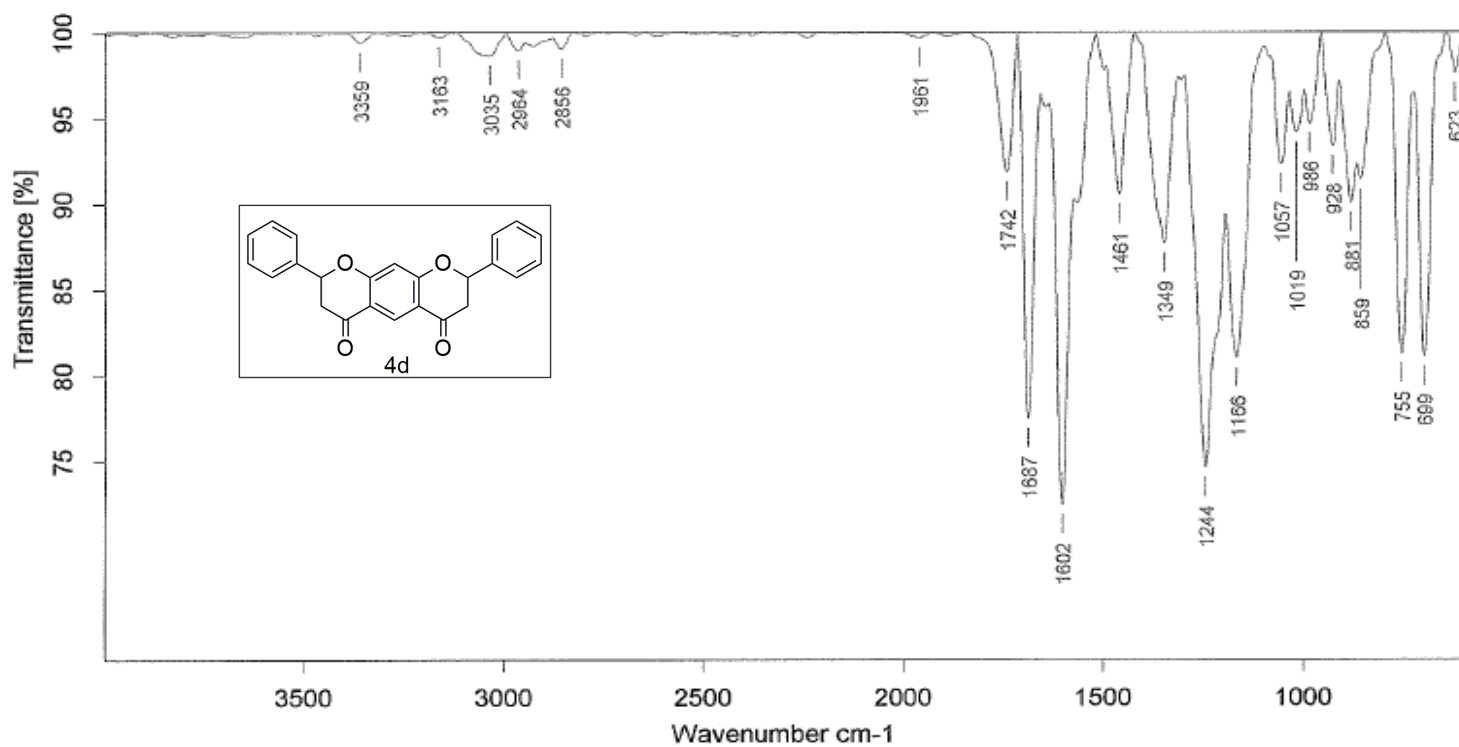


Figure 12: IR data of 2,8-diphenyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione (4d)

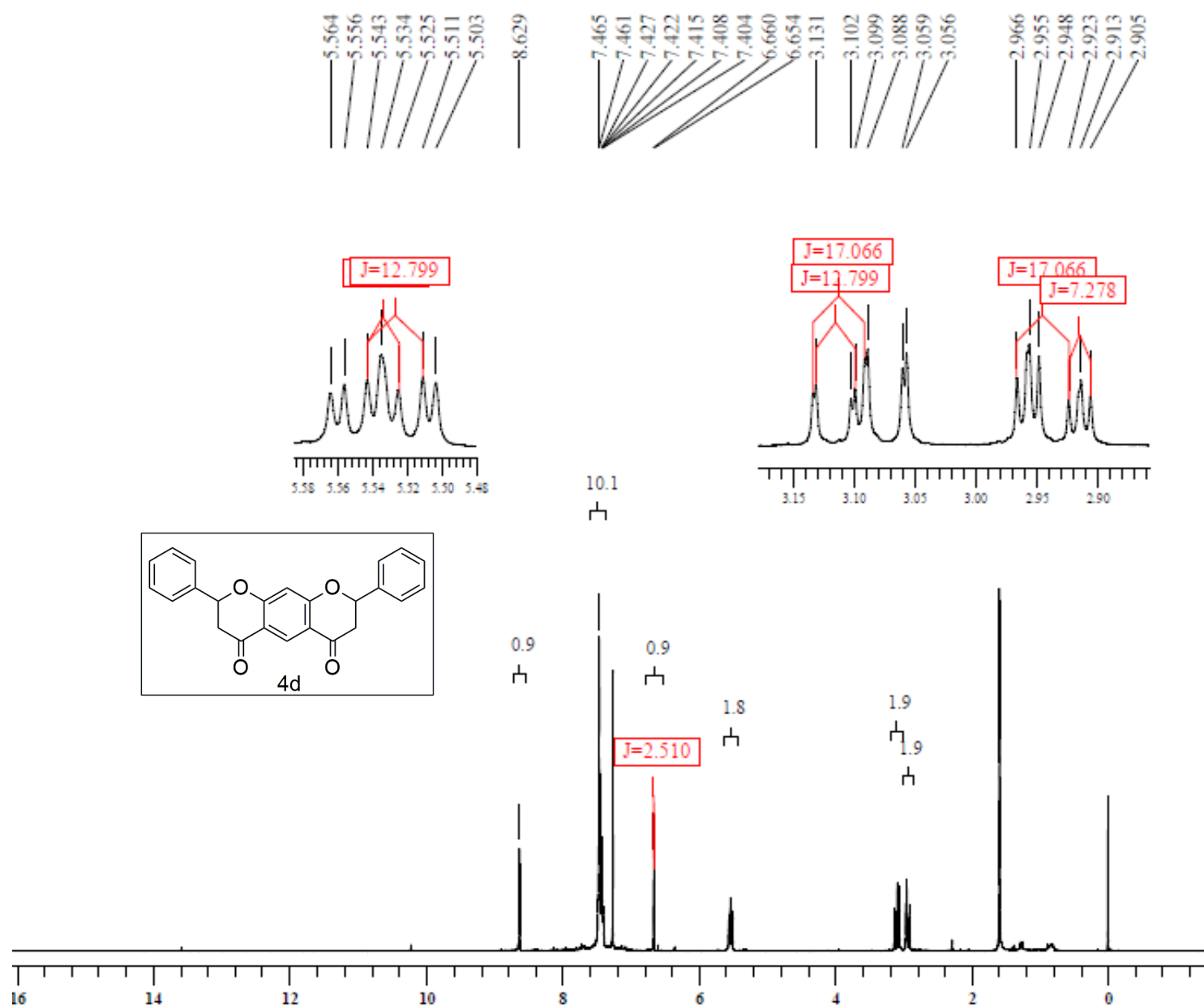


Figure 13: ^1H NMR of 2,8-diphenyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione (4d)

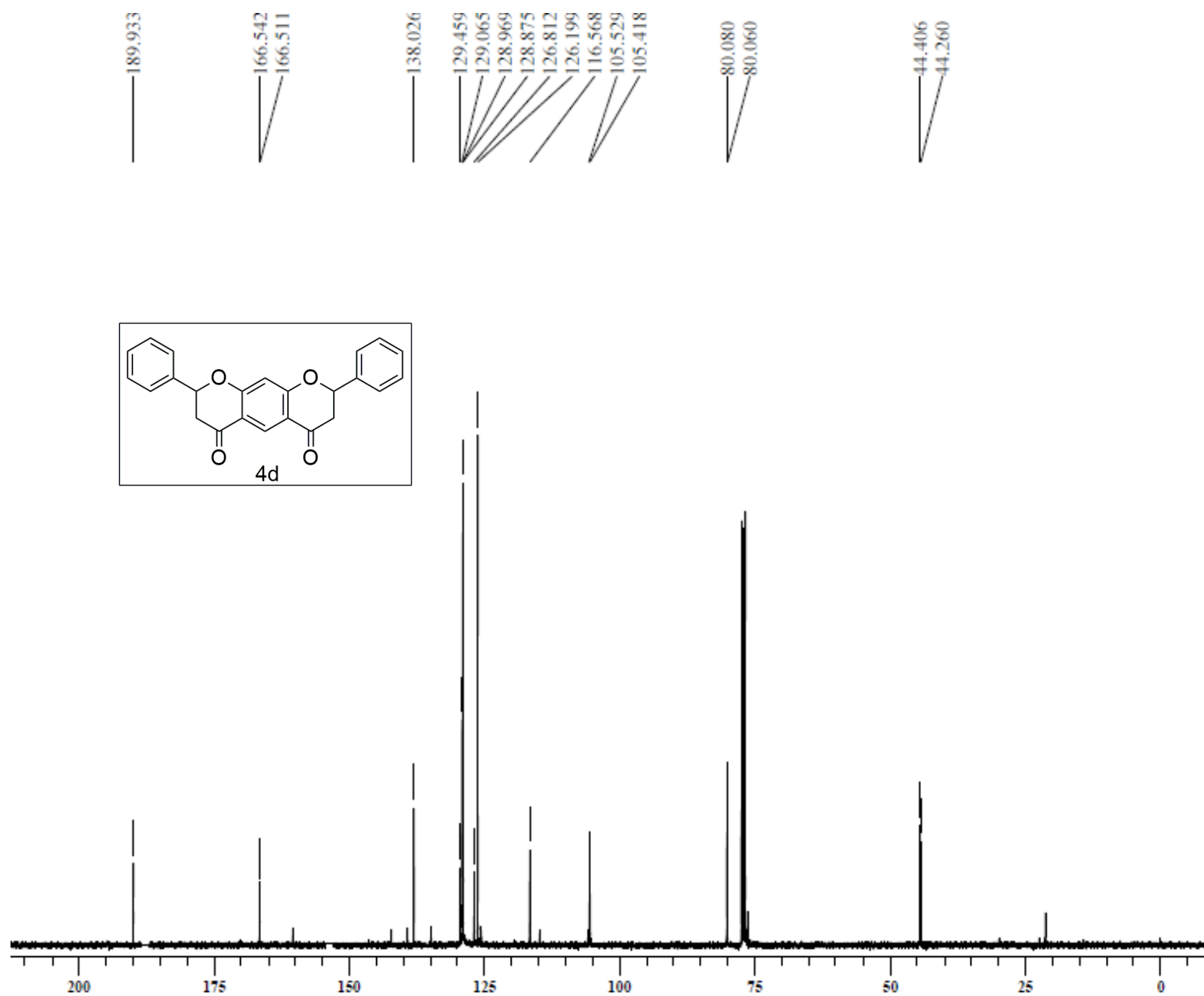
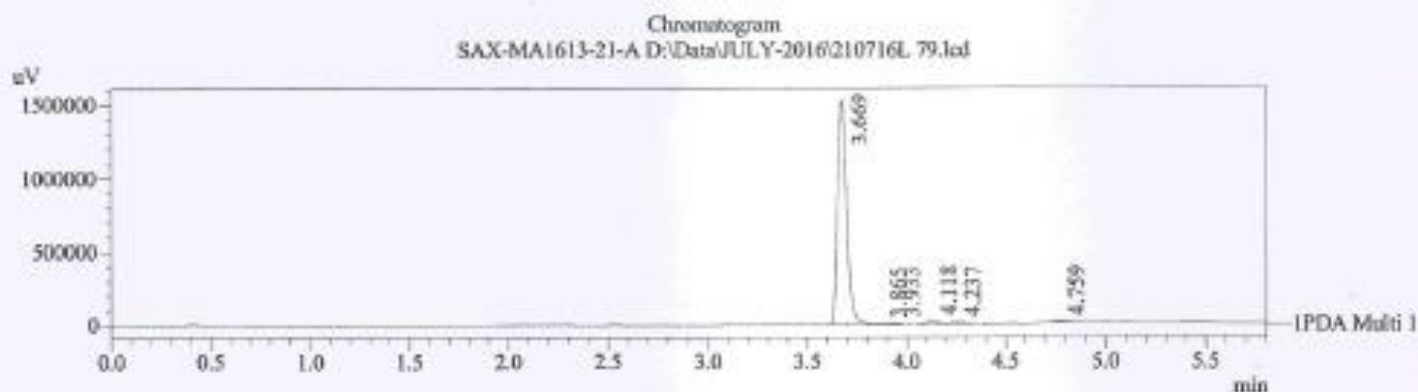
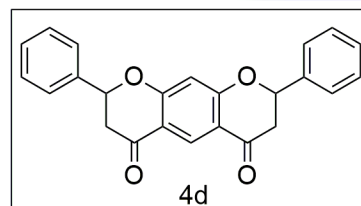


Figure 14: ¹³CNMR of 2,8-diphenyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione (4d)

Method File : AF- KE-5.5.lcm
 Tuning File :D:\Data\Tuning Files\Tuning-ESI-15092014.lct
 Vial No :41
 Description :Kinetex EVO C-18 (50 X3.0mm,2.6um)
 Mobile Phase : A :2.5mM NH₄OOCH in water+5%ACN,
 B:ACN+ 5% 2.5mM NH₄OOCH in Water
 T/B% :0.01/5,4/95,5.5/95
 Flow : 0.8ml / min(Gradient).

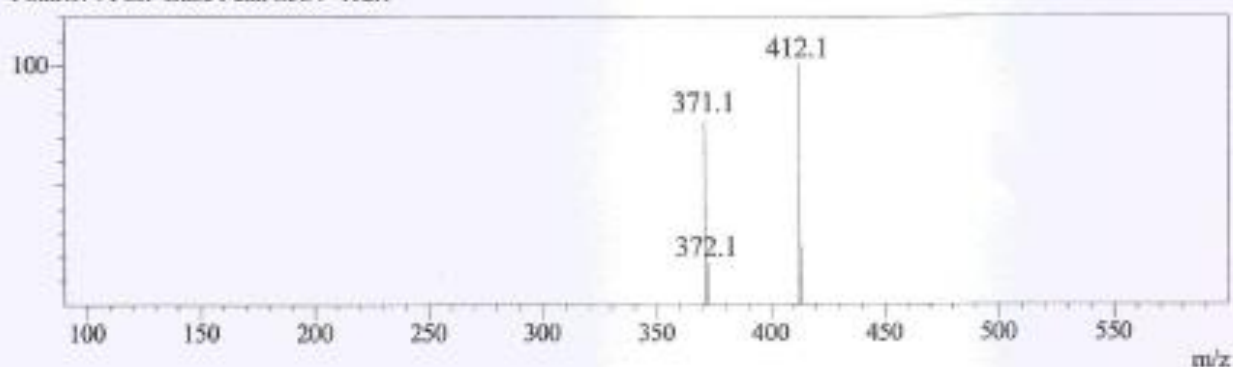


1 PDA Multi 1 / 252nm 4nm

MS Spectrum Graph

Ret. Time:3.667-3.733

Polarity: Pos. Base Peak m/z : 412.1



PeakTable

PDA Ch1 252nm 4nm

Peak#	Ret. Time	Area	Area %
1	3.669	4736931	97.208
2	3.865	5104	0.105
3	3.933	16191	0.332
4	4.118	56566	1.161
5	4.237	26692	0.548
6	4.759	31476	0.646
Total		4872961	100.000

Figure 15: LCMS of 2,8-diphenyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione (4d)

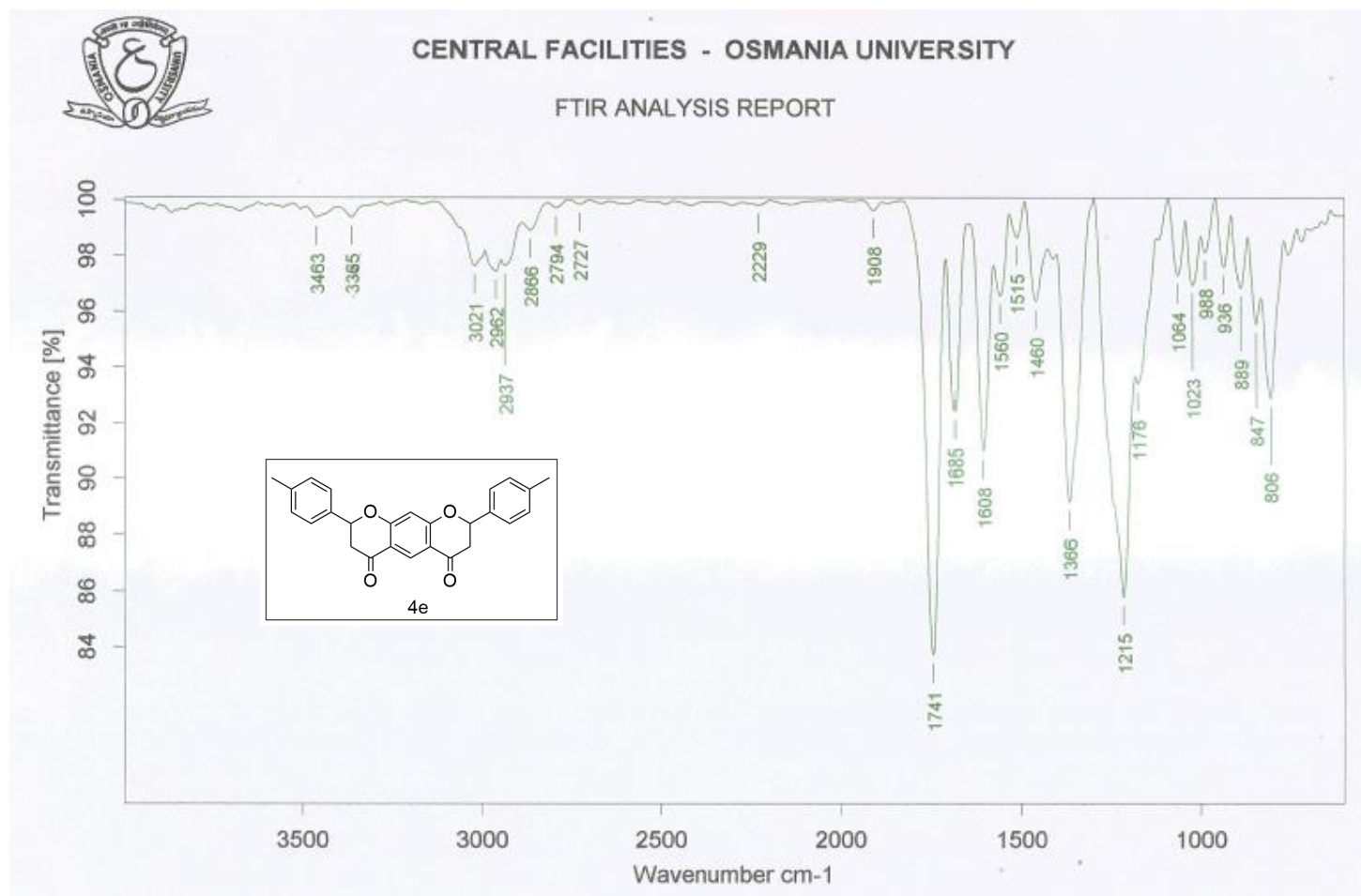
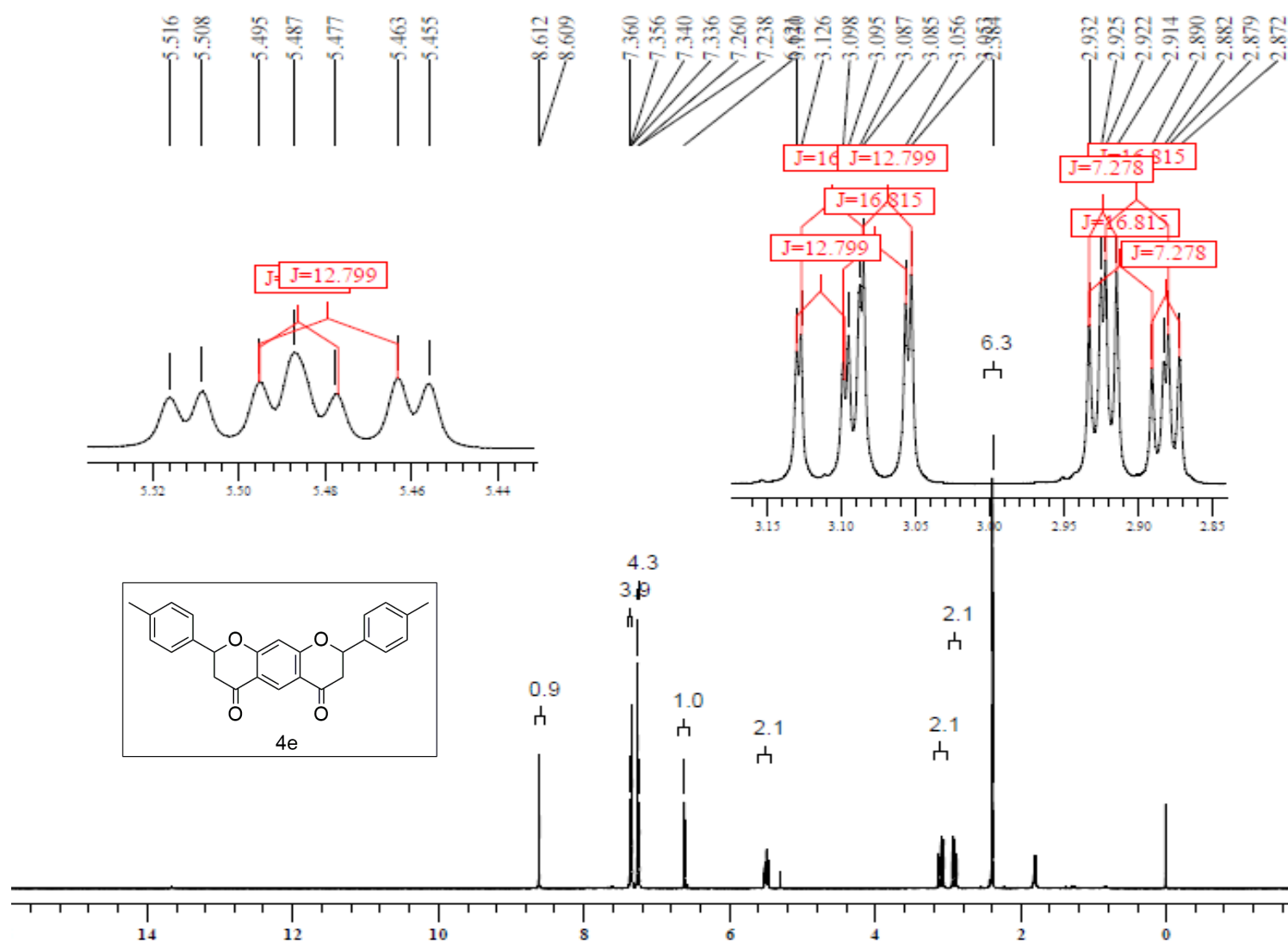


Figure 16: IR data of 2,8-di-p-tolyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione(4e)

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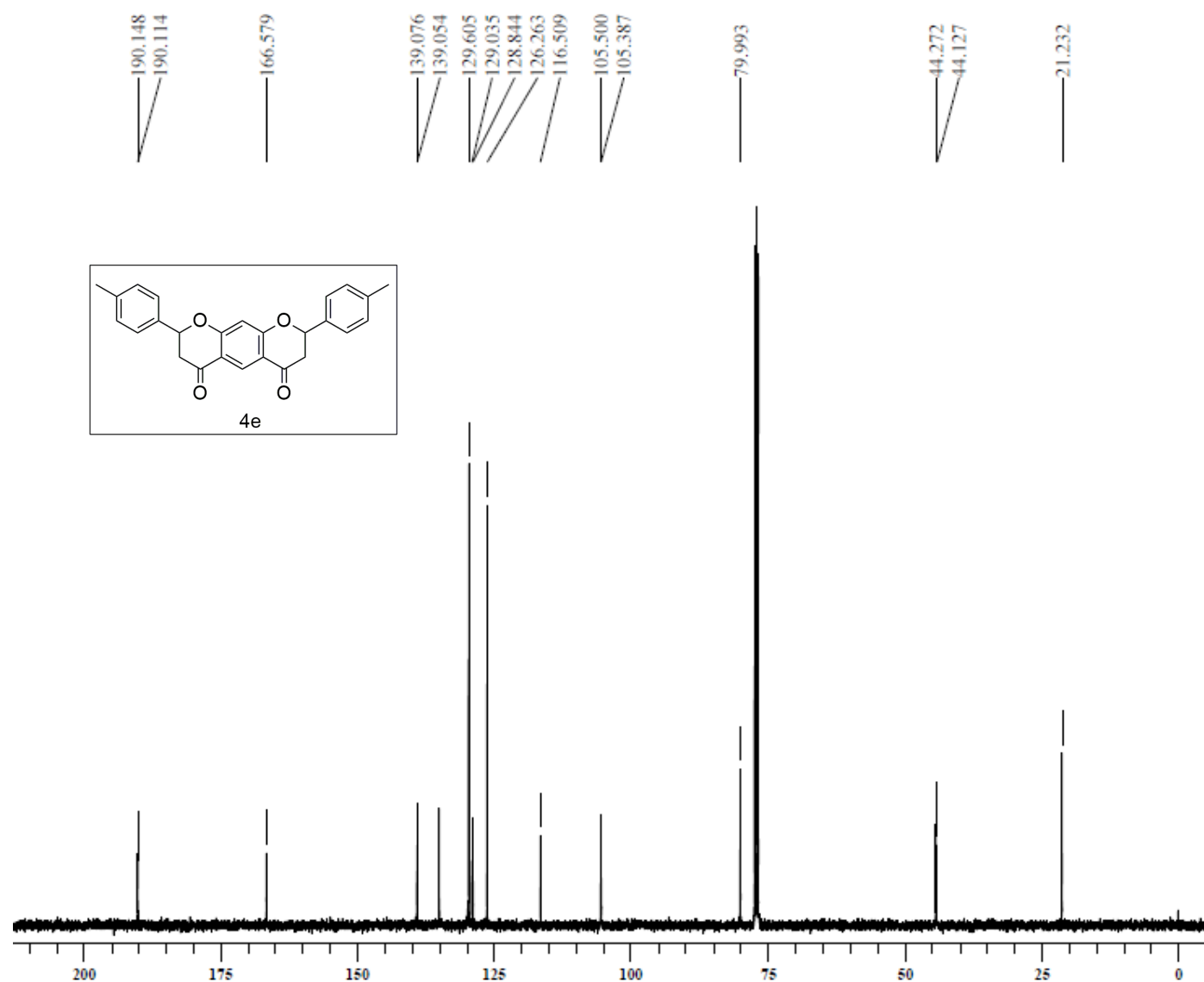
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Figure 17: ^1H NMR of 2,8-di-p-tolyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione(4e)

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Figure 18: ^{13}C NMR of 2,8-di-p-tolyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione (4e)

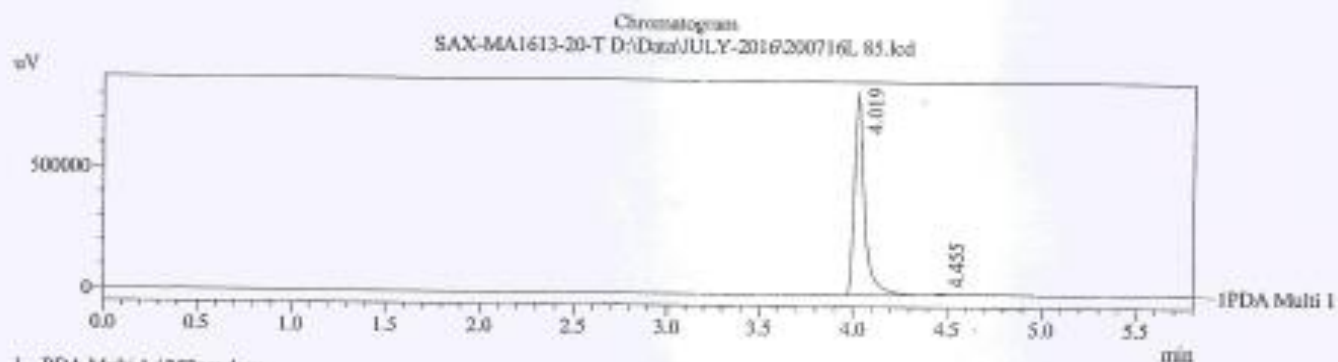
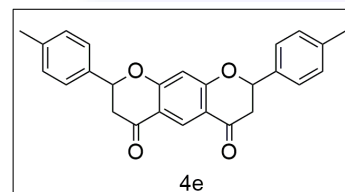
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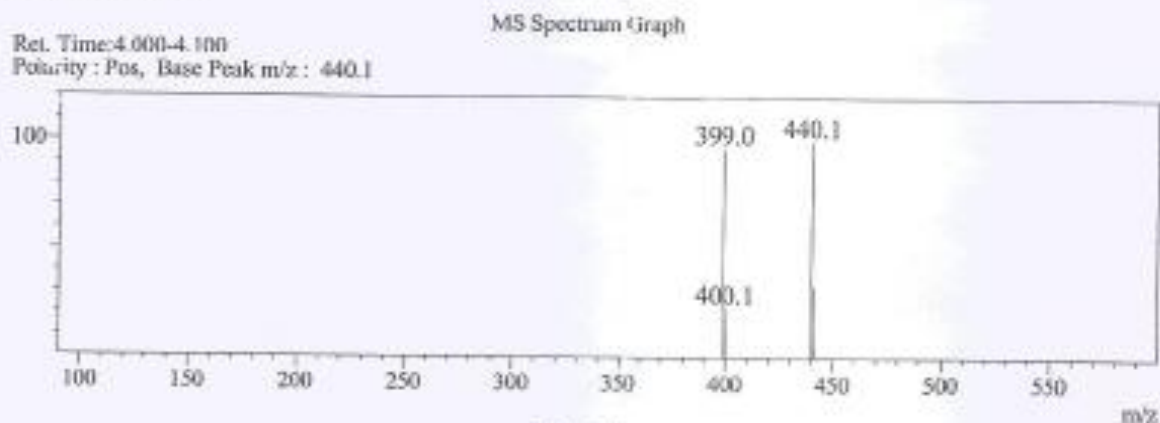
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Method File : AF-KE-5.5.lcm
 Tuning File :D:\Data\Tuning Files\Tuning-ESI-15092014.lct
 Vial No :44
 Description :Kinetex EVO C-18 (50 X3.0mm,2.6um)
 Mobile Phase : A :2.5mM NH₄OOCH in water+5%ACN,
 B:ACN+ 5% 2.5mM NH₄OOCH in Water
 T/B% :0.01/5,4/95,5.5/95
 Flow : 0.8ml / min(Gradient).



Ret. Time:4.000-4.100
 Polarity : Pos, Base Peak m/z : 440.1



PDA Ch1 253nm 4nm

PeakTable

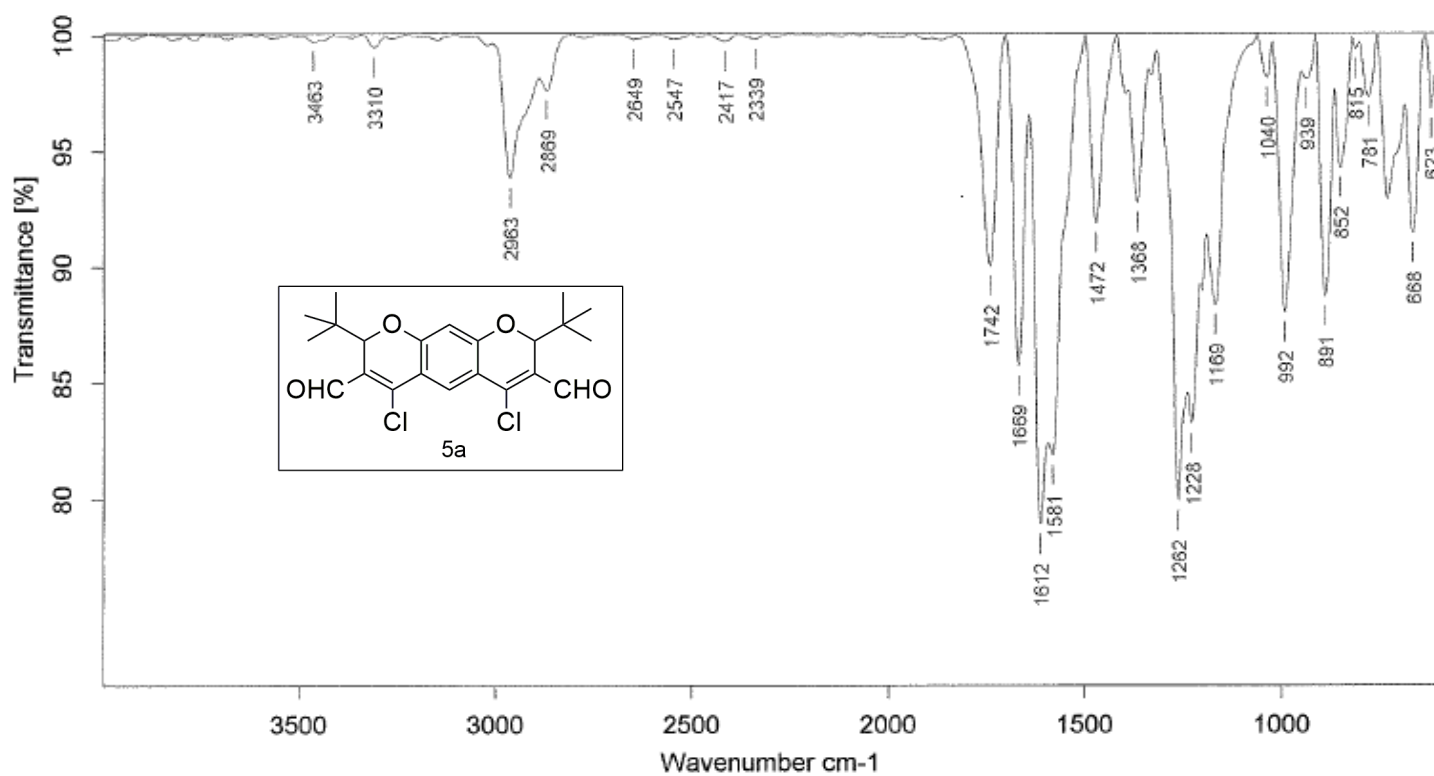
Peak#	Ret. Time	Area	Area %
1	4.019	3115027	99.370
2	4.455	19735	0.630
Total		3134762	100.000

Figure 19: LCMS data of 2,8-di-p-tolyl-2,3,7,8-tetrahydropyrano[3,2-g]chromene-4,6-dione(4e)



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96 Figure 20: IR data of 2,8-di-tert-butyl-4,6-dichloro-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5a)

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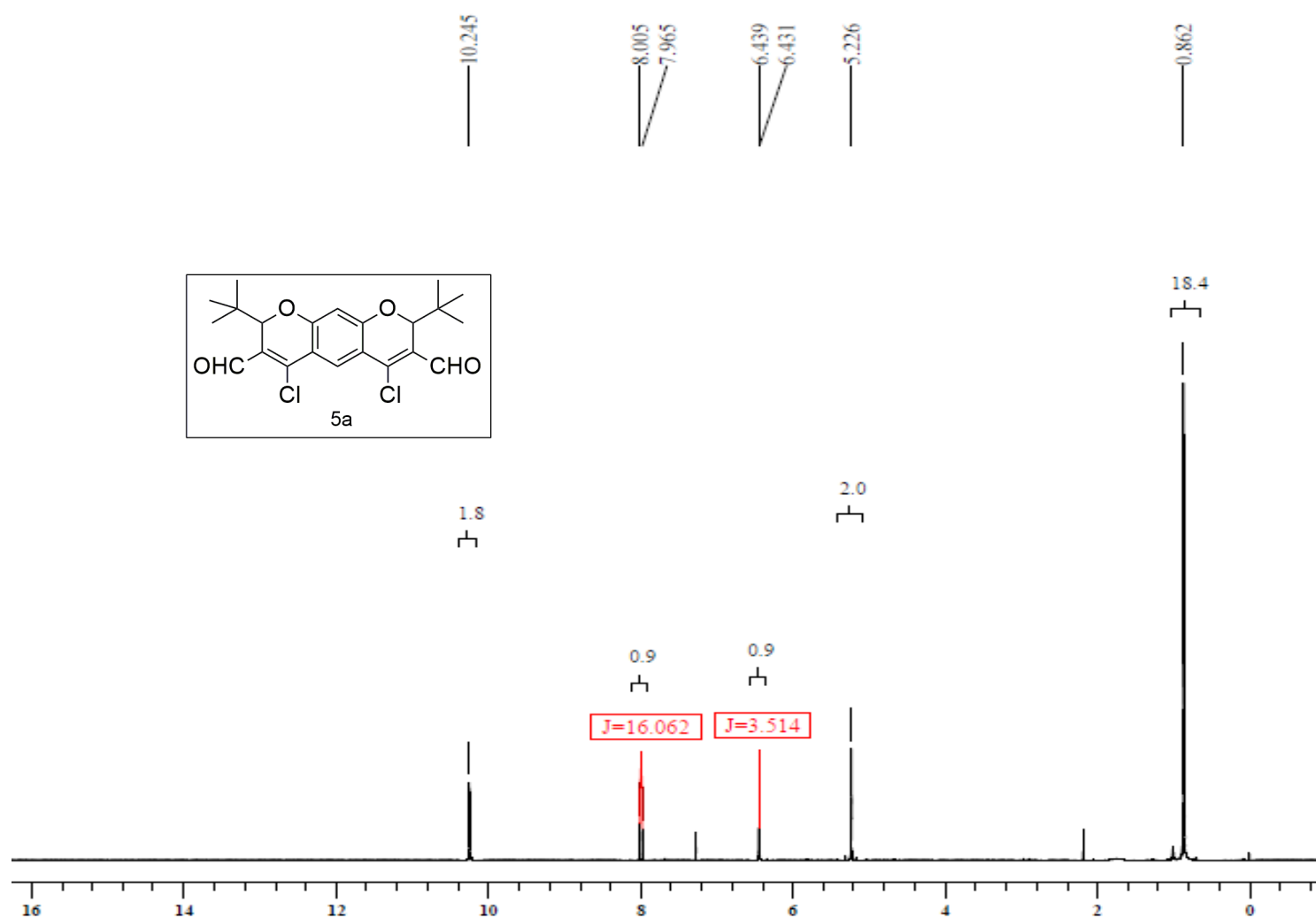


Figure 21: ^1H NMR of 2,8-di-tert-butyl-4,6-dichloro-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5a)

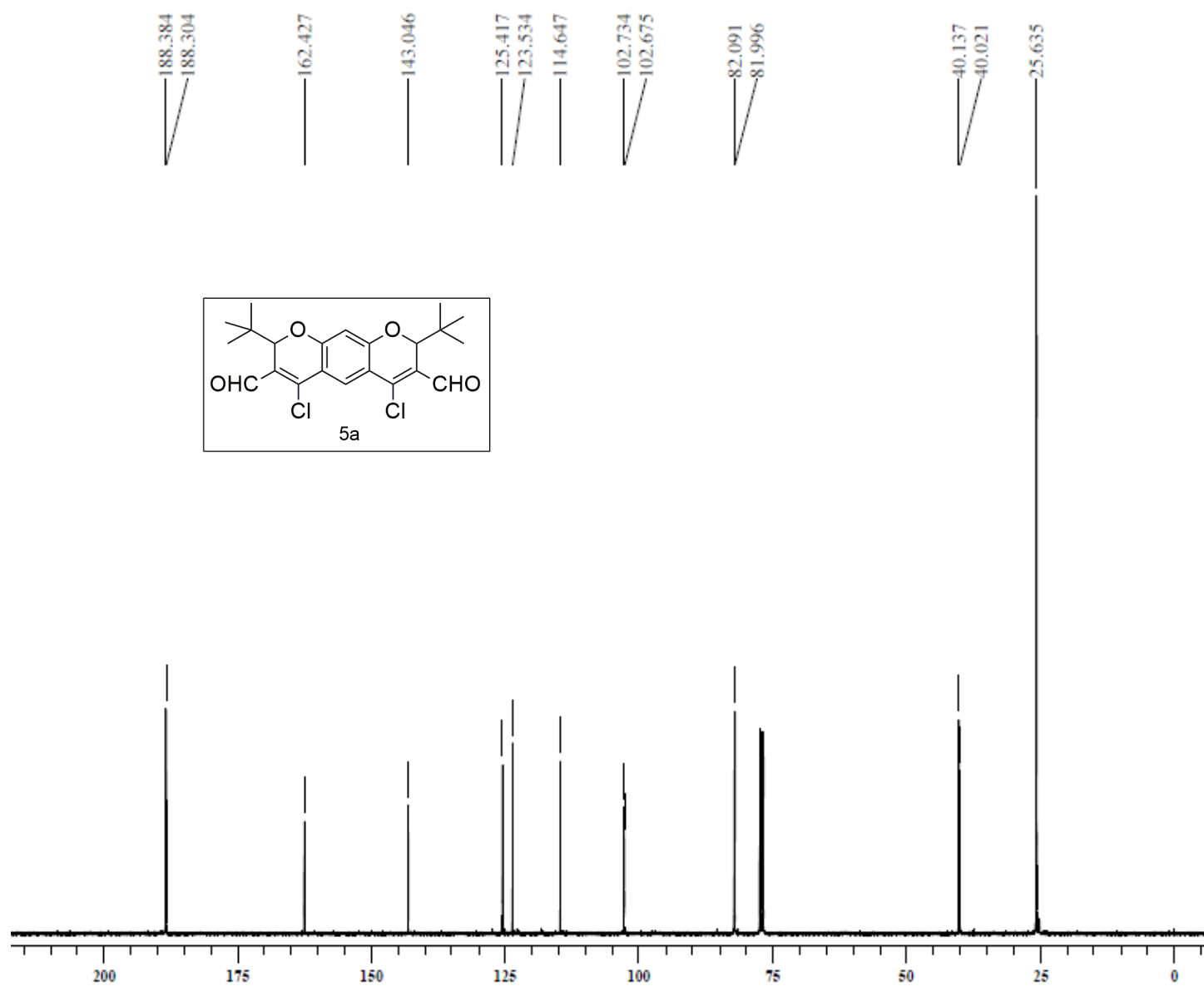
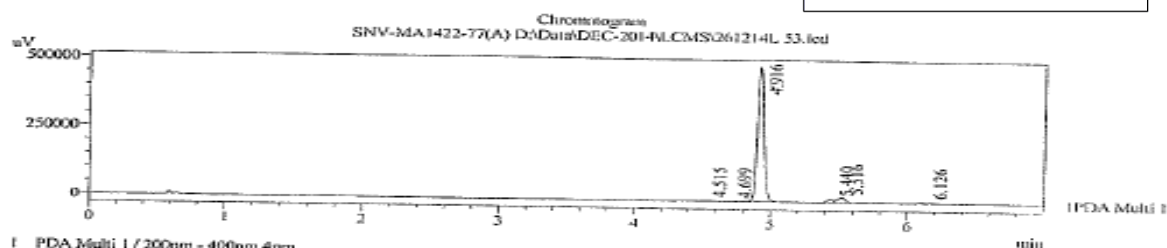
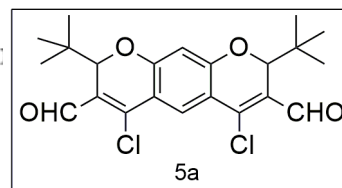
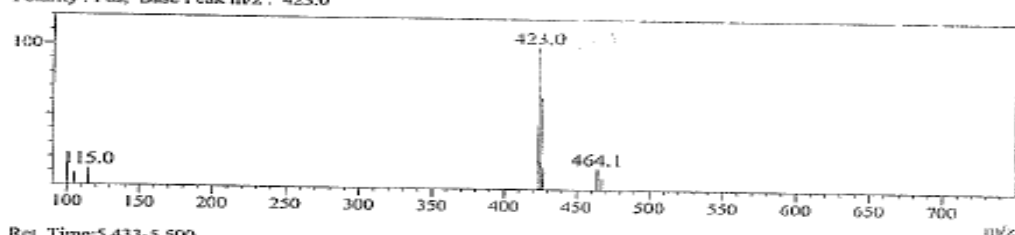


Figure 22: ^{13}C NMR of 2,8-di-tert-butyl-4,6-dichloro-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde(5a)

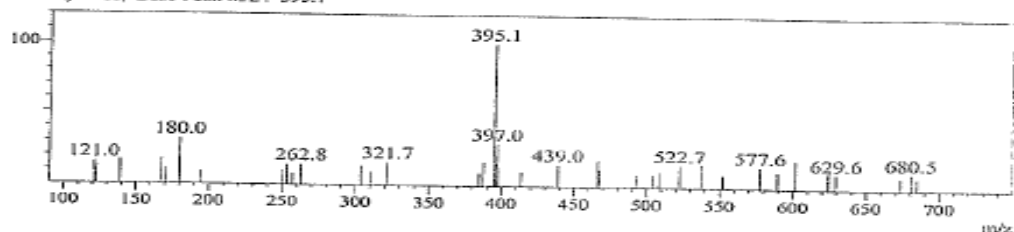
Method File : lcms.lcm
 Tuning File : D:\Data\Tuning Files\Tuning-ESI-15092014.lct
 Vial No : 26
 [Description] : Description : Gemini NX C-18 (50 X4.6mm,3.0um)
 Mobile Phase : A :2.5mM NH₄OOCH in water+5%ACN,
 B:ACN+ 5% 2.5mM NH₄OOCH in Water
 T/B% :0.01/5,0.5/5,3.5/100,7/100
 Flow : 1.2ml / min(Gradient).



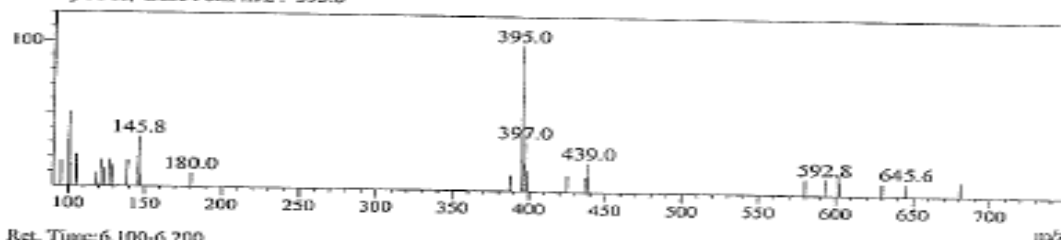
MS Spectrum Graph
 Ret. Time:4.900-5.000
 Polarity : Pos, Base Peak m/z : 423.0



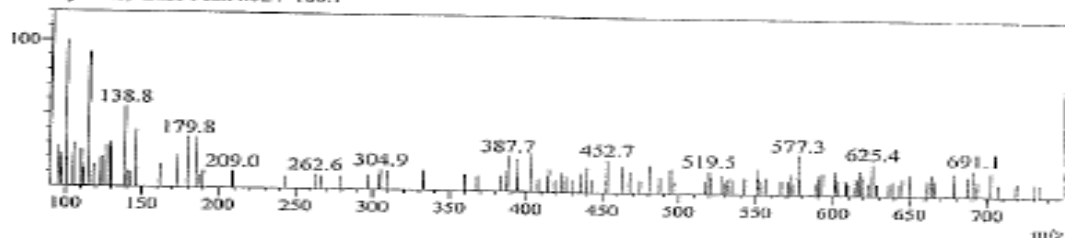
Ret. Time:5.433-5.500
 Polarity : Pos, Base Peak m/z : 395.1



Ret. Time:5.500-5.567
 Polarity : Pos, Base Peak m/z : 395.0



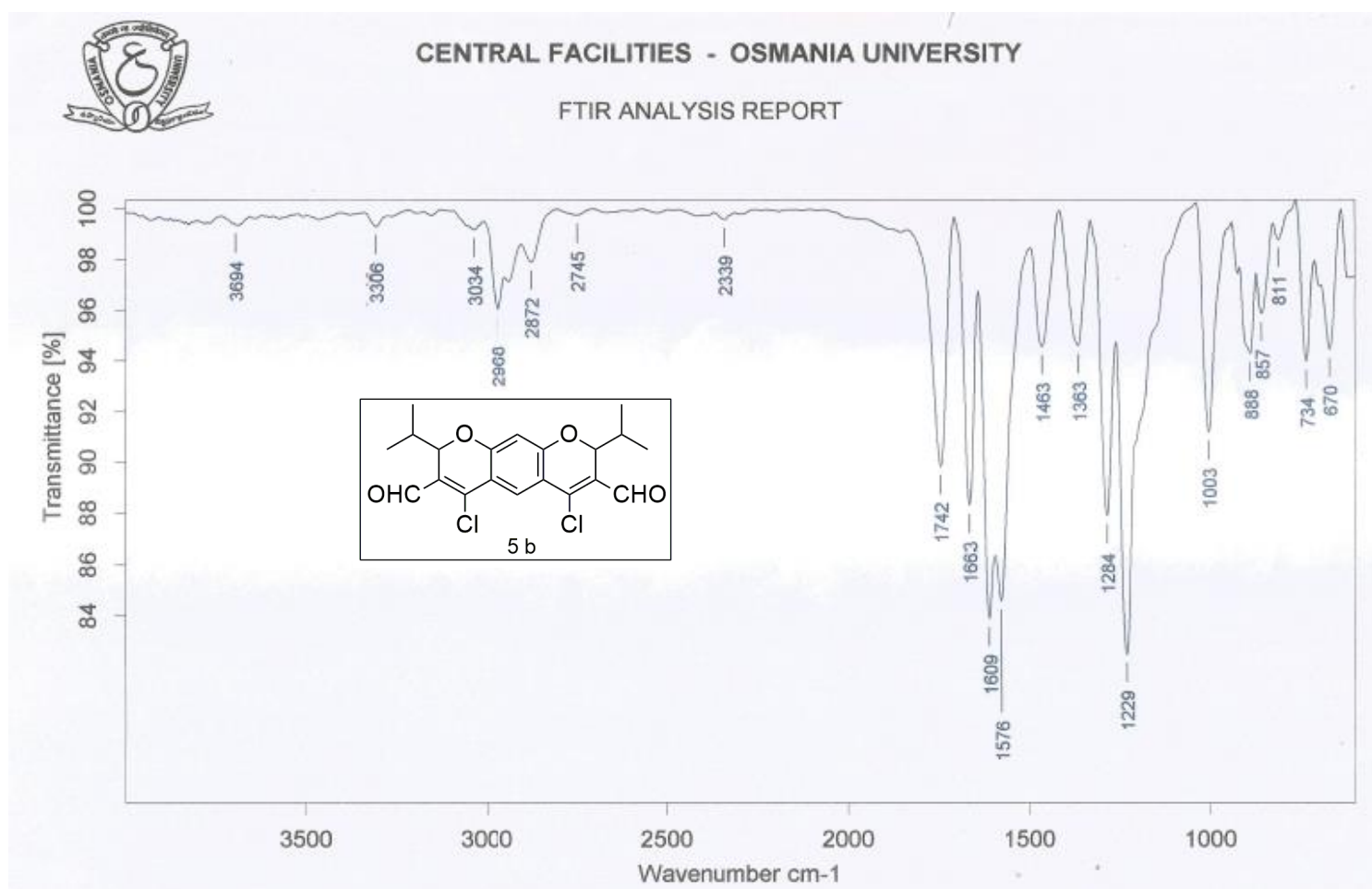
Ret. Time:6.100-6.200
 Polarity : Pos, Base Peak m/z : 100.1



PDA Ch1 200nm - 400nm 4nm

Peak#	Ret. Time	Area	Area %
1	4.515	6354	0.362
2	4.699	545	0.031
3	4.916	1592568	90.703
4	5.440	43341	2.408
5	5.516	77170	4.395
6	6.126	35828	2.041
Total		1755806	100.000

Figure 23: LCMS of 2,8-di-tert-butyl-4,6-dichloro-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde(5a)



40
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42 Figure 24: IR data of 4,6-dichloro-2,8-diisopropyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5b)
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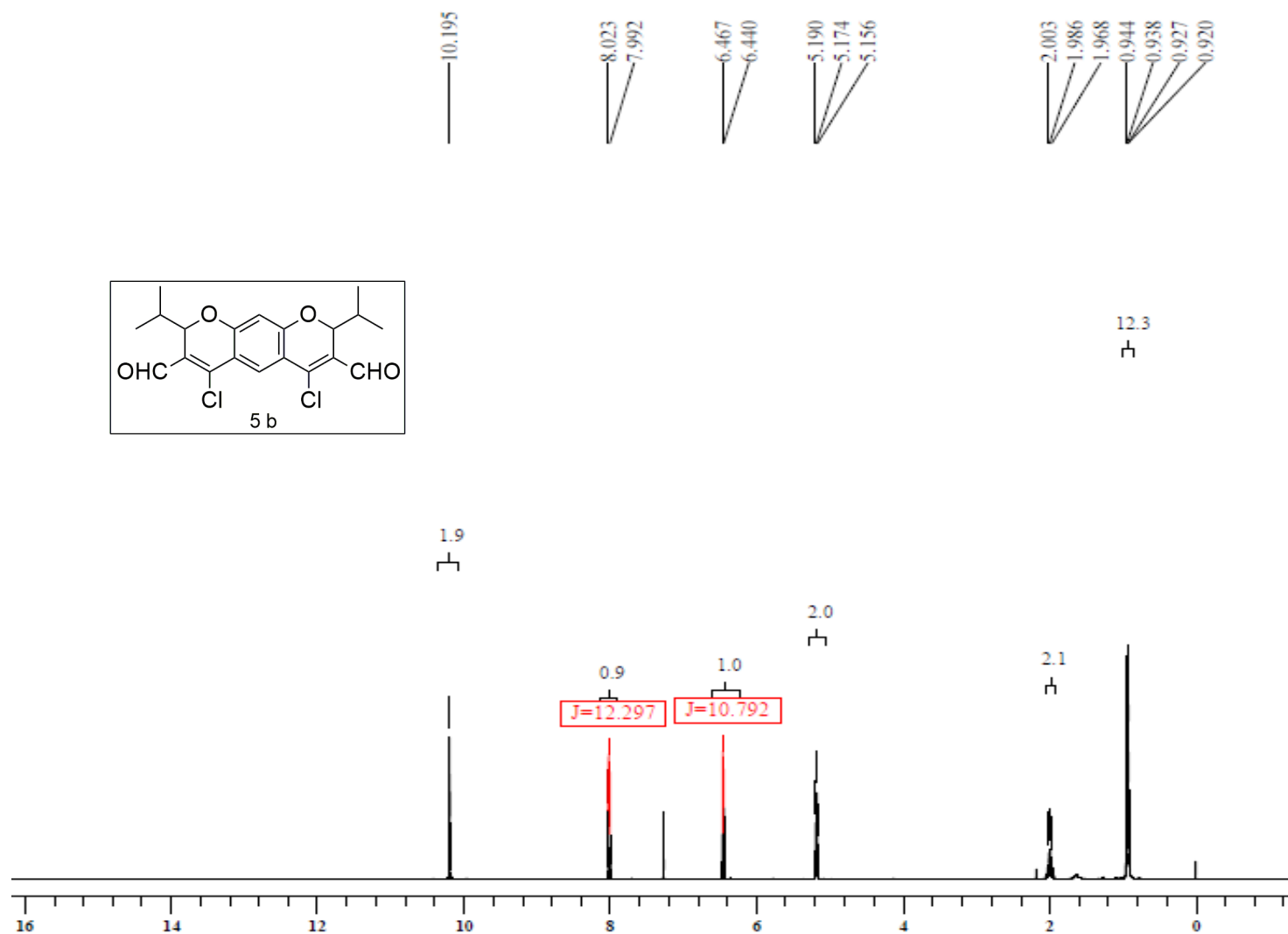


Figure 25: ^1H NMR of 4,6-dichloro-2,8-diisopropyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5b)

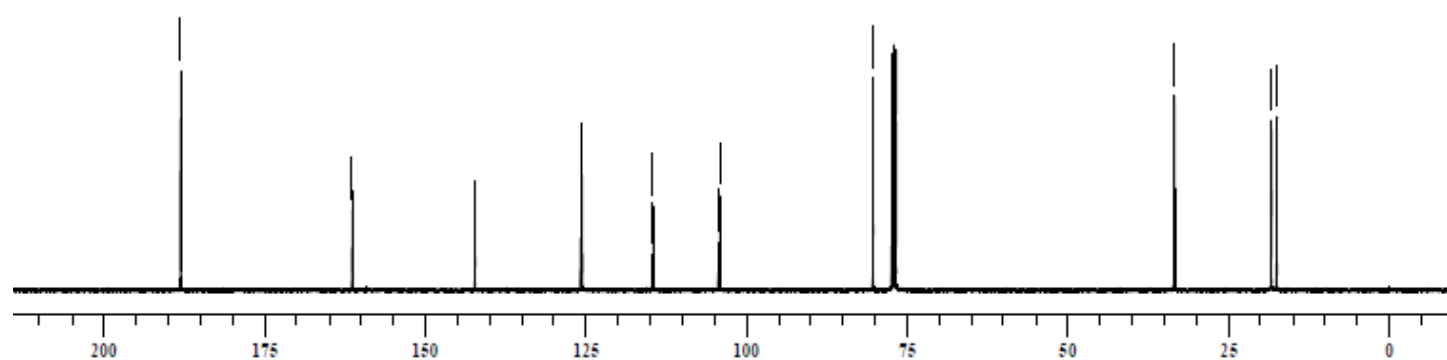
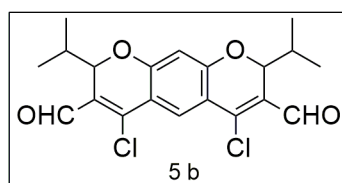
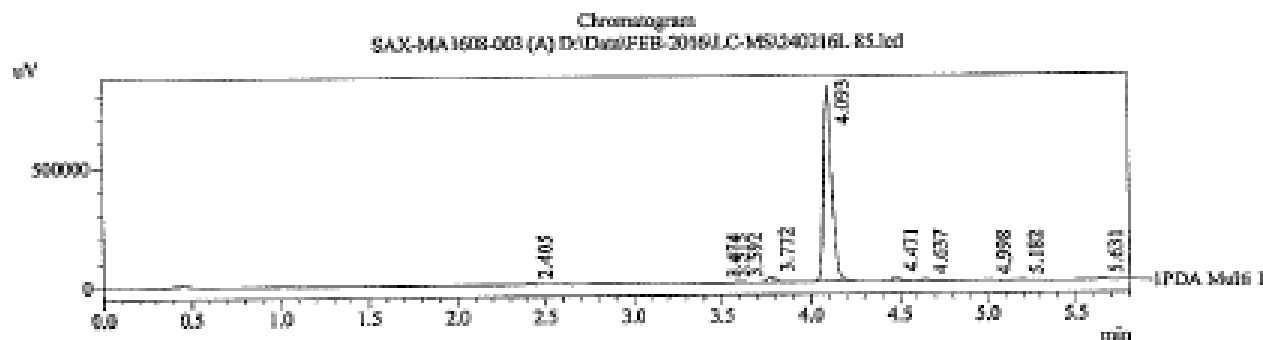
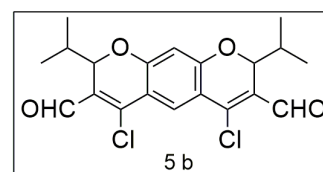


Figure 26: ¹³C NMR data of 4,6-dichloro-2,8-diisopropyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5b)

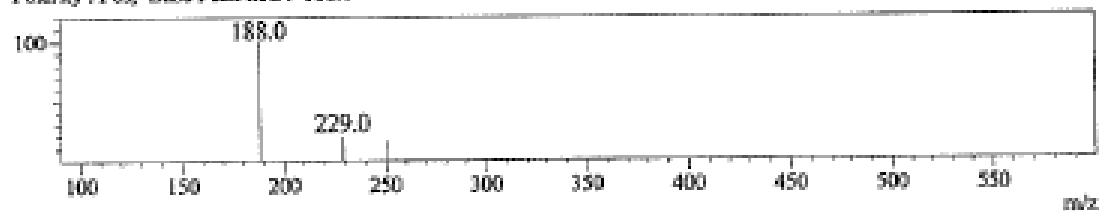
Tuning File :D:\Data\Tuning Files\Tuning-ESI-15092014.lct
Vial No :27
Description :Kinetex EVO C-18 (50 X3.0mm,2.6um)
Mobile Phase : A :2.5mM NH₄OOCH in water+5%ACN,
B:ACN+ 5% 2.5mM NH₄OOCH in Water
T/B% :0.01/5,4/95,5,5/95
Flow : 0.8ml / min(Gradient).



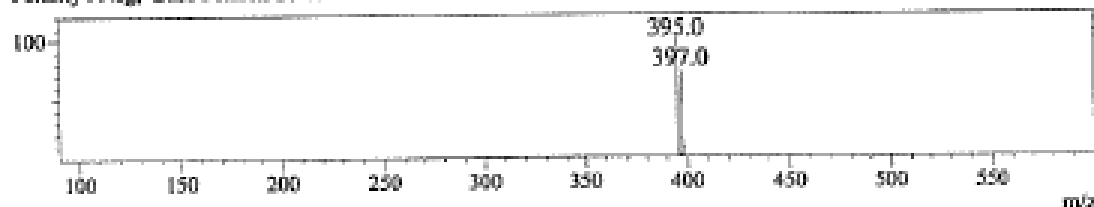
1 PDA Multi 1 / 246nm 4nm

MS Spectrum Graph

Ret. Time:3.767-3.833
Polarity : Pos, Base Peak m/z : 188.0



Ret. Time:4.083-4.150
Polarity : Neg, Base Peak m/z : 395.0



PeakTable

PDA Ch1 246nm 4nm

Peak#	Ret. Time	Area	Area %
1	2.405	14214	0.521
2	3.474	5378	0.197
3	3.513	4192	0.154
4	3.592	11212	0.411
5	3.772	70021	2.568
6	4.093	2528277	92.718
7	4.471	46638	1.710
8	4.637	16009	0.587
9	4.998	3736	0.137
10	5.182	8149	0.299
11	5.631	19014	0.697
Total		2726839	100.000

Figure 27: LCMS of 4,6-dichloro-2,8-diisopropyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5b)

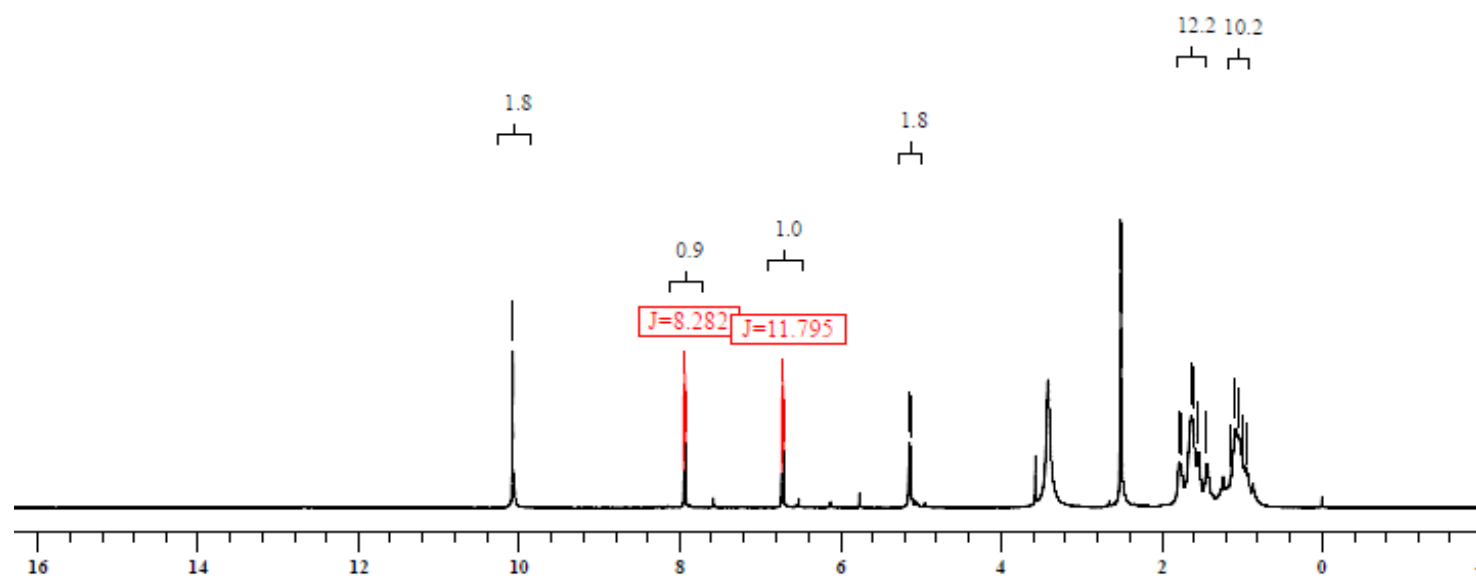
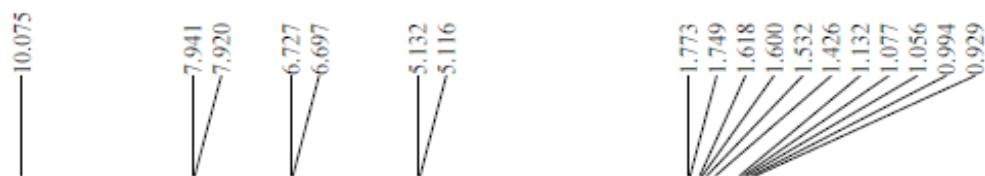
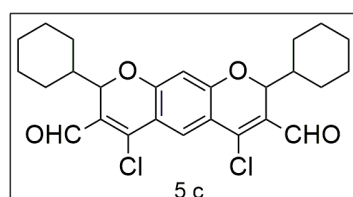


Figure 28: ^1H NMR of 4,6-dichloro-2,8-dicyclohexyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5c)

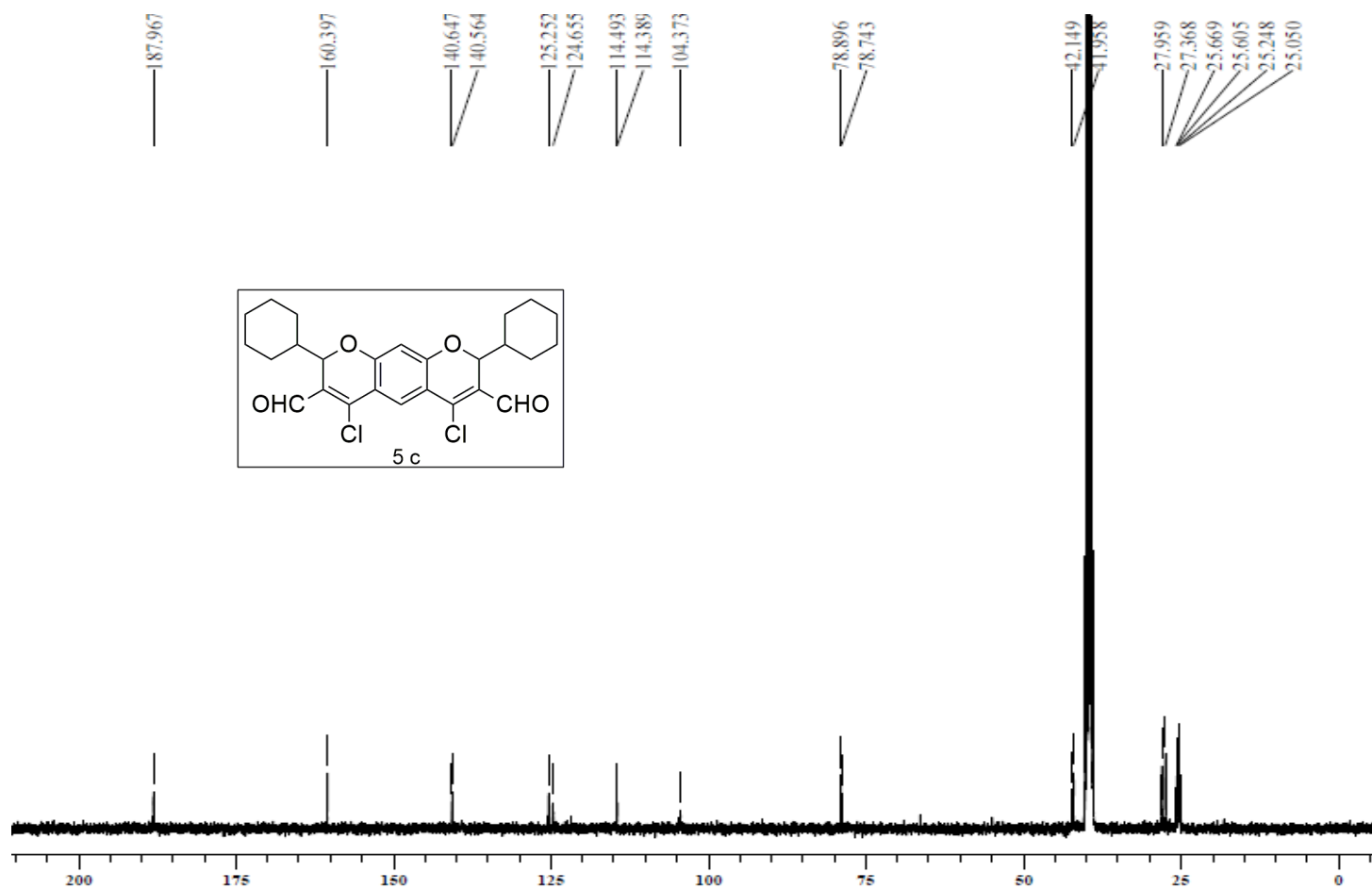
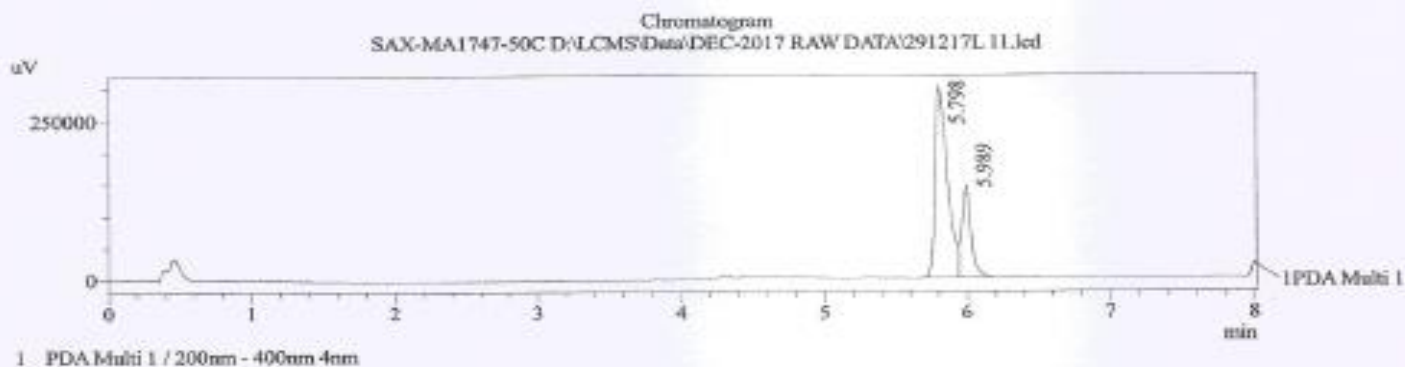
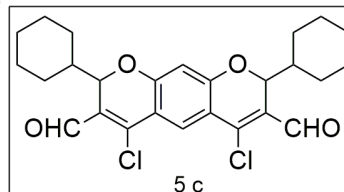


Figure 29: ¹³C NMR of 4,6-dichloro-2,8-dicyclohexyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5c)

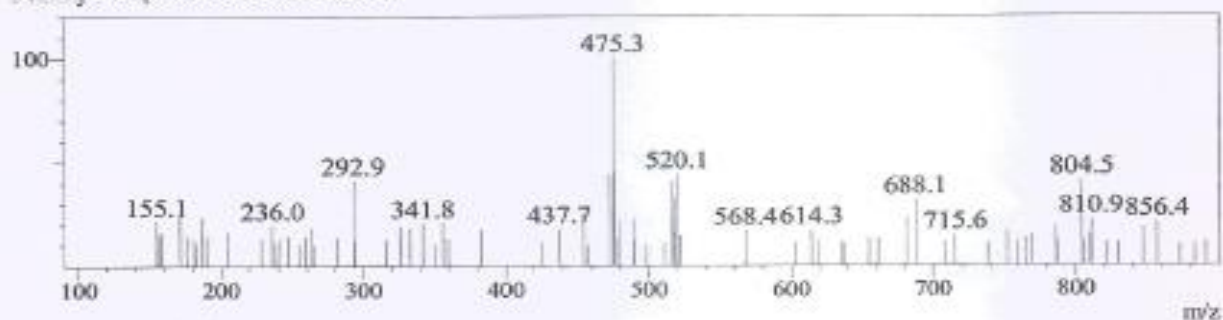
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 Tuning File :D:\LCMS\Tuning Files\Tuning-ESI-03022017 1et
 Vial No :4
 Description :Kinetex EVO C-18 (50 X3.0mm,2.6um)
 Mobile Phase : A :2.5mM NH₄OOCH in water+5%ACN,
 B:ACN+ 5% 2.5mM NH₄OOCH in Water
 T/B% :0.01/5,4/95,5.5/95
 Flow : 0.8ml / min(Gradient).



MS Spectrum Graph

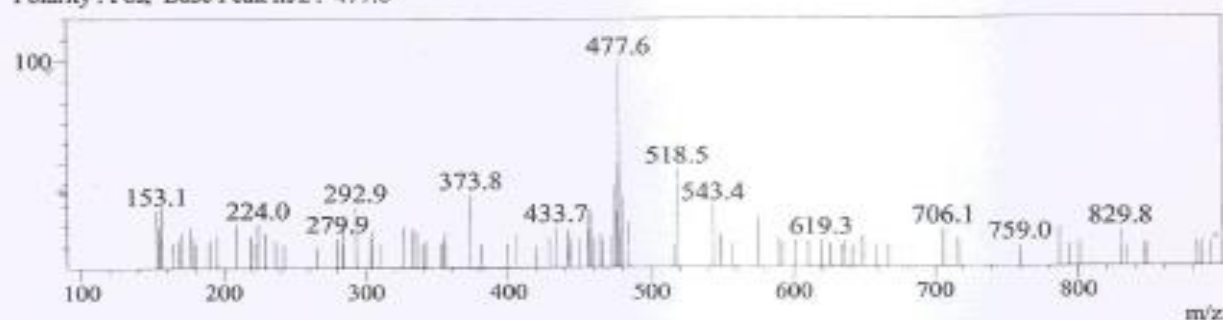
Ret. Time:5.767-5.867

Polarity : Neg, Base Peak m/z : 475.3



Ret. Time:5.933-6.033

Polarity : Pos, Base Peak m/z : 477.6



PeakTable

PDA Ch1 200nm - 400nm 4nm

Peak#	Ret. Time	Area	Area %
1	5.798	1890804	75.241
2	5.989	622186	24.759
Total		2512991	100.000

Figure 30: LCMS of 4,6-dichloro-2,8-dicyclohexyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5c)



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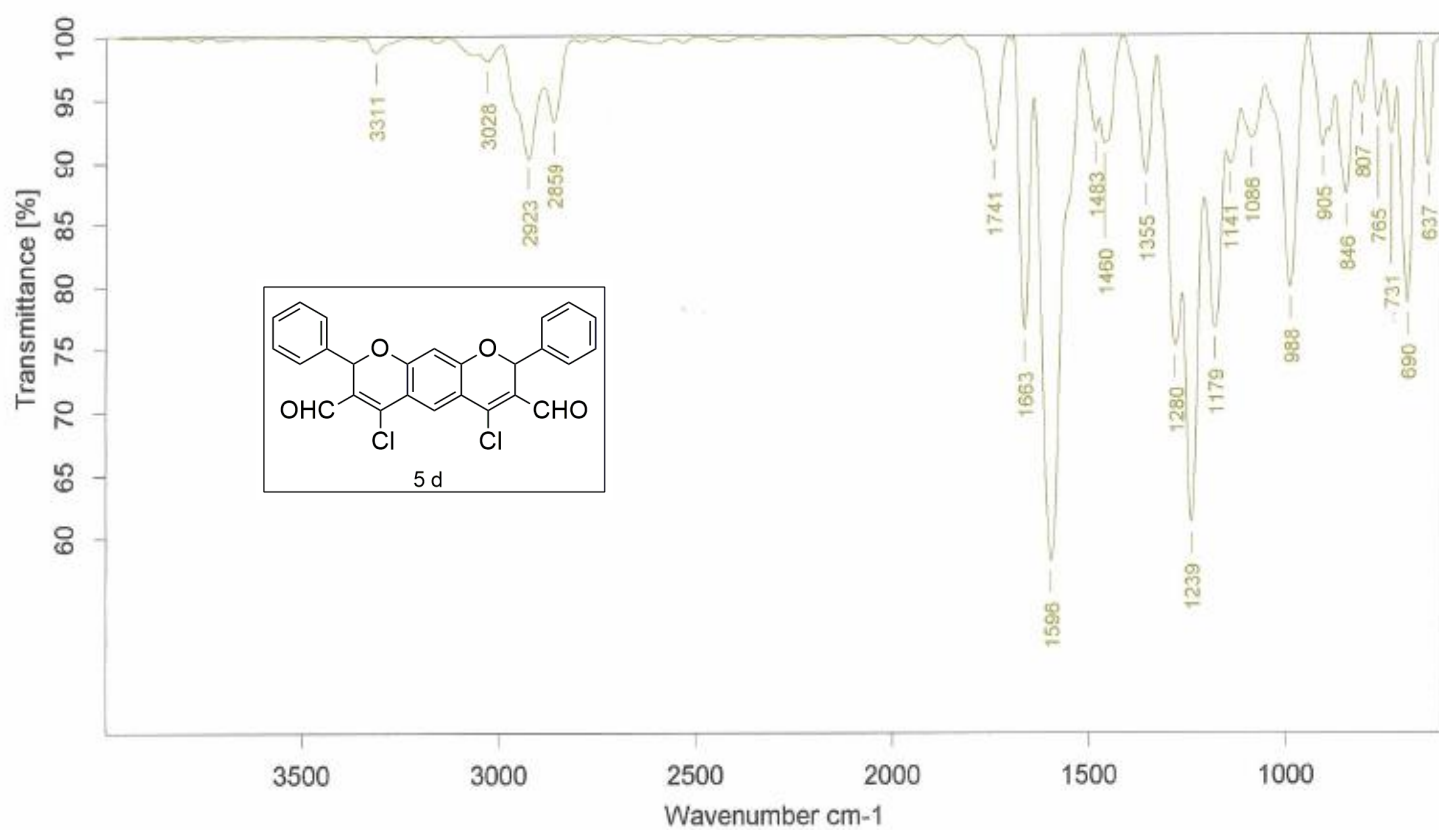


Figure 31: IR data of 4,6-dichloro-2,8-diphenyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5d)

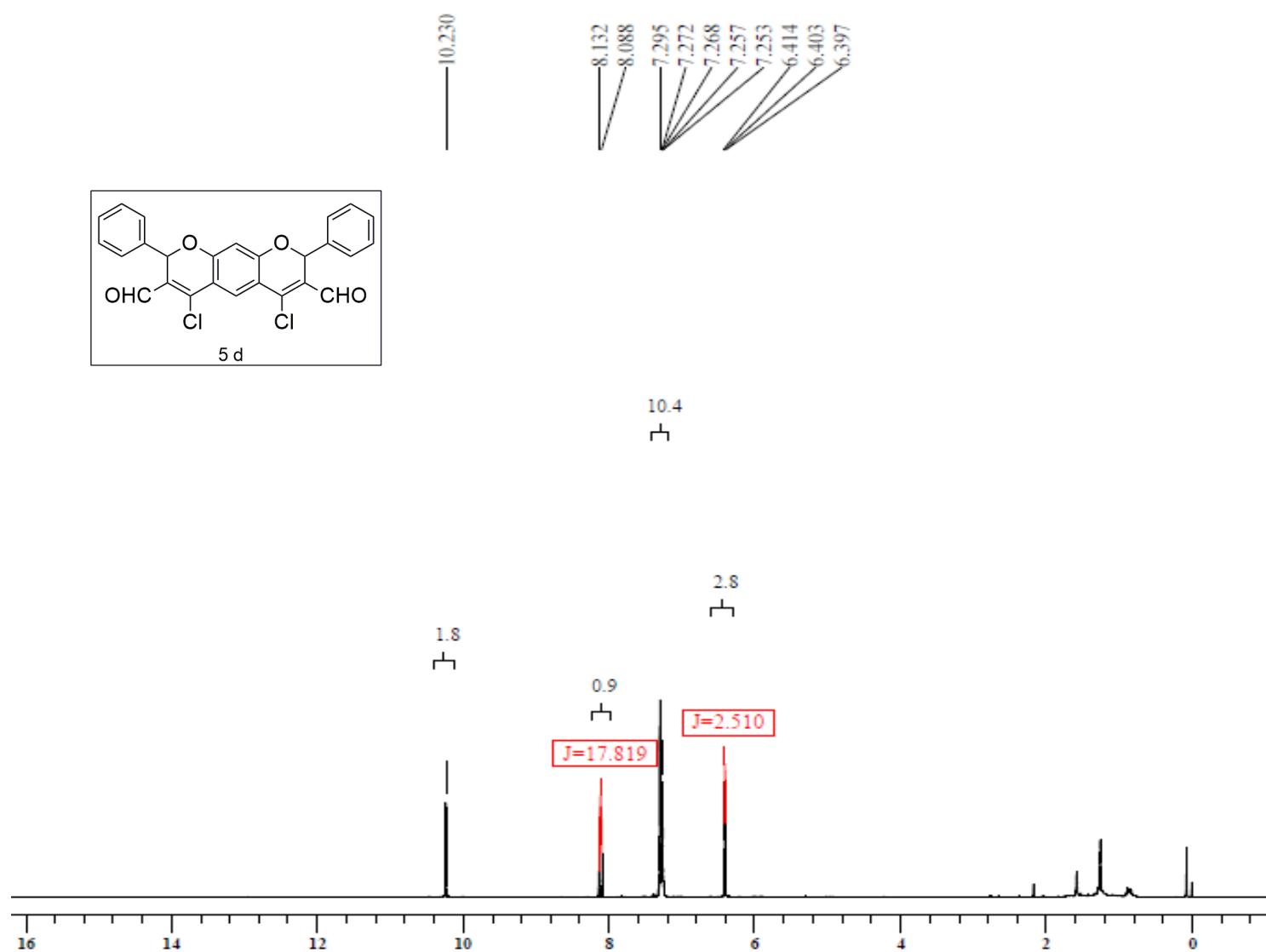
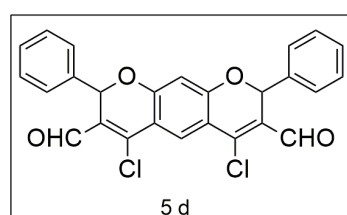
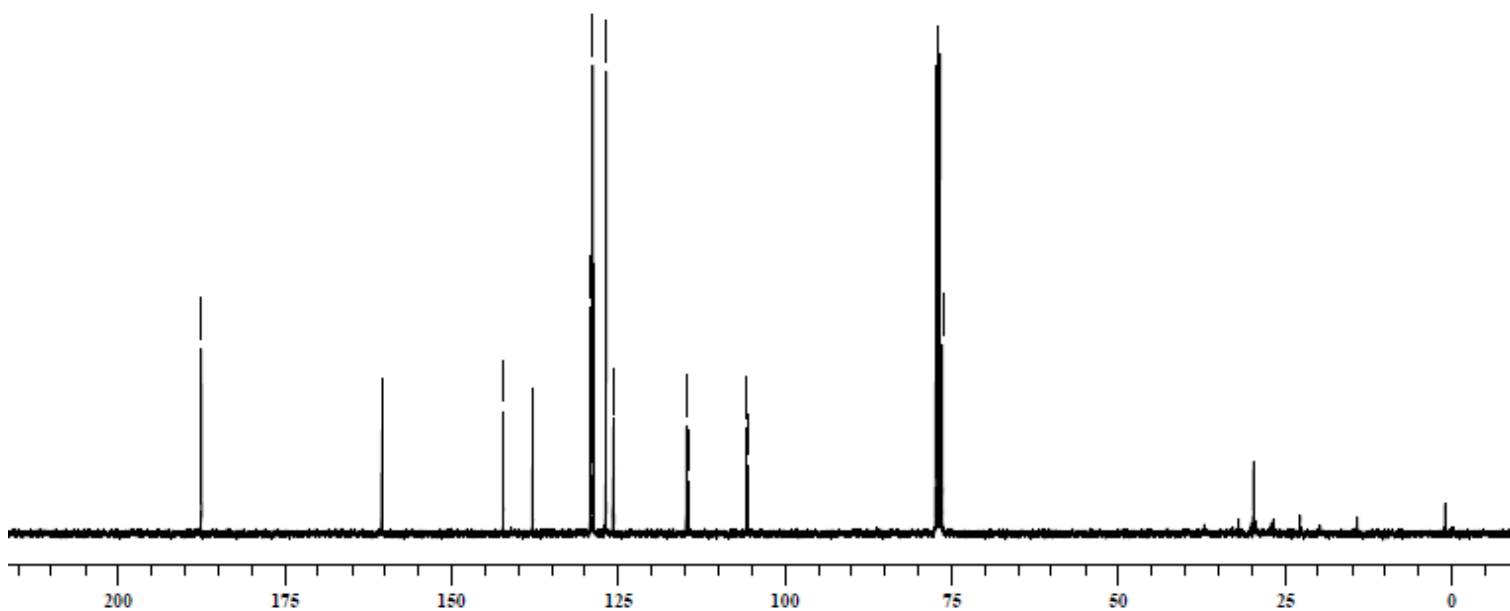
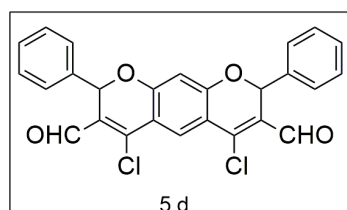
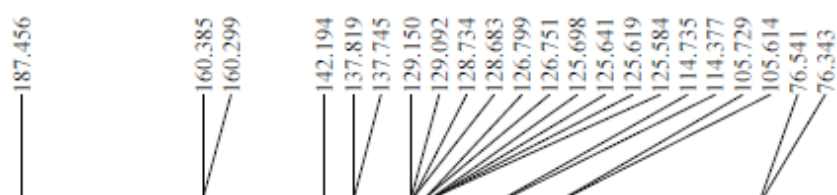


Figure 32: ^1H NMR of 4,6-dichloro-2,8-diphenyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde(5d)

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26 Figure 33: ¹³C NMR of 4,6-dichloro-2,8-diphenyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5d)

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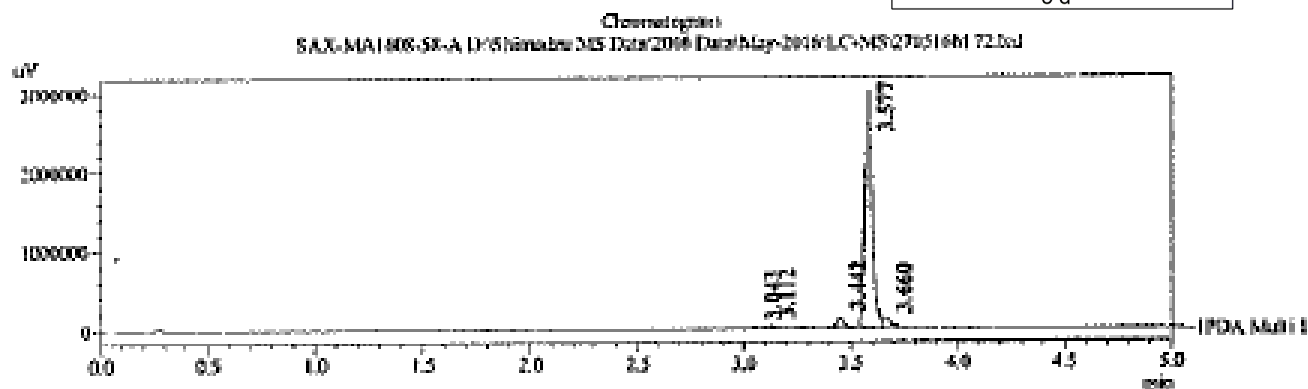
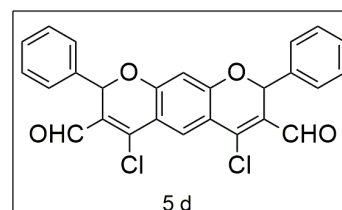
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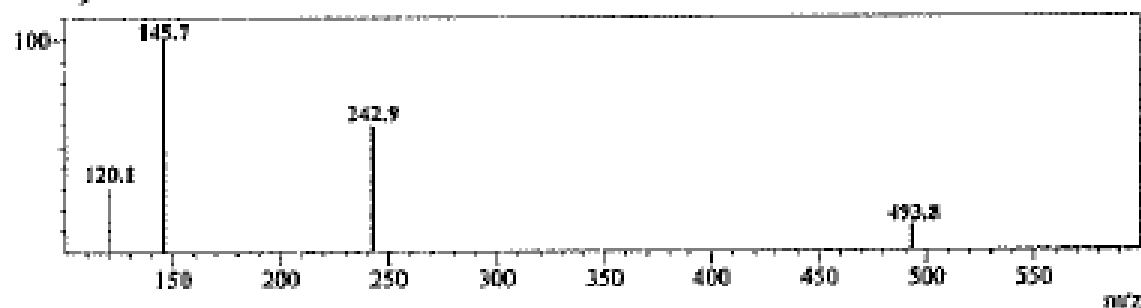
Method File : TFA AE-5.0mL/min
 Tuning File : DASHmonize MS Data(Tuning File:ESI 040314-1.Jet
 Vial : 123
 Description: Column: Asentis Express C-18(50X3.0mm,2.7um)
 Mobile Phase:A-0.025% Aq TFA+5% ACN,
 Mobile Phase:B- ACN+ 5% 0.025% Aq TFA
 T/B%:0.01/5,0.5/5,3/100,5/100
 Flow Rate:1.2mL/min(Gradient)



I : PDA Multi I / 200nm - 409nm 4nm

MS Spectrum

Ret Time:3.417-3.483
 Polarity:Pos: Base Peak:145.70



Ret Time:3.550-3.600
 Polarity:Pos: Base Peak:464.90

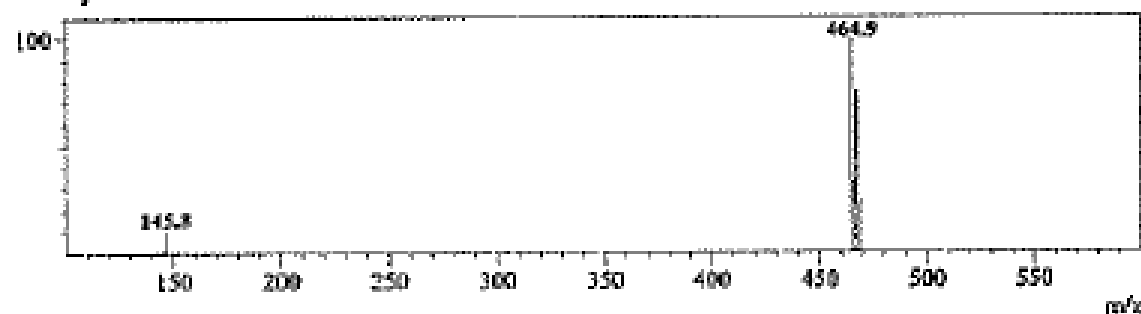
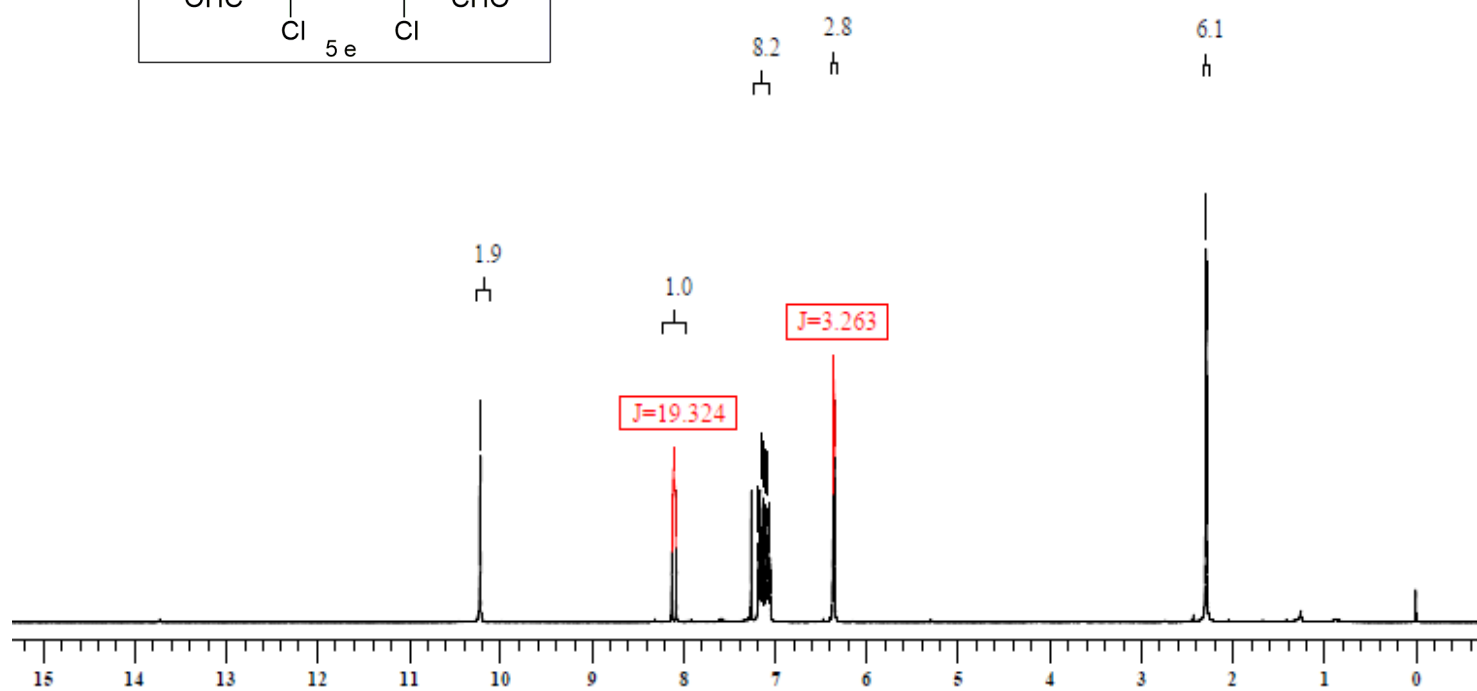
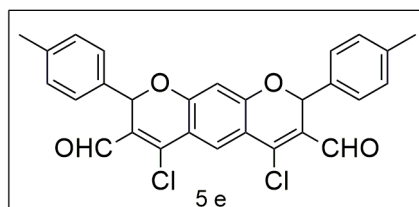


Figure 34: LCMS of 4,6-dichloro-2,8-diphenyl-2,8-dihydropyrano [3,2-g] chromene-3,7-dicarbaldehyde (5d)

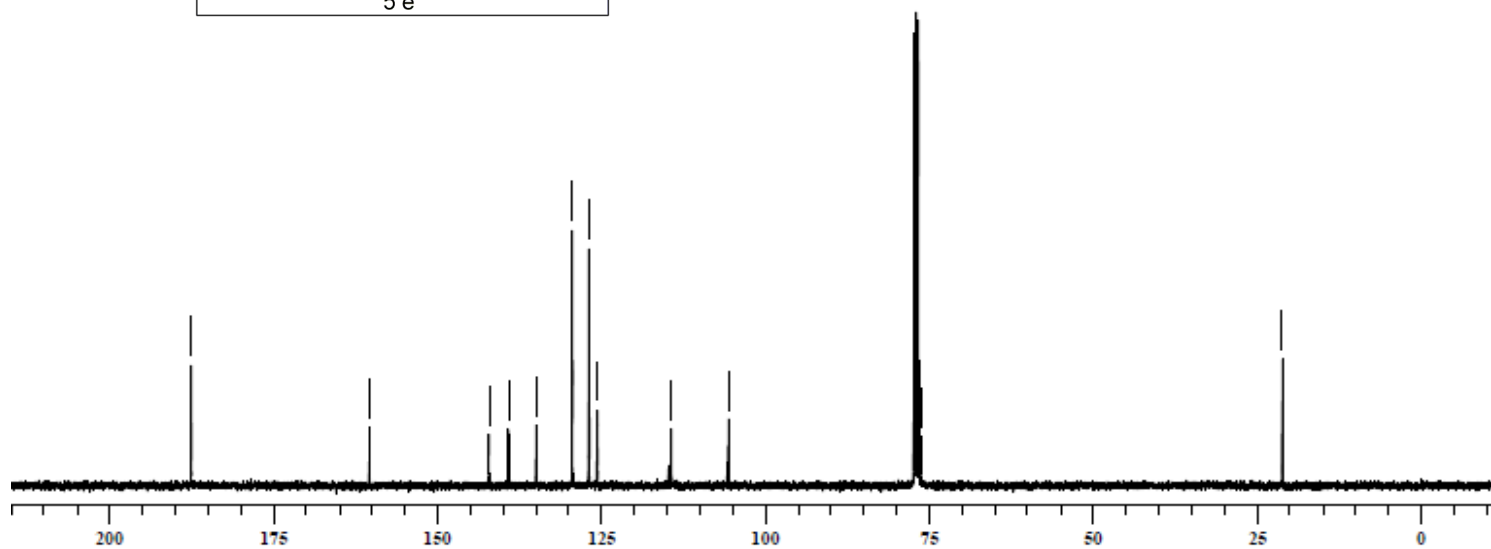
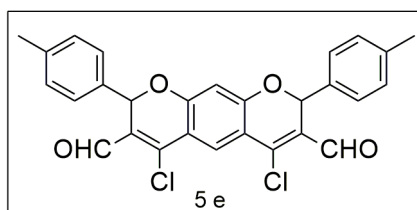
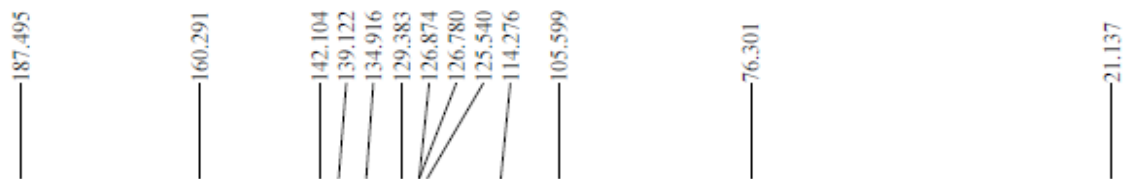
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Figure 35: ¹H NMR of 4,6-dichloro-2,8-di-p-tolyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5e)

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Figure 36: ^{13}C NMR of 4,6-dichloro-2,8-di-p-tolyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde(5e)

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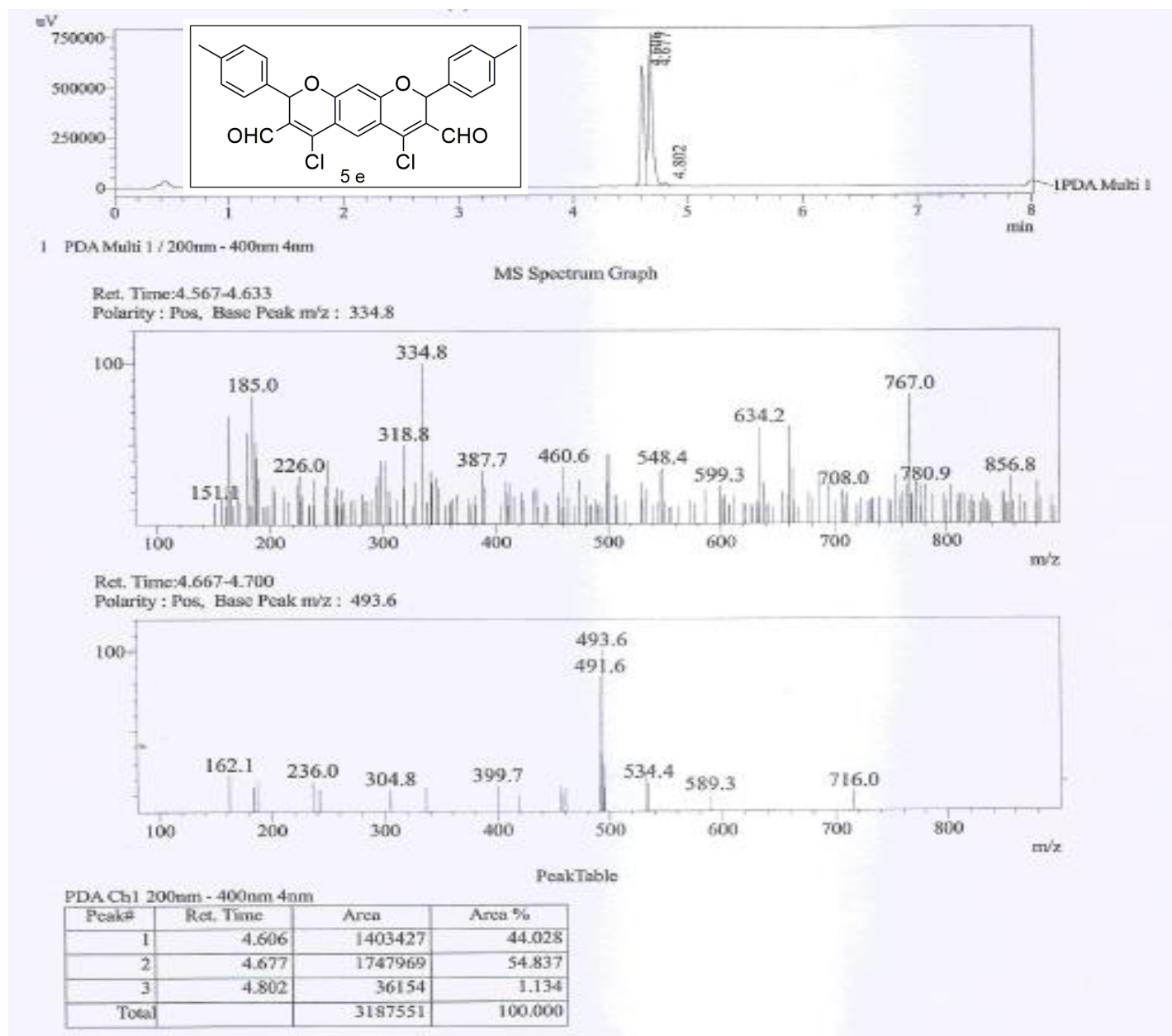


Figure 37: LCMS of 4,6-dichloro-2,8-di-p-tolyl-2,8-dihydropyrano[3,2-g]chromene-3,7-dicarbaldehyde (5e)