CuI/1,10-Phen/PEG Promoted Decarboxylation of 2, 3-Diarylacrylic acids: Synthesis of Stilbenes under Neutral and Microwave Conditions with in situ Generated Recyclable Catalyst

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Supporting Materials

Content

1. **General**

2,3-Diaryl acrylic acids were directly obtained through Perkin reaction except **1a** and **1r**. Other reagents and chromatography grade solvents were obtained from commercial sources and used without further purification unless otherwise stated. Petroleum ether (PE) used refers to the boiling fraction of 60–90 ºC. All microwave assisted reactions were carried out with Microwave Synthesizer (WBFY-205, Gongyi City Yu Hua Instrument Co. Ltd, China), and the reaction temperature was detected by infrared thermometer. The melting points are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were measured on a 400 MHz spectrometer (^{1}H 400 MHz, ^{13}C 100 MHz) using $CDCl_3$ or DMSO-d_6 as the solvent at room temperature. Chemical shifts are reported in parts per million (ppm) and are calibrated using residual undeuterated solvent as an internal reference. HRMS spectra were recorded on a LC-Q-TOF (ESI) apparatus. The single X-ray diffraction measurement was performed on X-ray diffractometer. The morphology was characterized using scanning electron microscopy (SEM, 15 kV). Thermogravimetric analysis (TGA) was carried out with a heating rate of 3 deg/min in a flowing argon atmosphere.

2. Drawing of the crystal structure of 2g

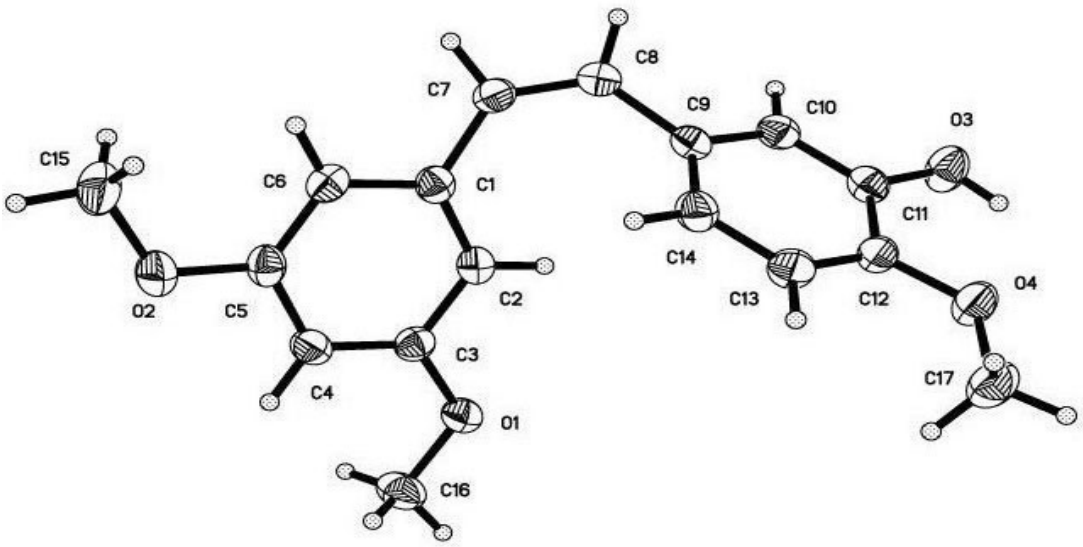
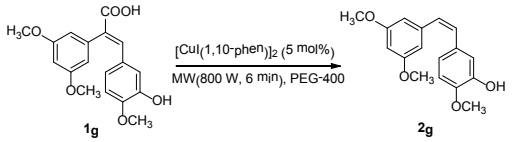


Figure 1 X-ray crystal structure of **2g**

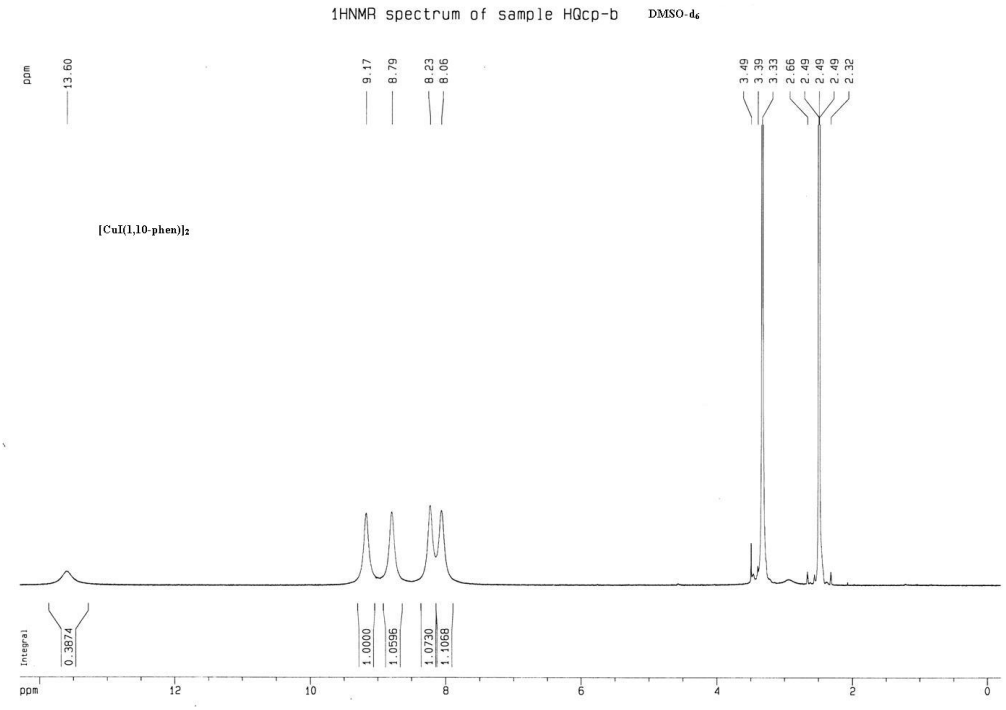
Table S1 Recyclability of [CuI(1,10-Phen)]$_2$



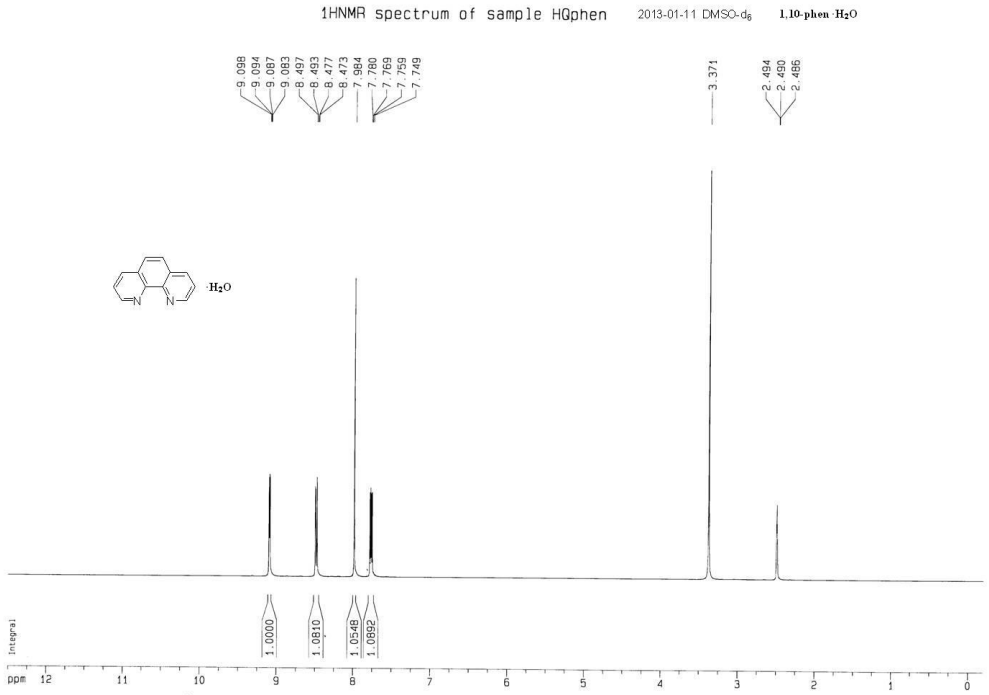
Run	Catalyst recovery(%)	T/min	Product yield(%)
1[a]	87	6	84
2[b]	86	6	83
3[b]	85	6	82
4[b]	85	6	82
5[b]	85	6	82

[a] Reaction condition: **1g** (50 mmol), [CuI(1,10-phen)]$_2$ (5 mol %) which was recovered from initial reaction, PEG-400 (100 ml), under N_2 atmosphere, the mixture was stirred under microwave irradiation (800 W, 180-190 ºC) for 6 min (2-min irradiation each time with a 5-min interval between). [b] The recovered catalyst was used under identical reaction conditions to those for the first run.

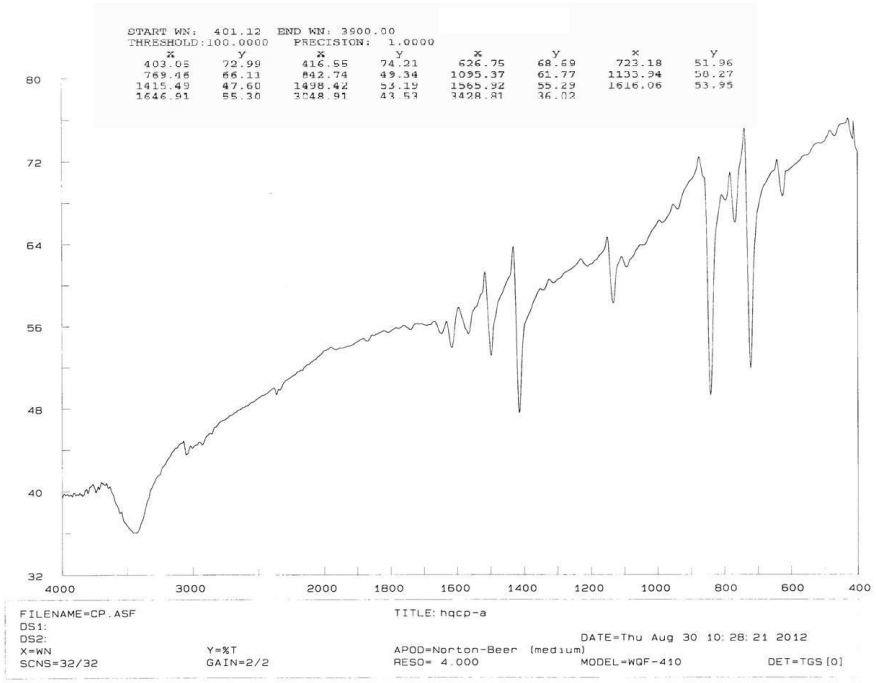
3. Characterization data of [CuI(1,10-phen)]$_2$



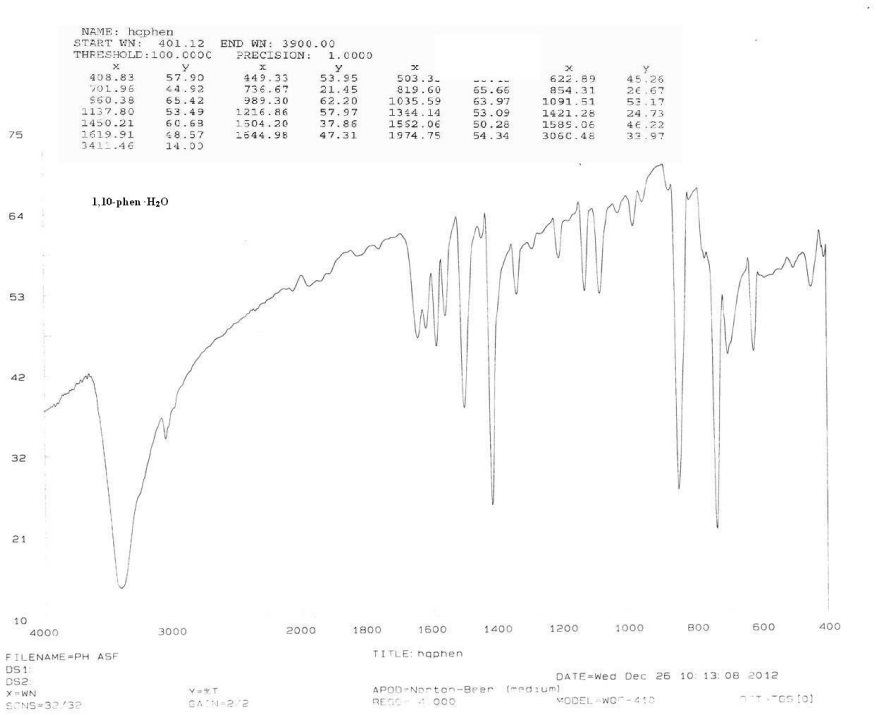
^{1}HNMR of [CuI(1,10-phen)]$_2$



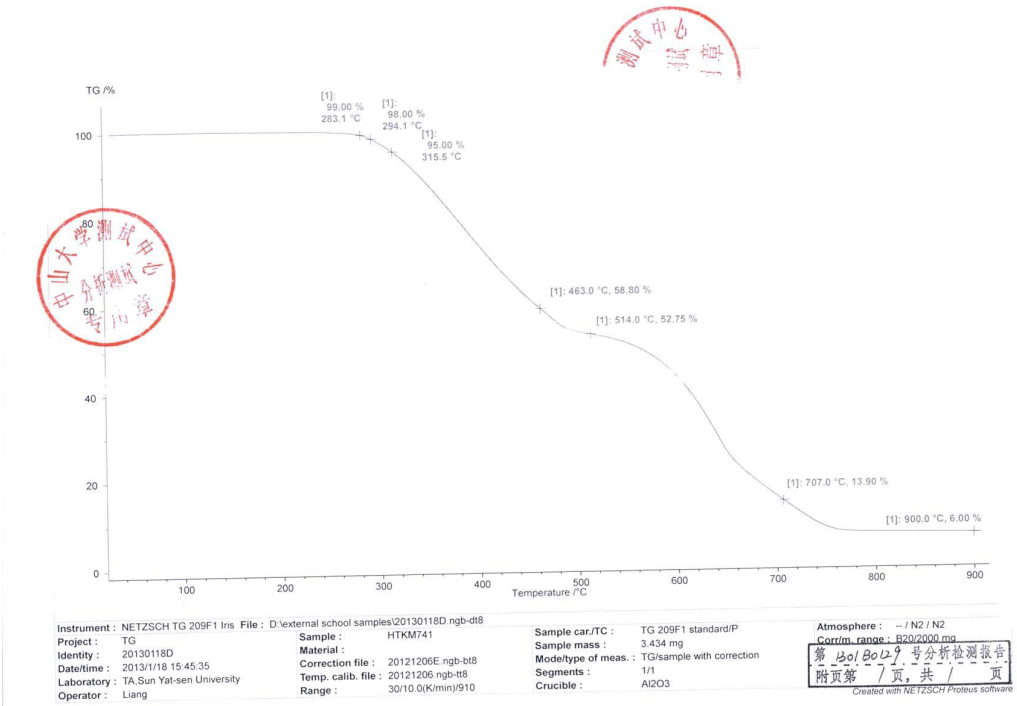
^{1}HNMR of 1,10-phen·H_2O



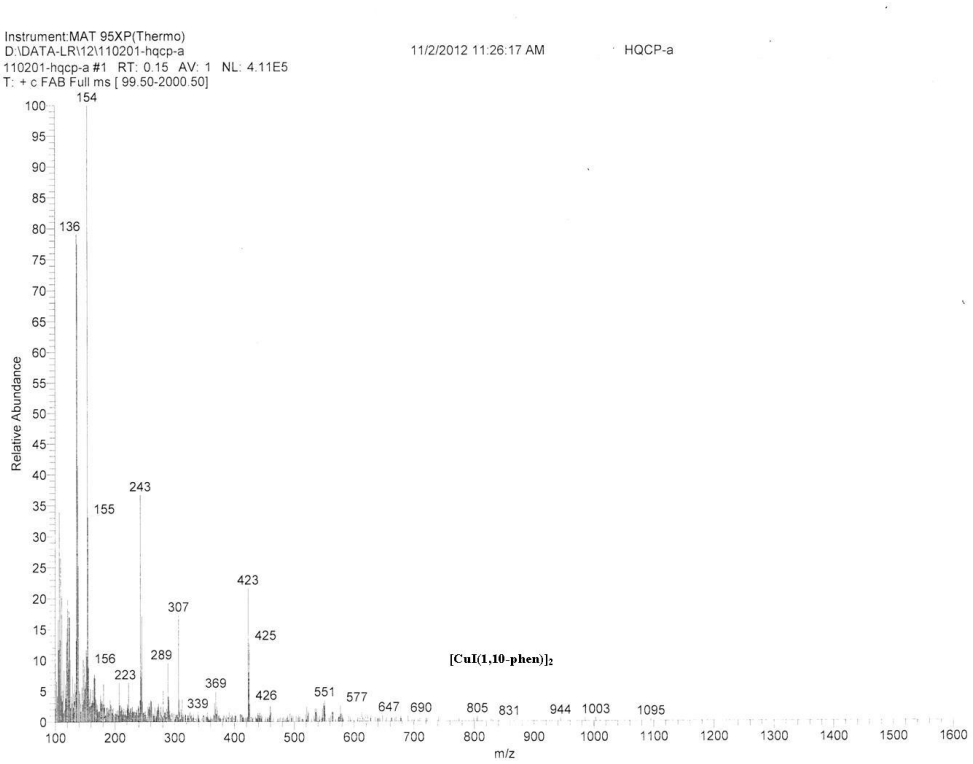
IR of $[CuI(1,10\text{-phen})]_2$



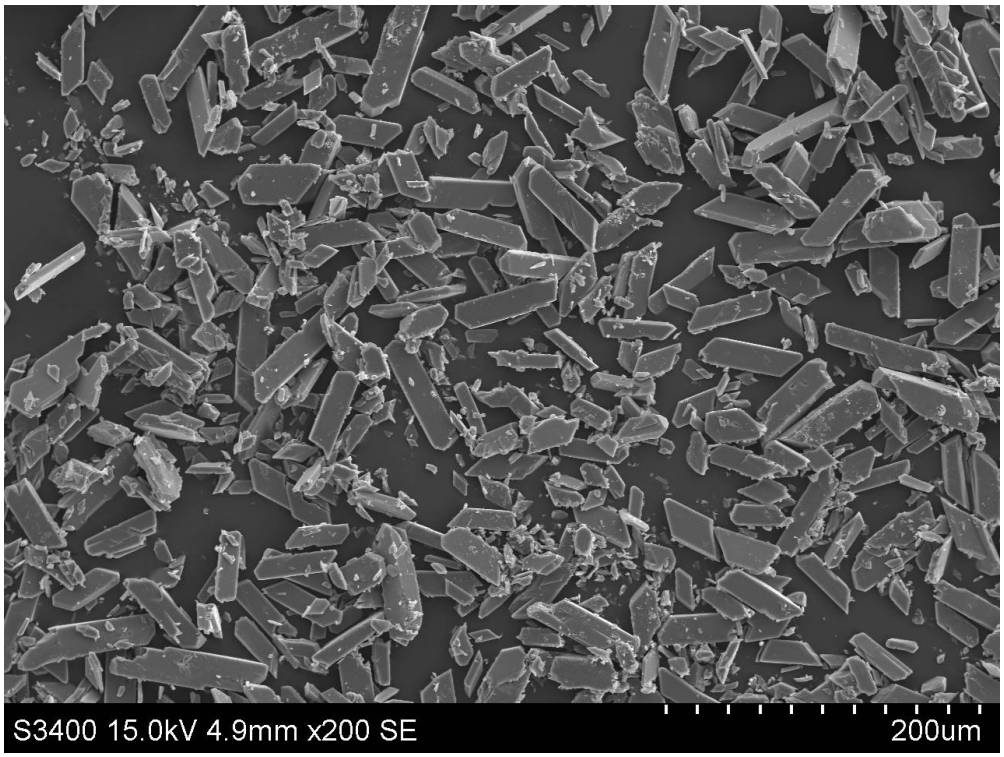
IR of 1,10-phen • H_2O

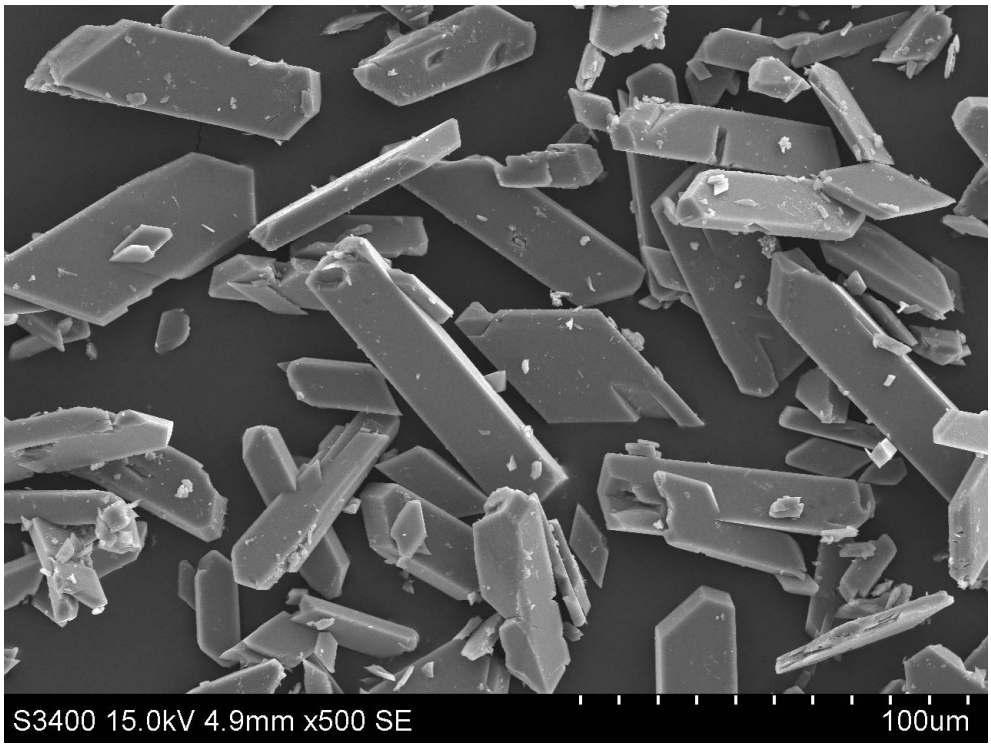


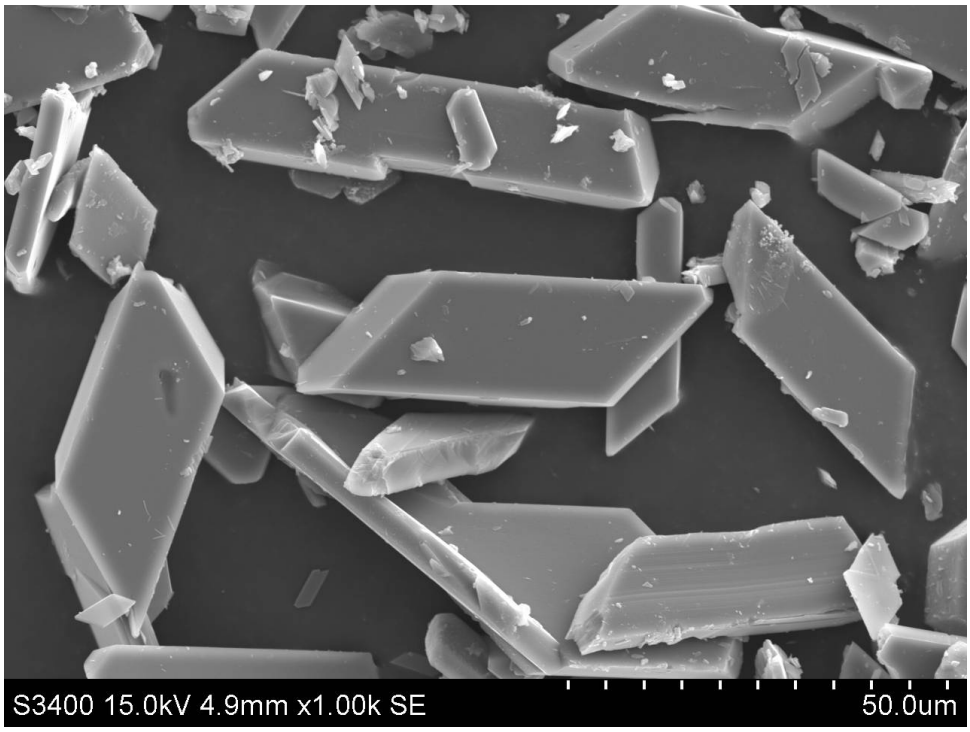
TG of $[CuI(1,10\text{-phen})]_2$



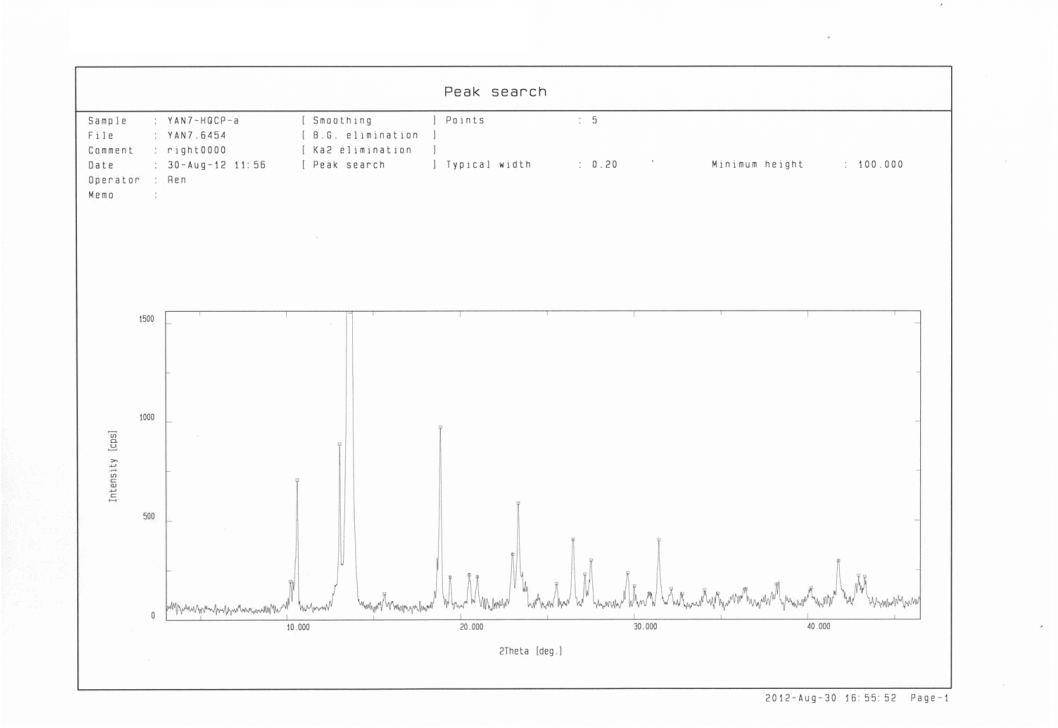
MS-FAB of $[CuI(1,10\text{-phen})]_2$





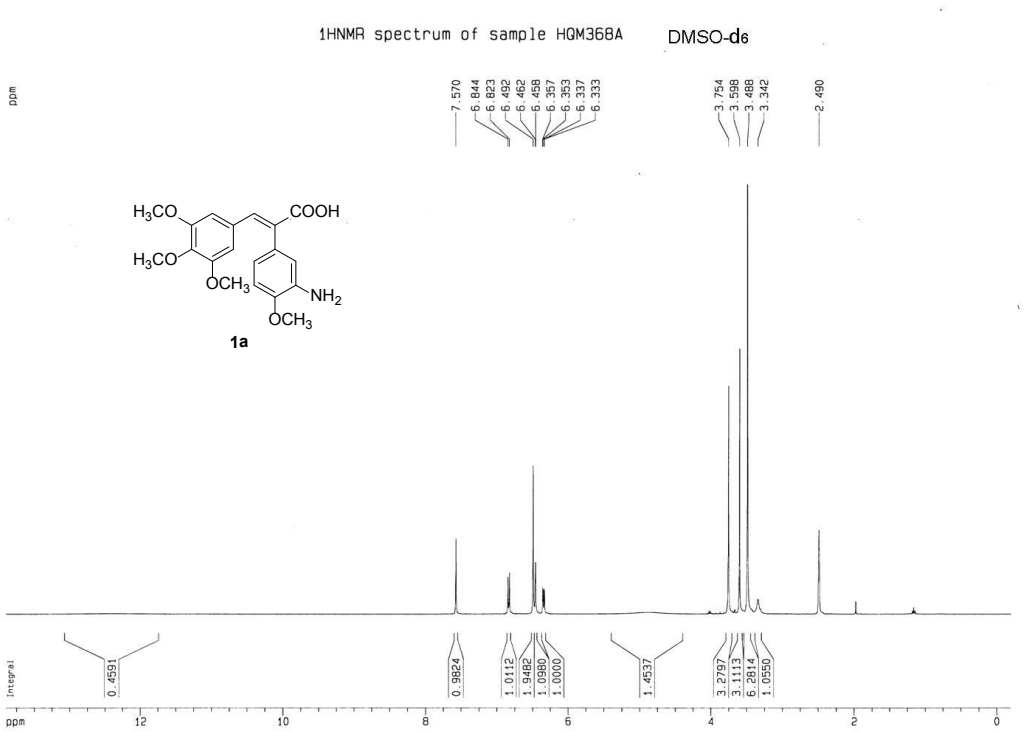


SEM of $[CuI(1,10\text{-phen})]_2$

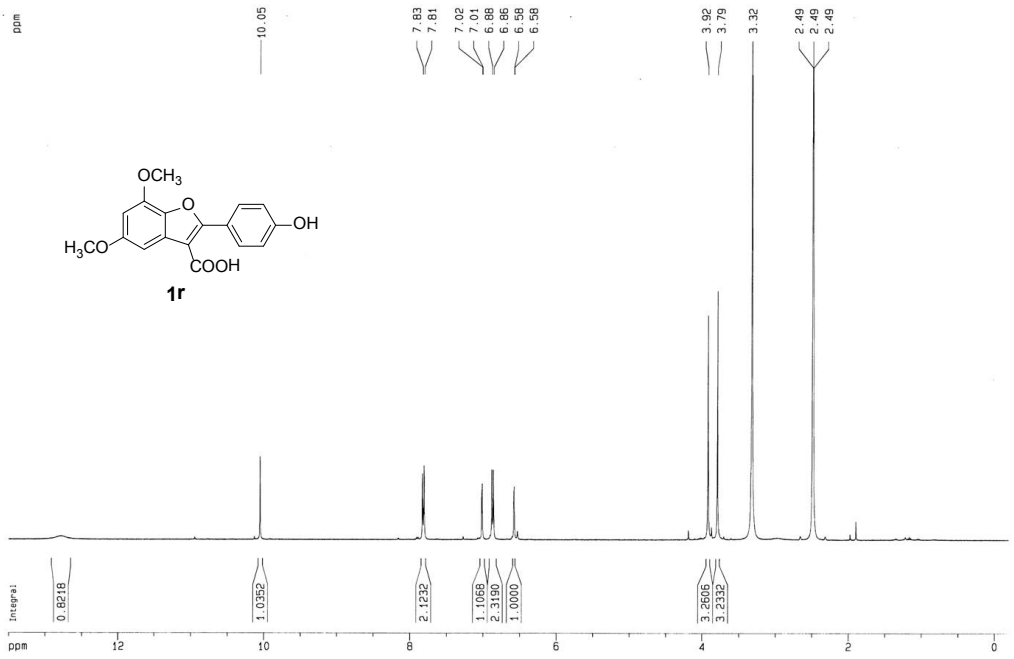


PXRD of $[Cu(1, 10\text{-phen})]_2$

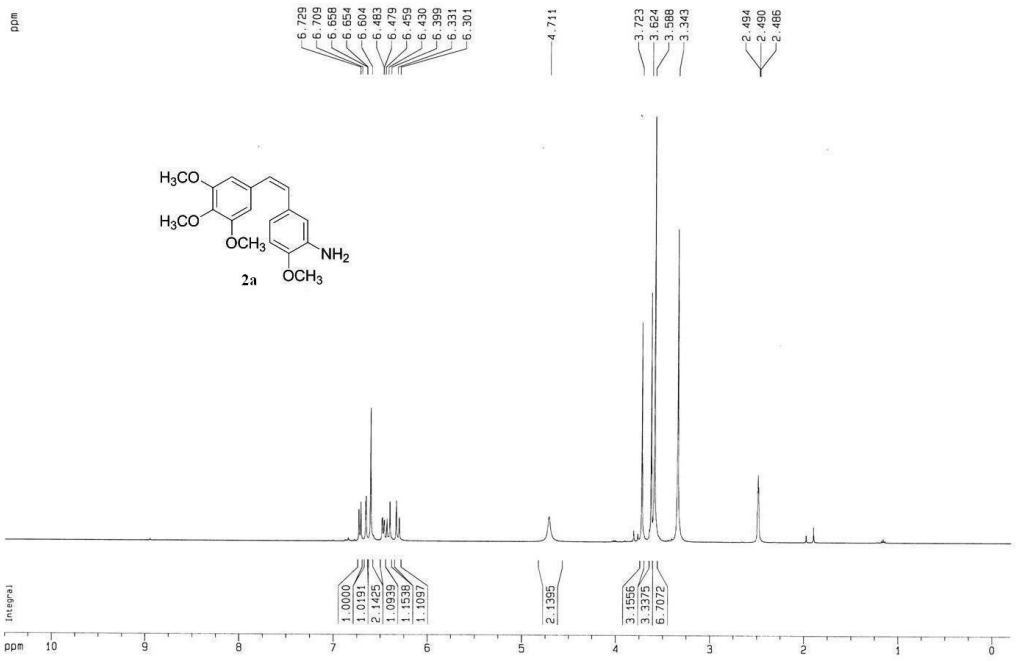
4. Spectra for products (1a, 1r and 2a-2s)



1HNMR spectrum of sample HQM298A



1HNMR spectrum of sample HQM315A DMSO-d_6



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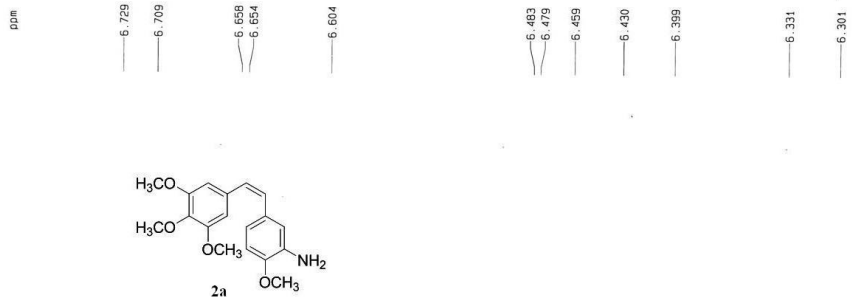
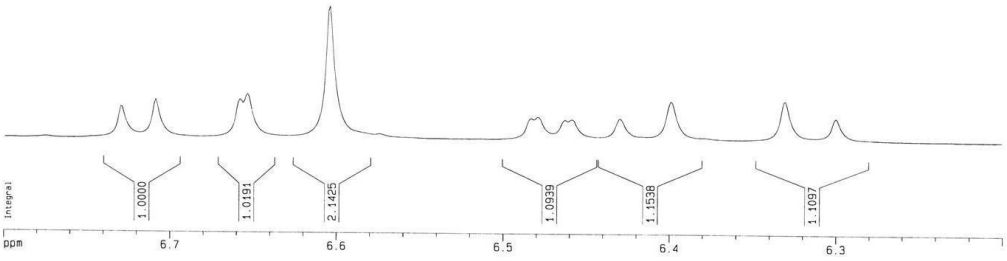
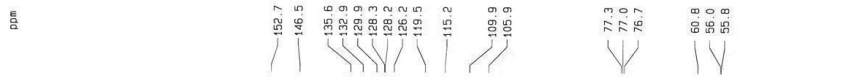
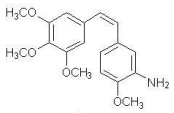


2a

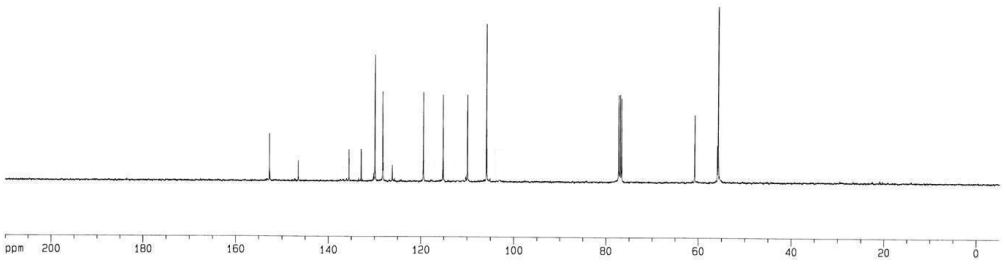


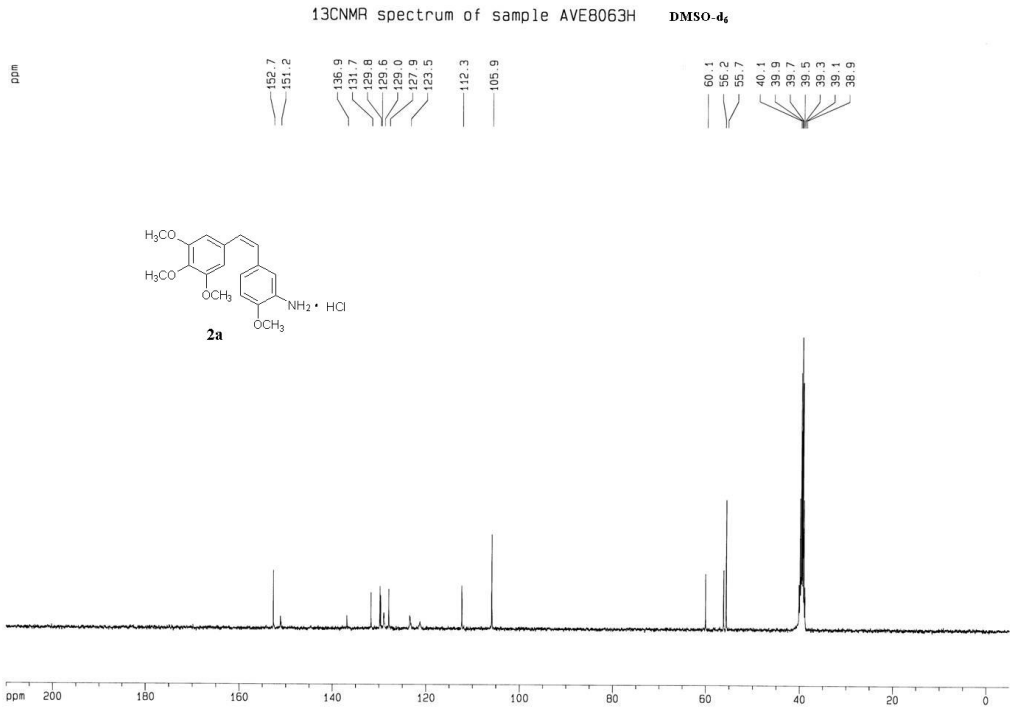
13CNMR spectrum of sample CHQM315A $CDCl_3$



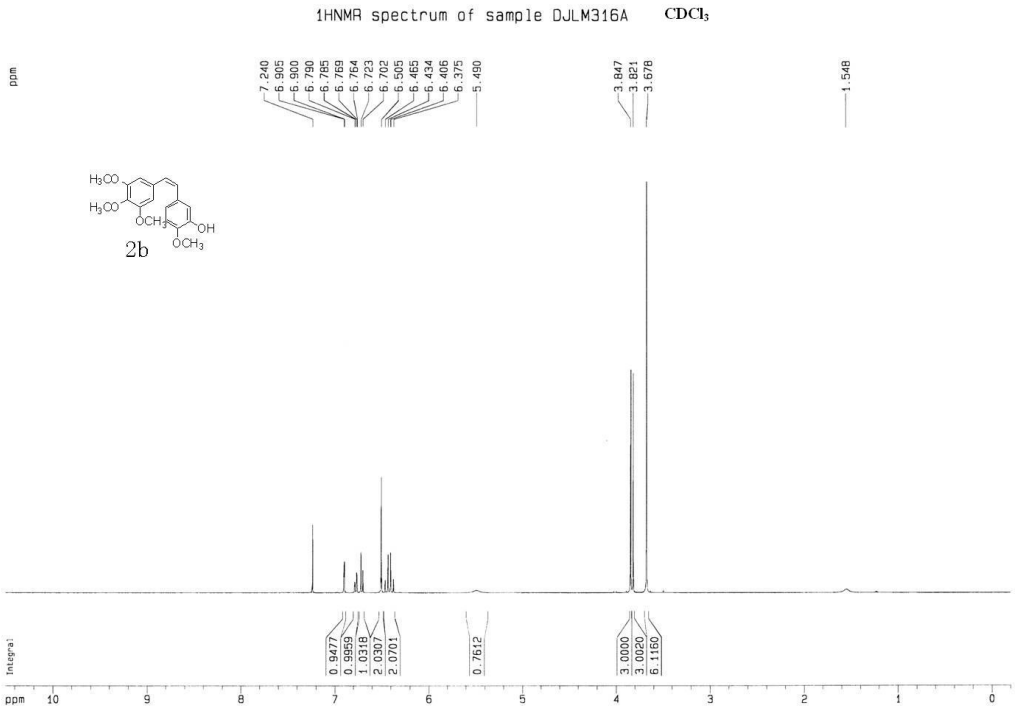


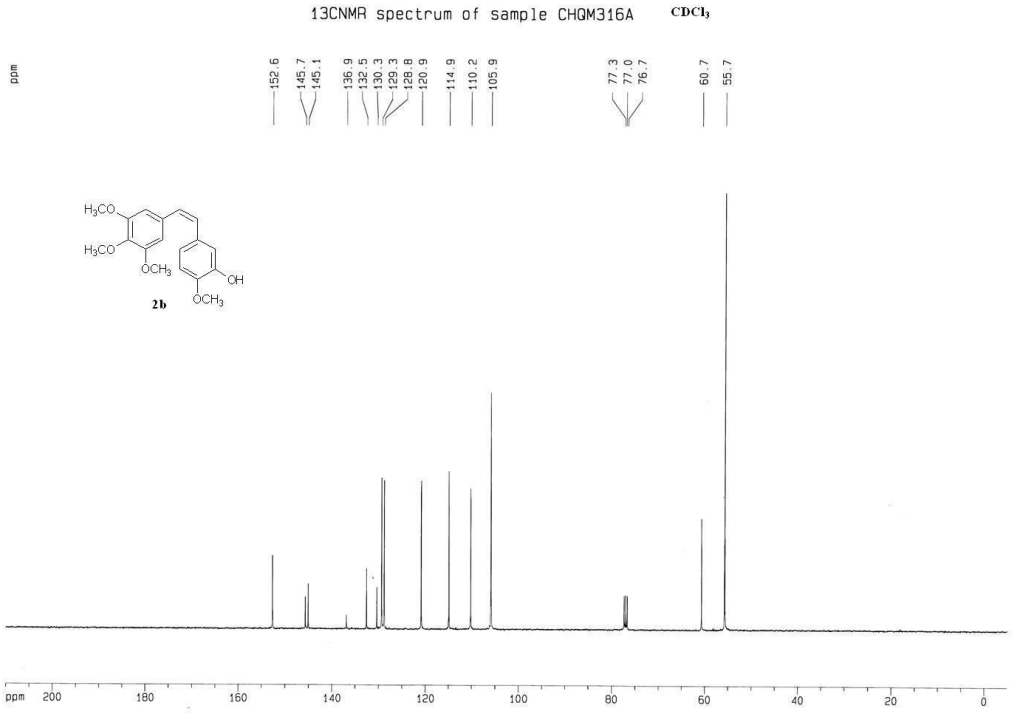
2a





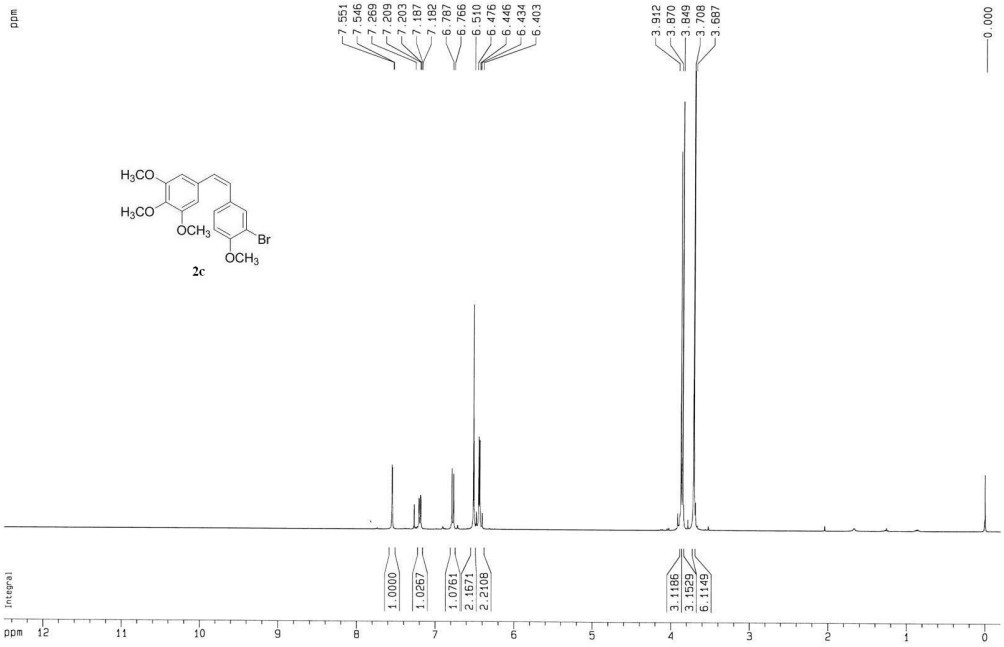
Z-3'-animo-3,4,4',5-tetramethoxystilbene (2a)



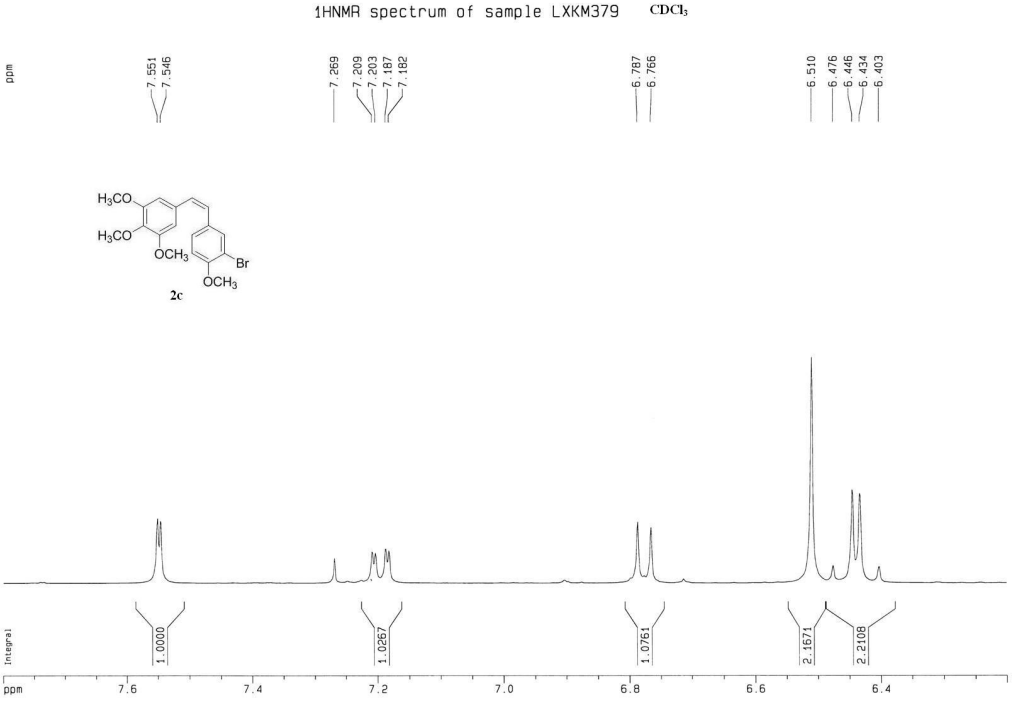


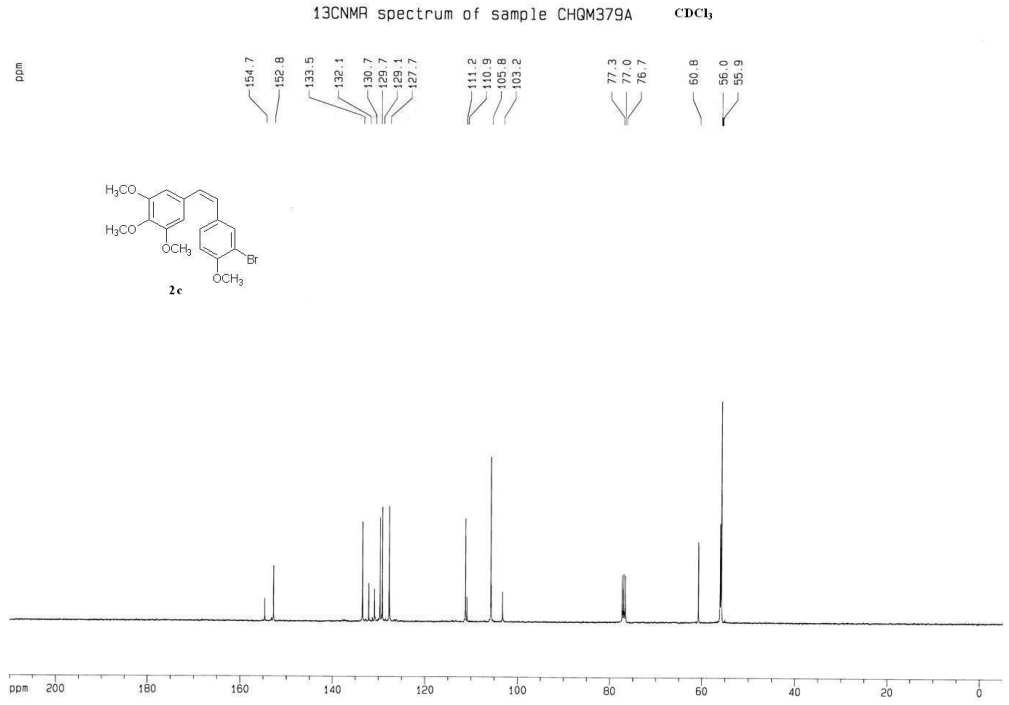
Z-3’-hydroxyl-3,4,4’,5-tetramethoxystilbene(2b)

1HNMR spectrum of sample LXKM379 $CDCl_3$

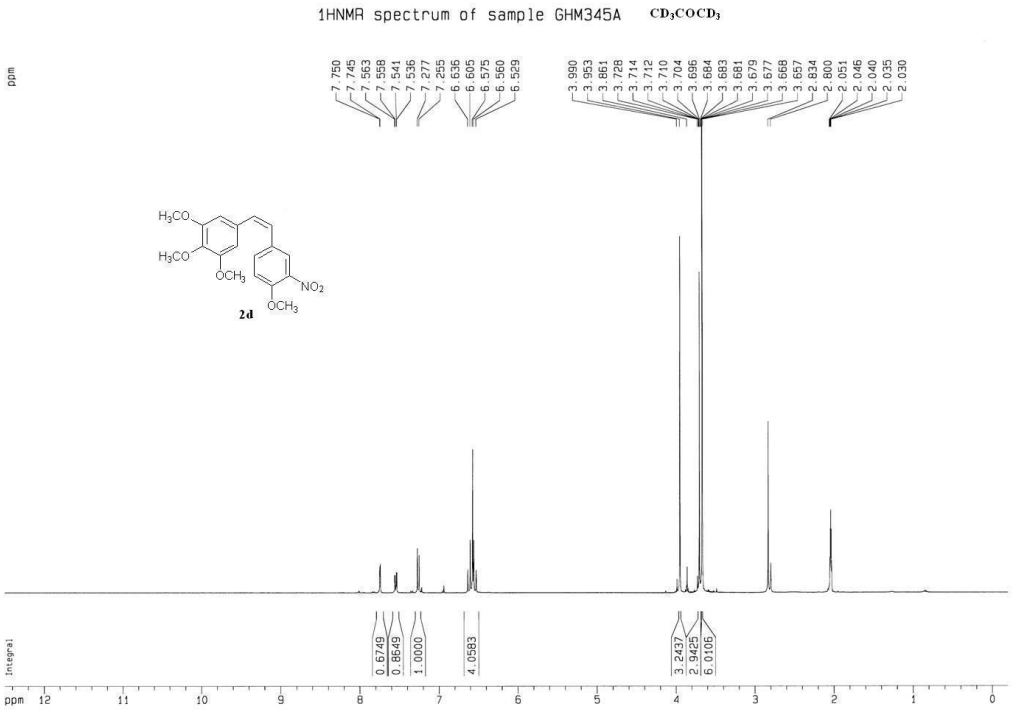


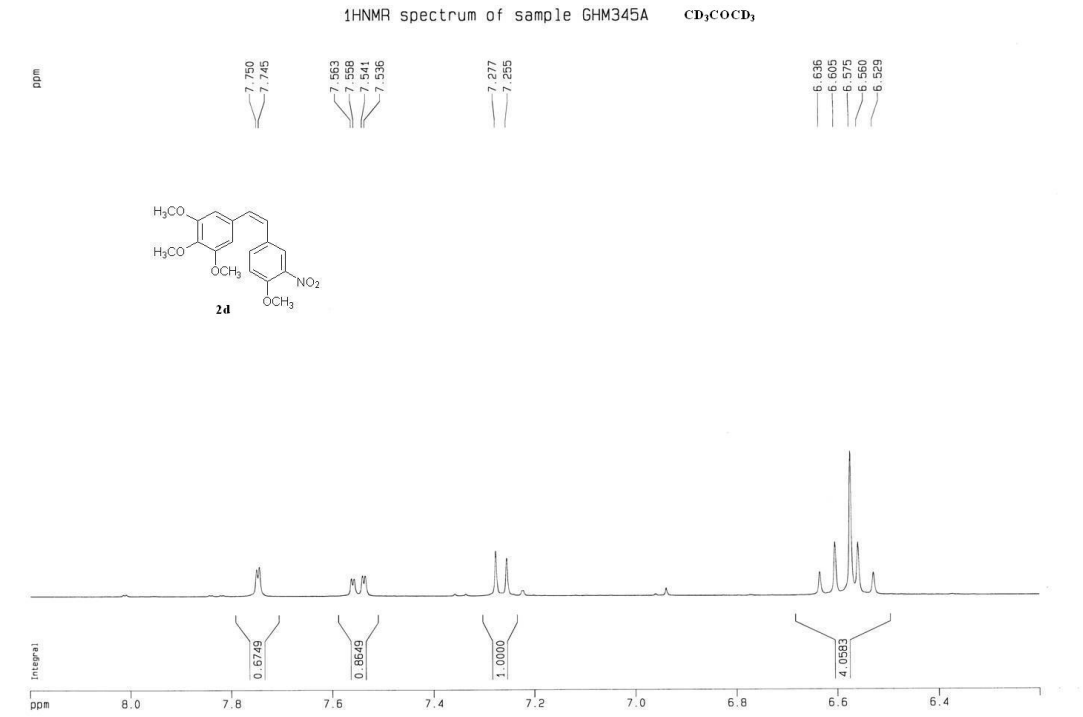
1HNMR spectrum of sample LXKM379 $CDCl_3$

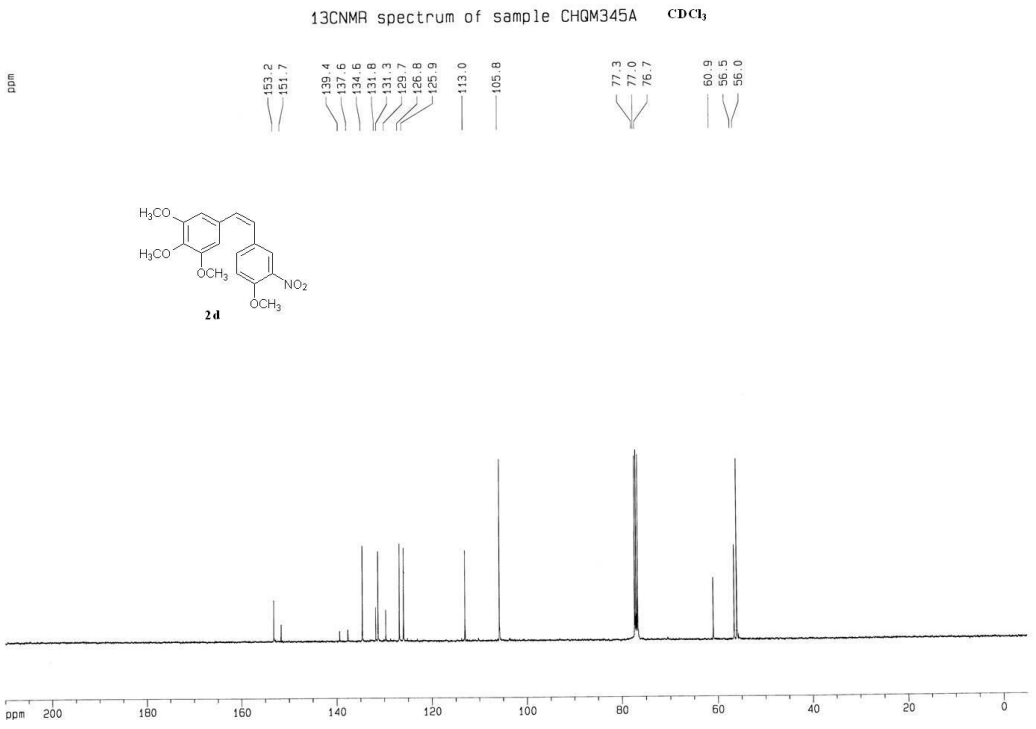




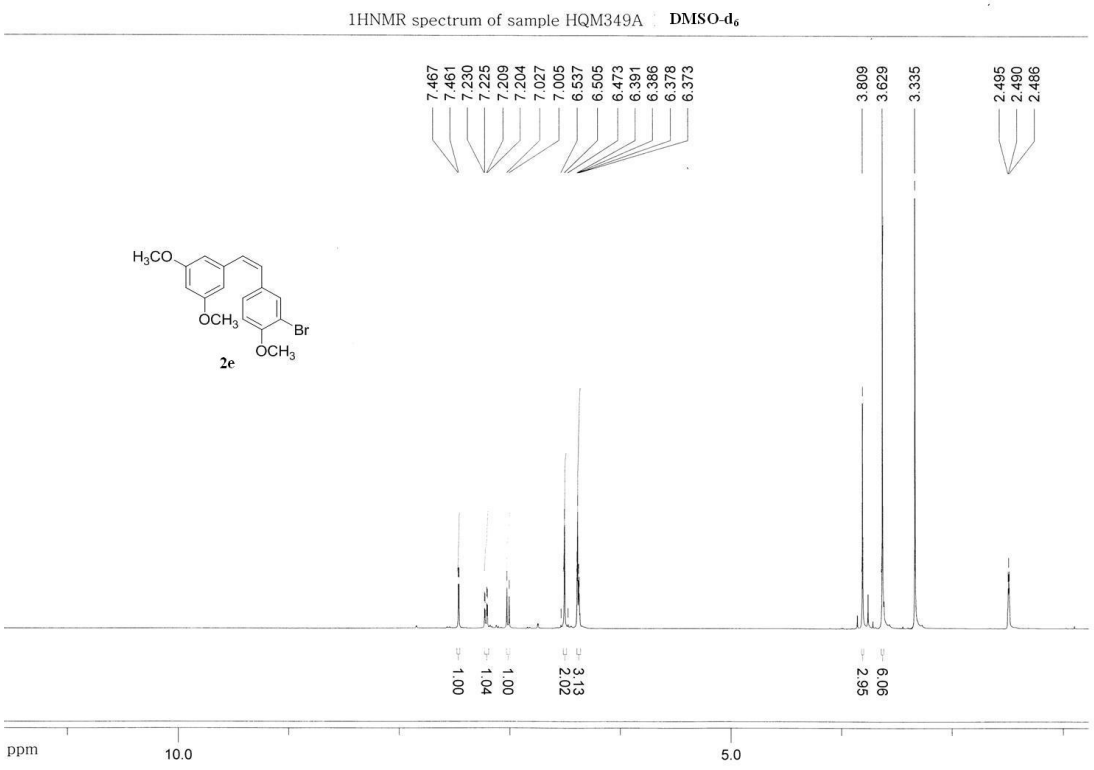
Z-3'-bromo-3,4,4',5-tetramethoxystilbene (2c)







Z-3’-nitro-3,4,4’,5-tetramethoxystilbene(2d)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7.461
7.230
7.225
7.209
7.204
7.027
7.005
6.537
6.505
6.473
6.391
6.386
6.378
6.373
3.809
3.629
3.335
2.495
2.490
2.486
H3CO
OCH3
Br
OCH3
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1.00
3.13
2.02
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ppm
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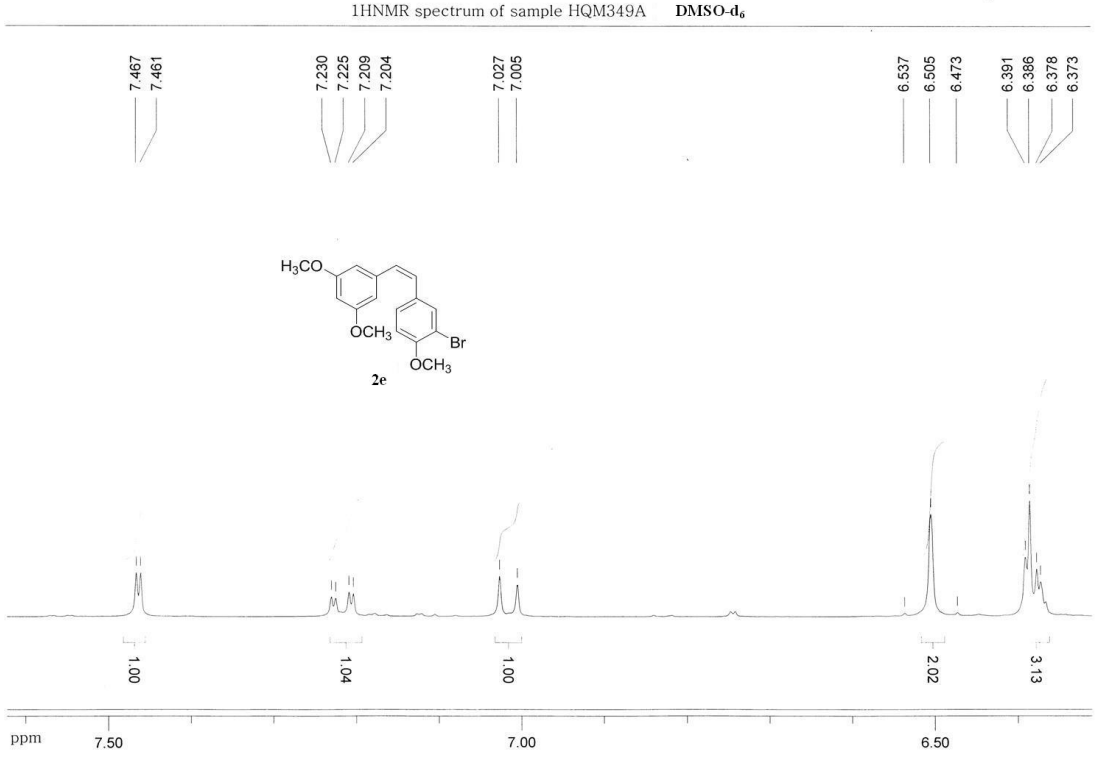
5.0


1HNMR spectrum of sample HQM349A DMSO-d6
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7.230
7.225
7.209
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7.005
6.537
6.505
6.473
6.391
6.386
6.378
6.373
H3CO
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1.00
1.04
1.00
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3.13
ppm
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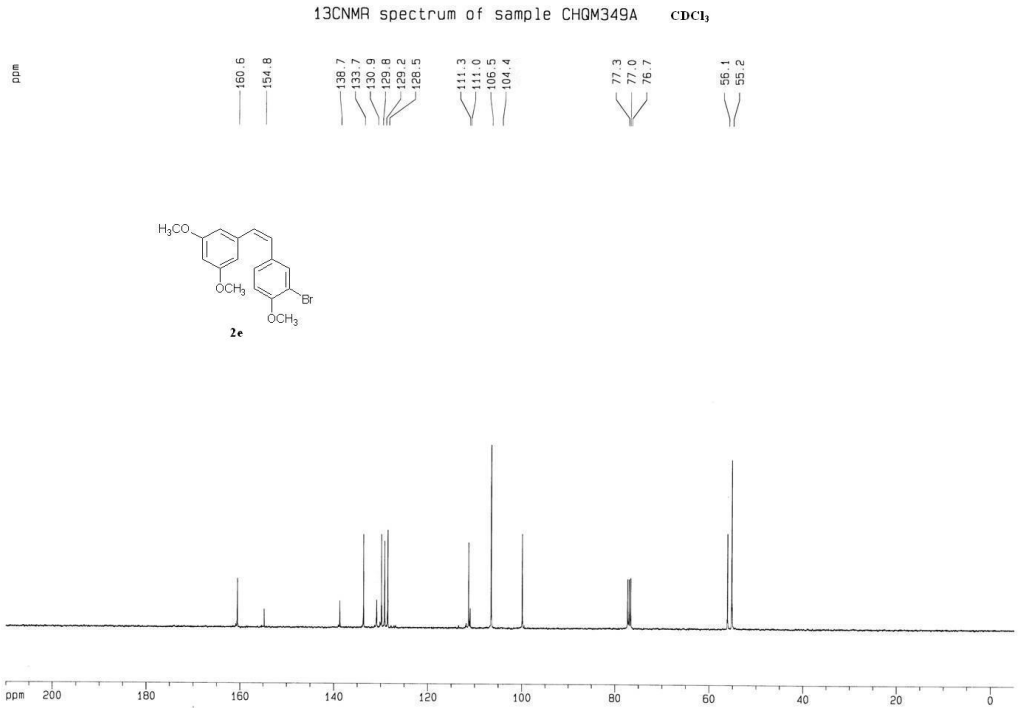
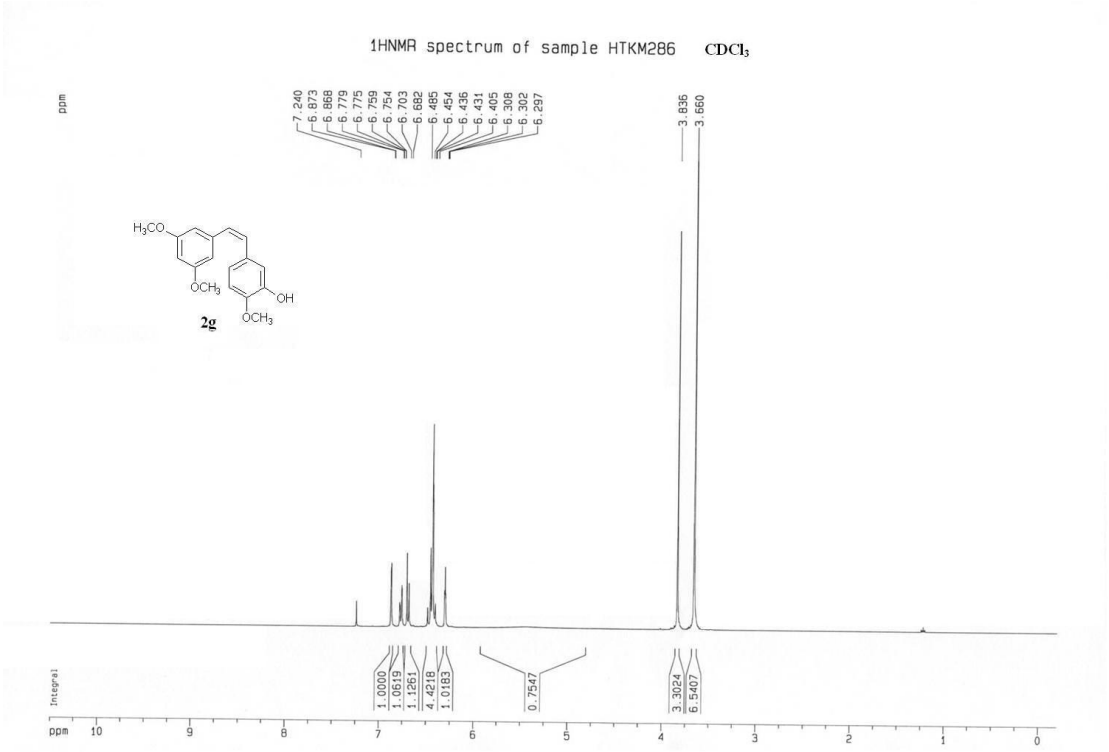
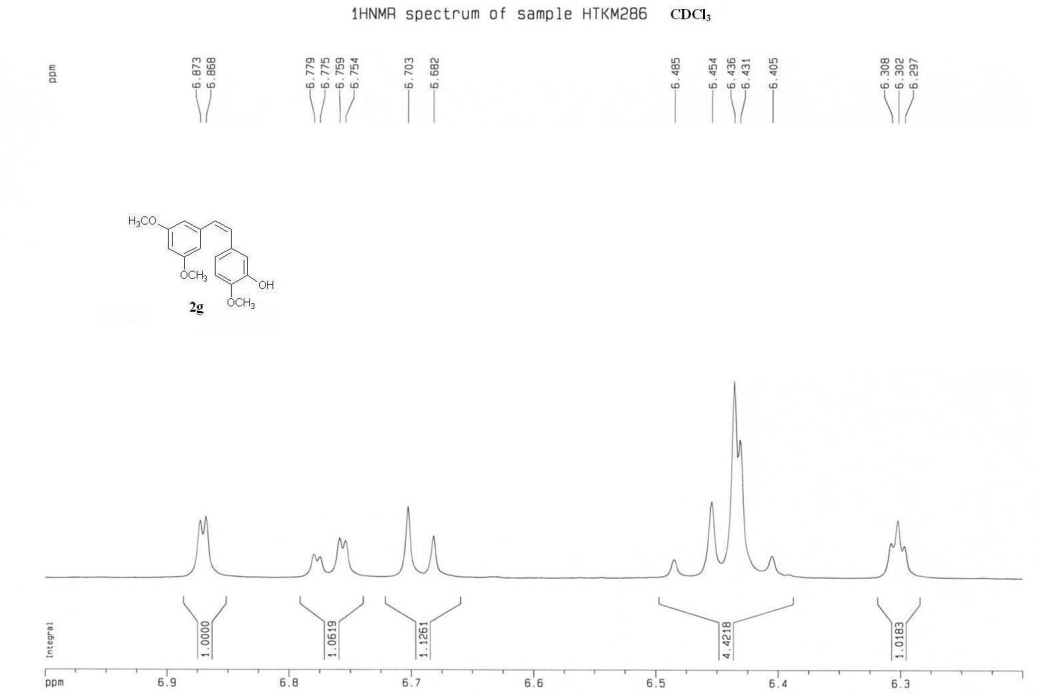
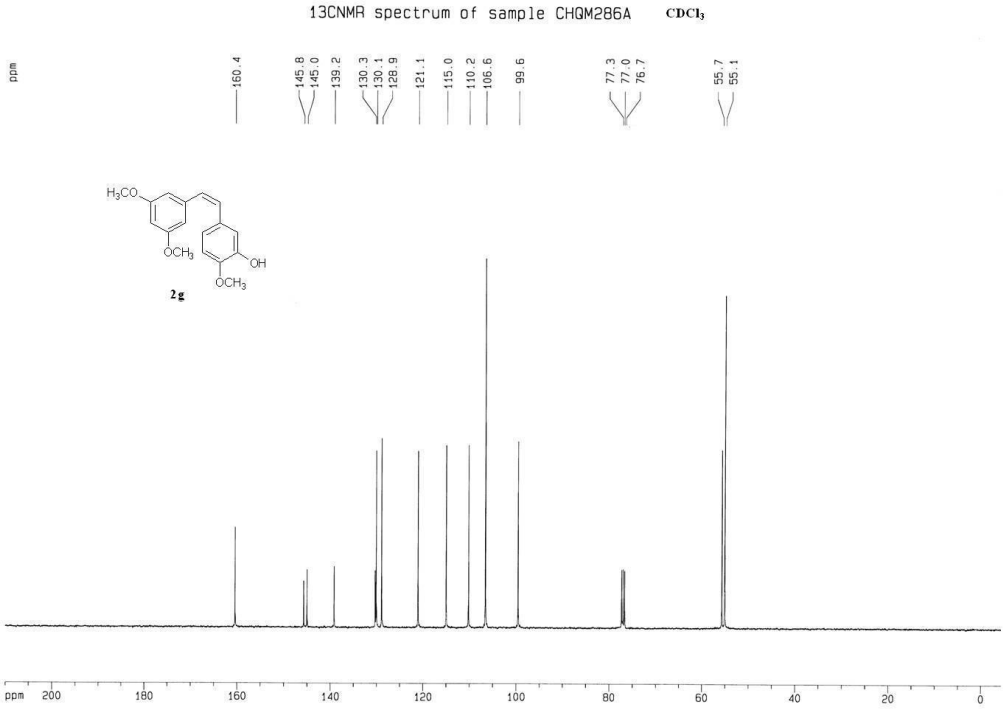
7.00
6.50



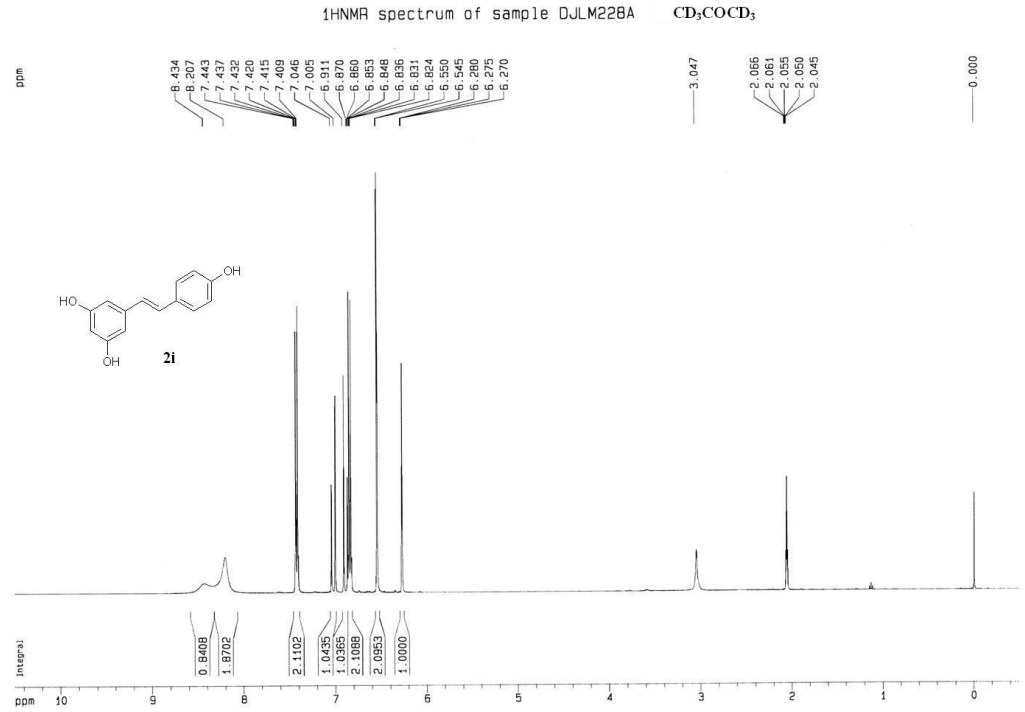
Z-3'-bromo-3,4',5-trimethoxystilbene (2e)

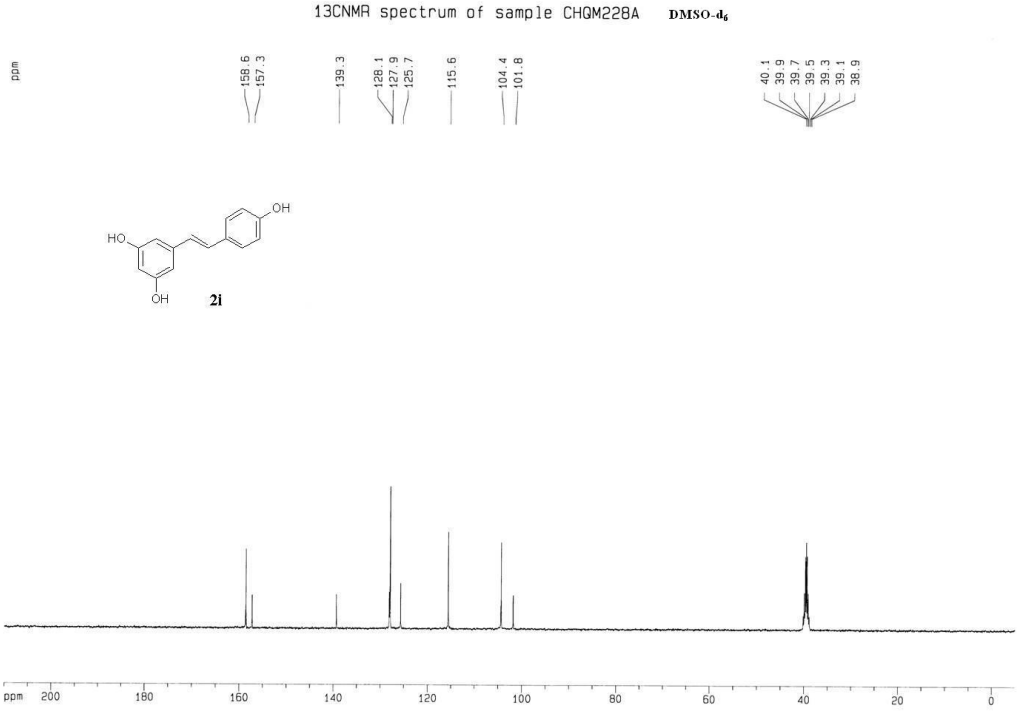




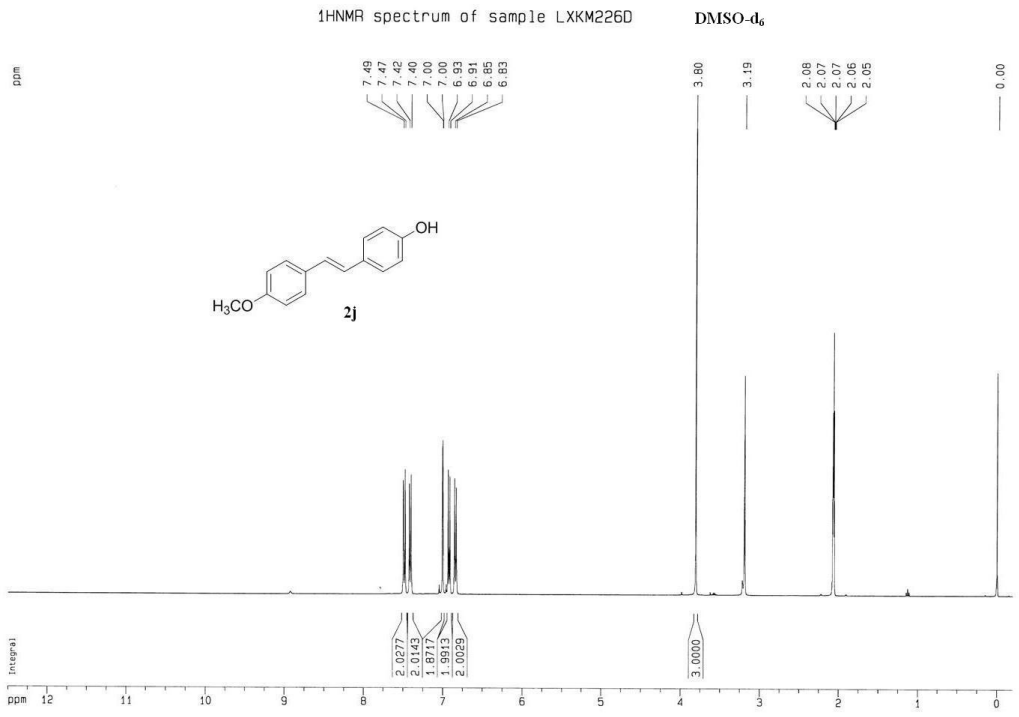


Z-3'-hydroxyl-3,4',5-trimethoxystilbene (2g)





E-3,4’,5-trihydroxylstilbene (2i)


1HNMR spectrum of sample LXKM226D
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7.00
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6.91
6.85
6.83
3.80
3.19
2.08
2.07
2.07
2.06
2.05
0.00
OH
H3CO
2j
Integral
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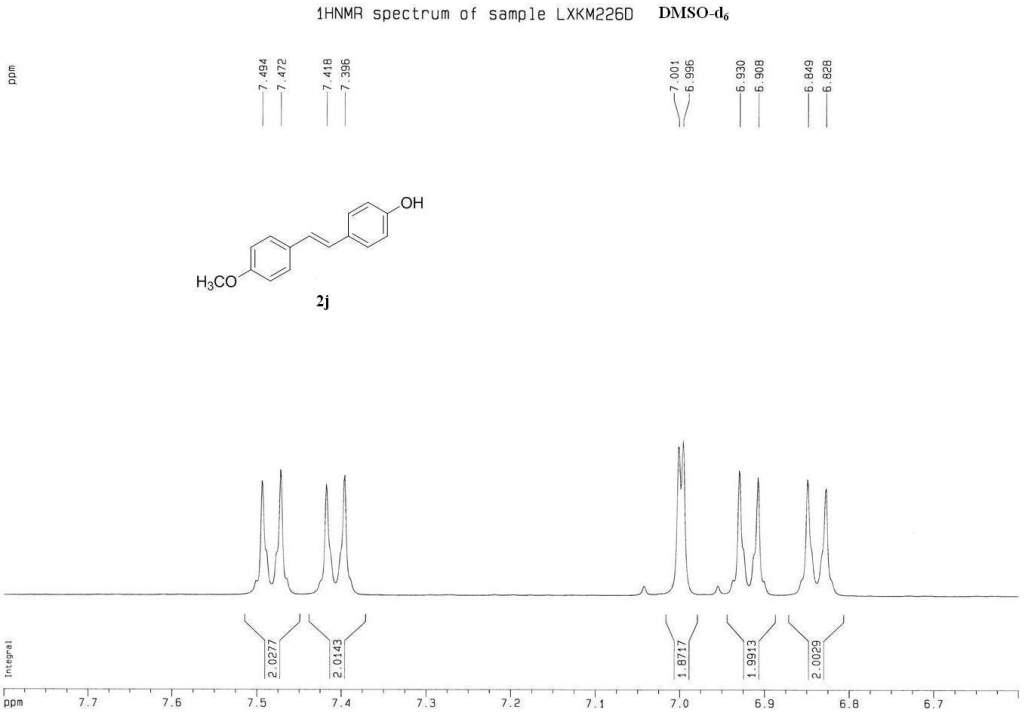
3.0000
ppm 12 11 10 9 8 7 6 5 4 3 2 1 0


1HNMR spectrum of sample LXKM226D
DMSO-d6
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6.908
6.849
6.828
OH
H3CO
2j
Integral
2.0277
2.0143
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1.9913
2.0029
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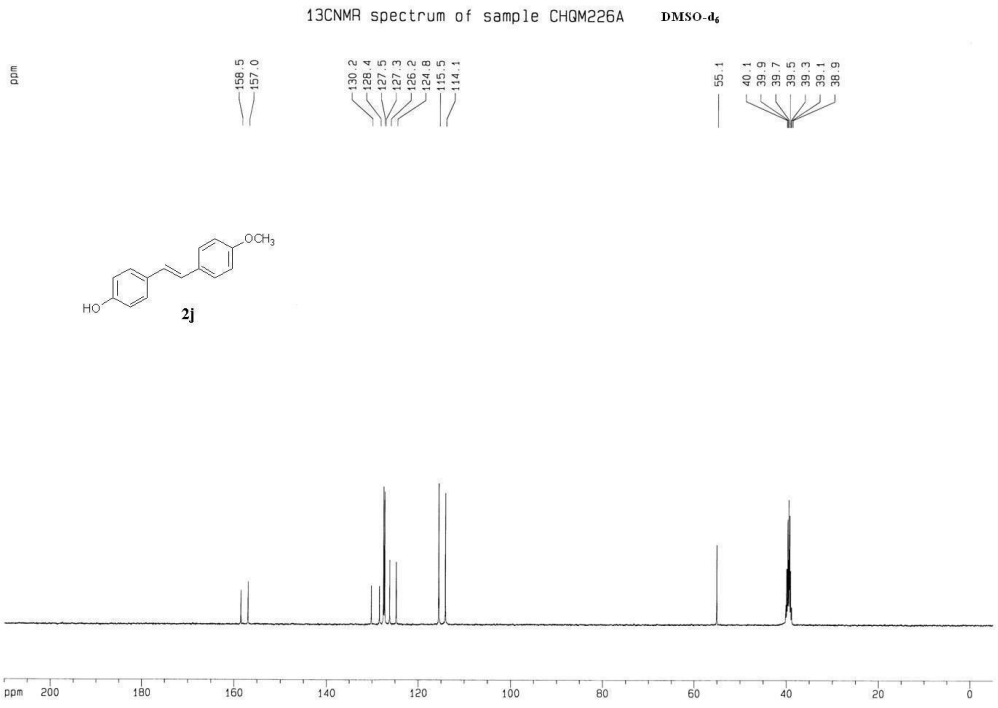
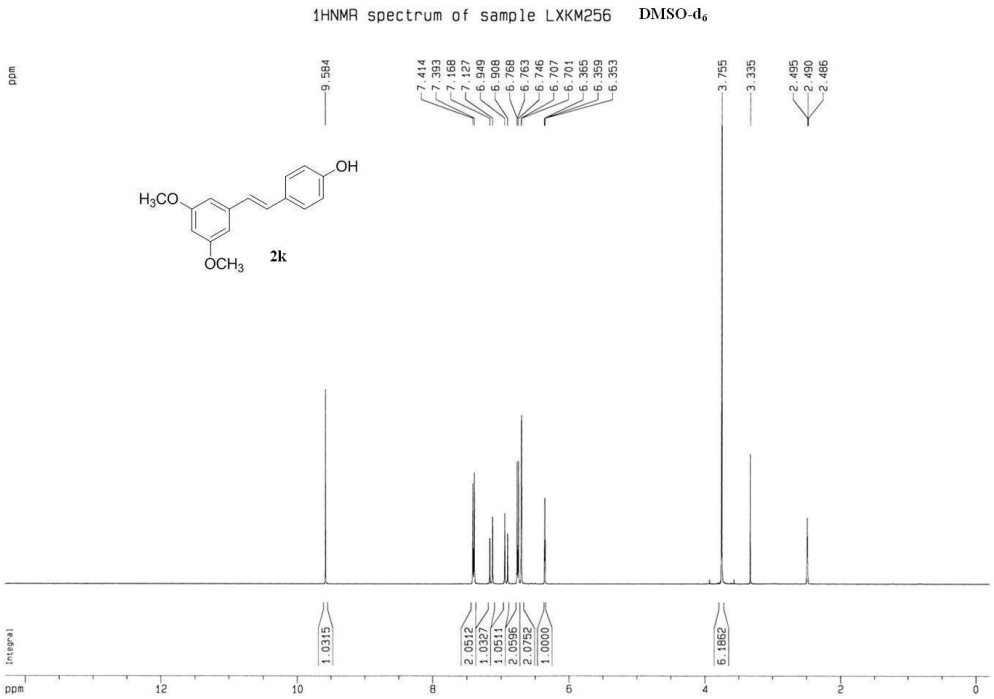
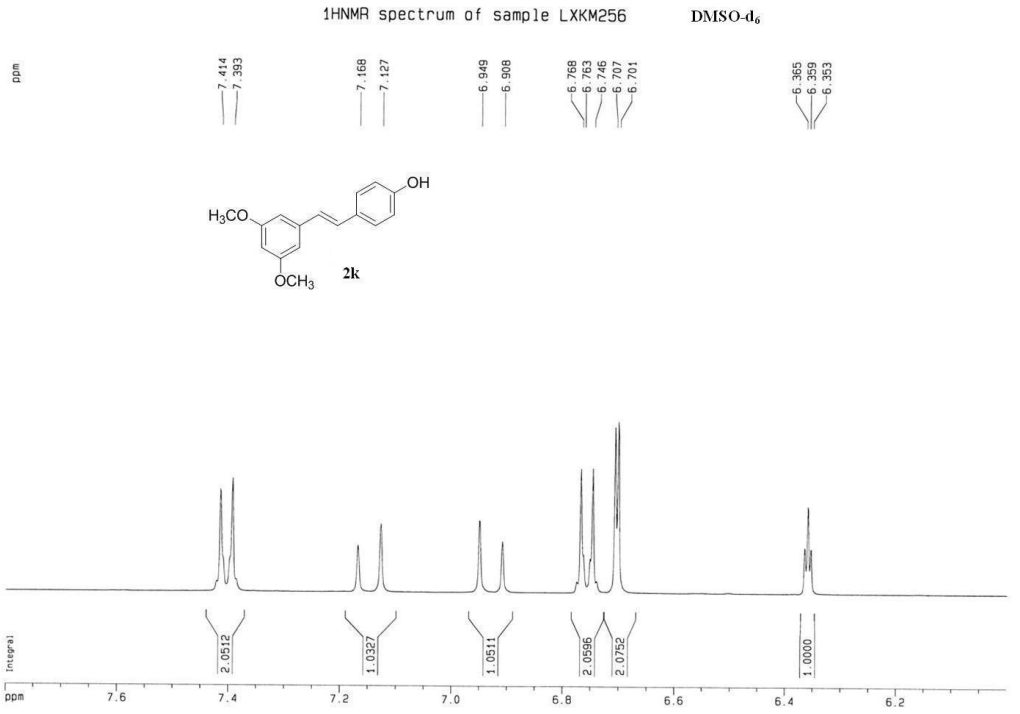
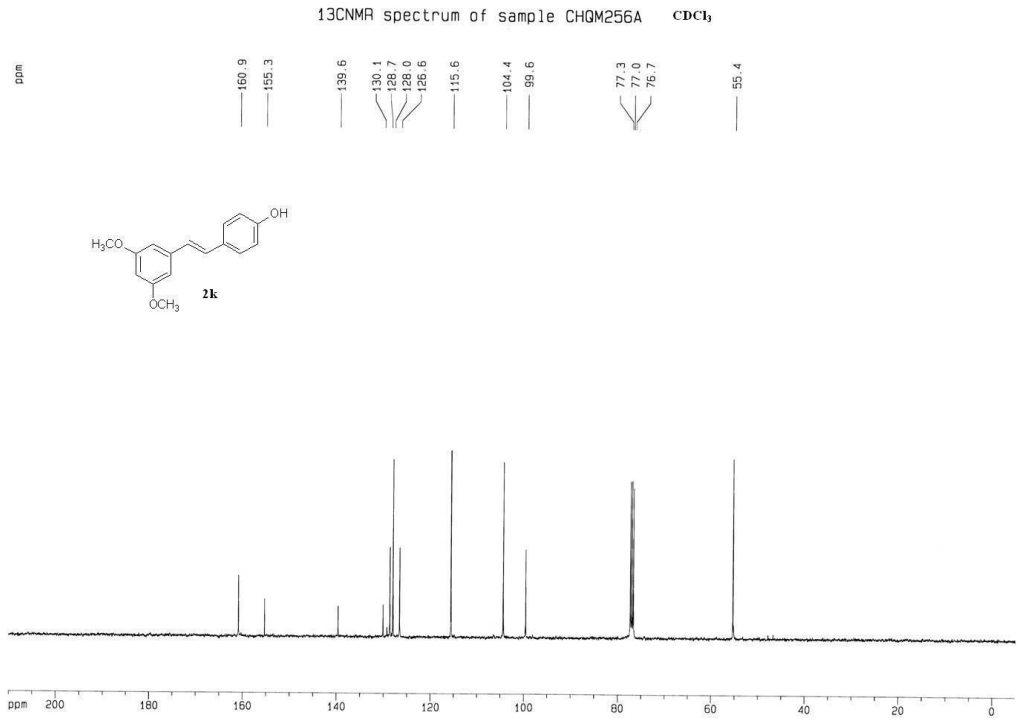


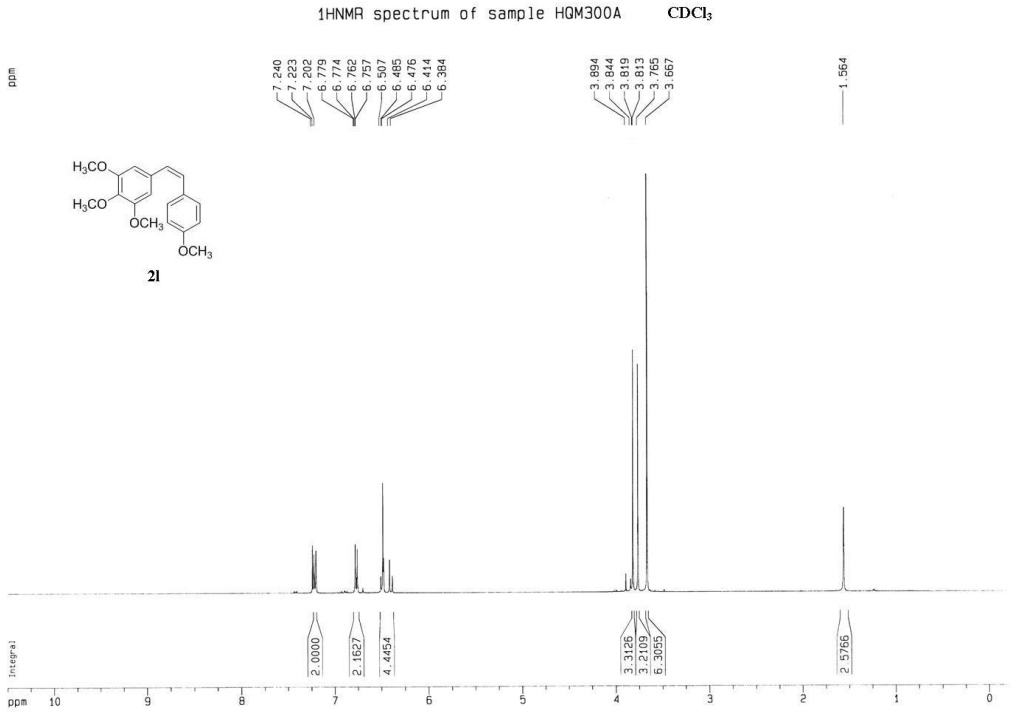
E-4’-hydroxy-4-methoxylstilbene (2j)







E-4’-hydroxy-3,5-dimethoxylstilbene (2k)


1HNMR spectrum of sample HQM300A CDCl3
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7.202
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6.757
6.507
6.485
6.476
6.414
6.384
3.894
3.844
3.819
3.813
3.765
3.667
1.564
H3CO
H3CO
OCH3
OCH3
2l
Integral
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4.4454
3.3126
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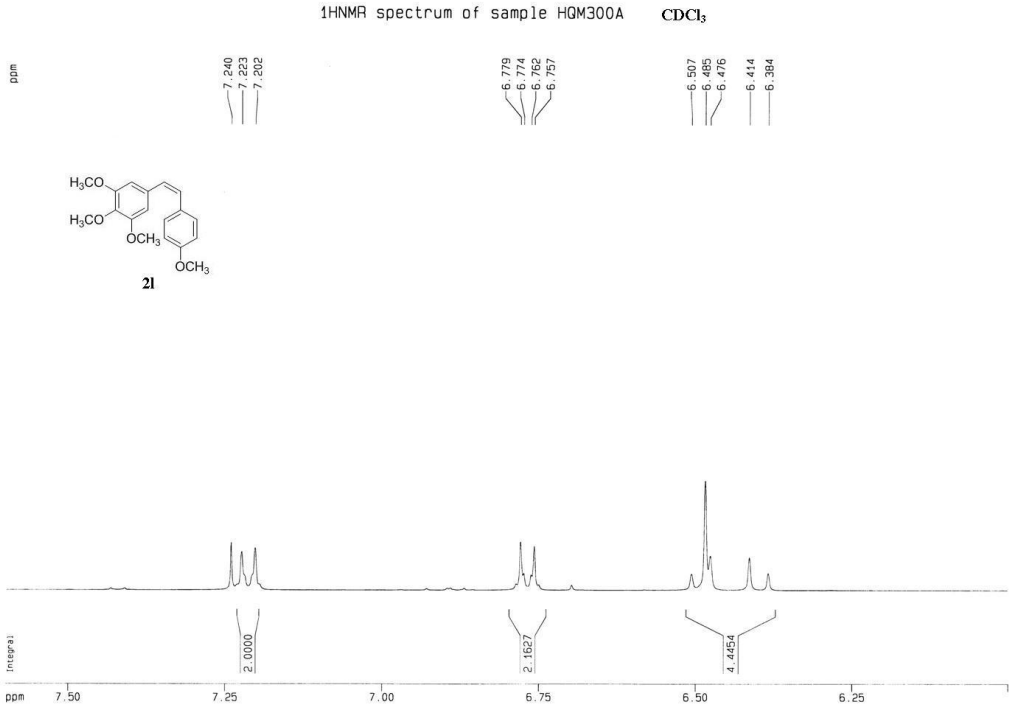
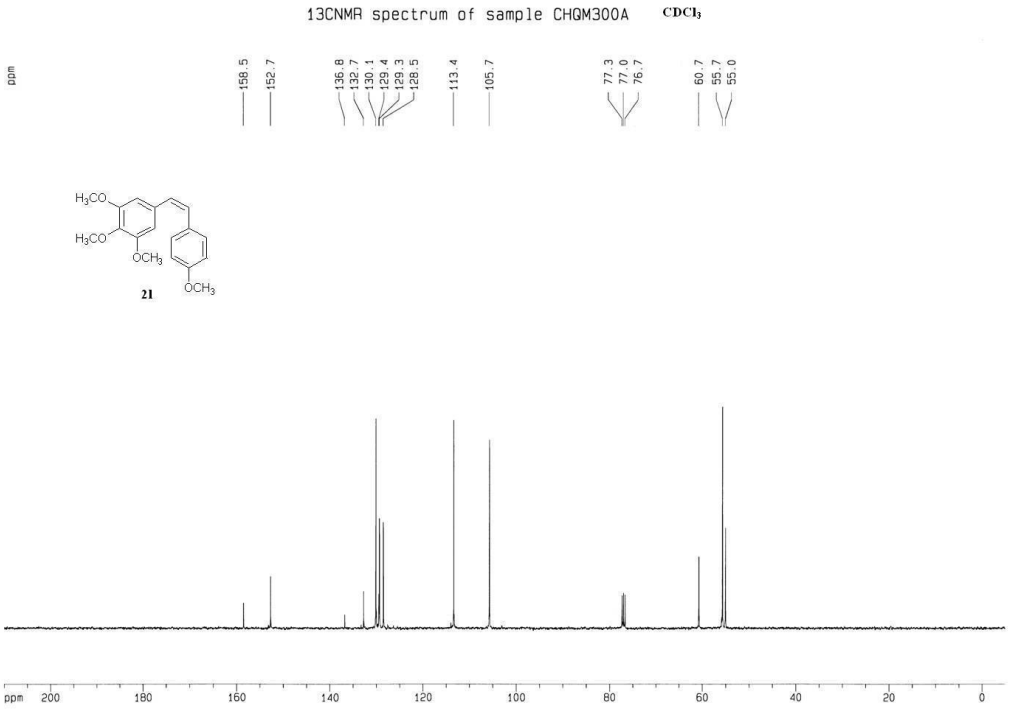
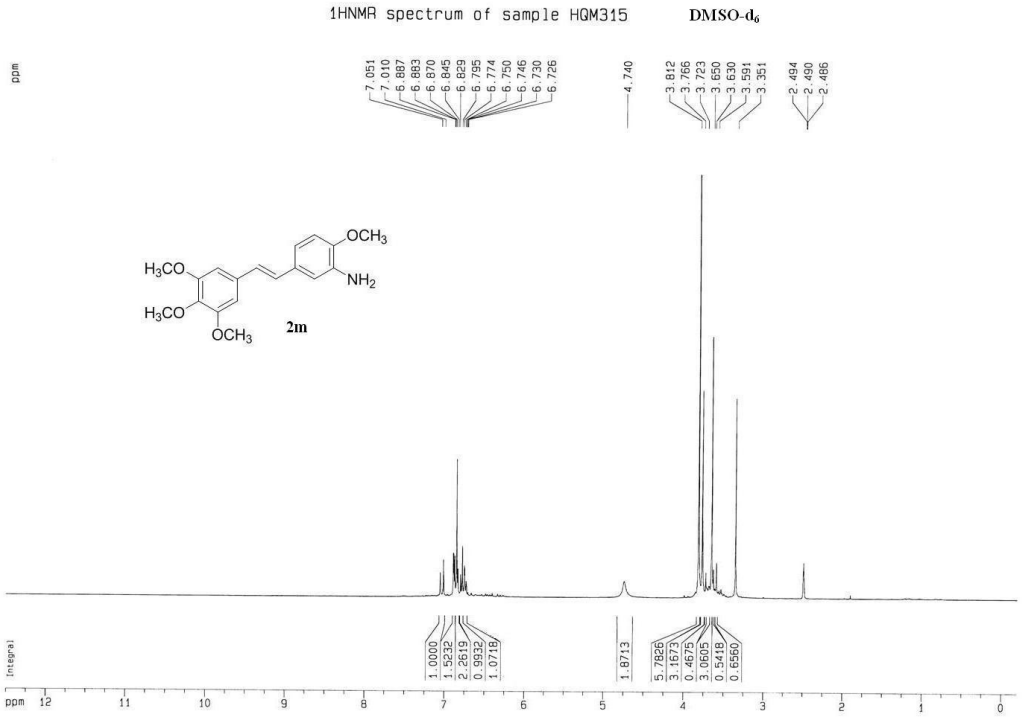
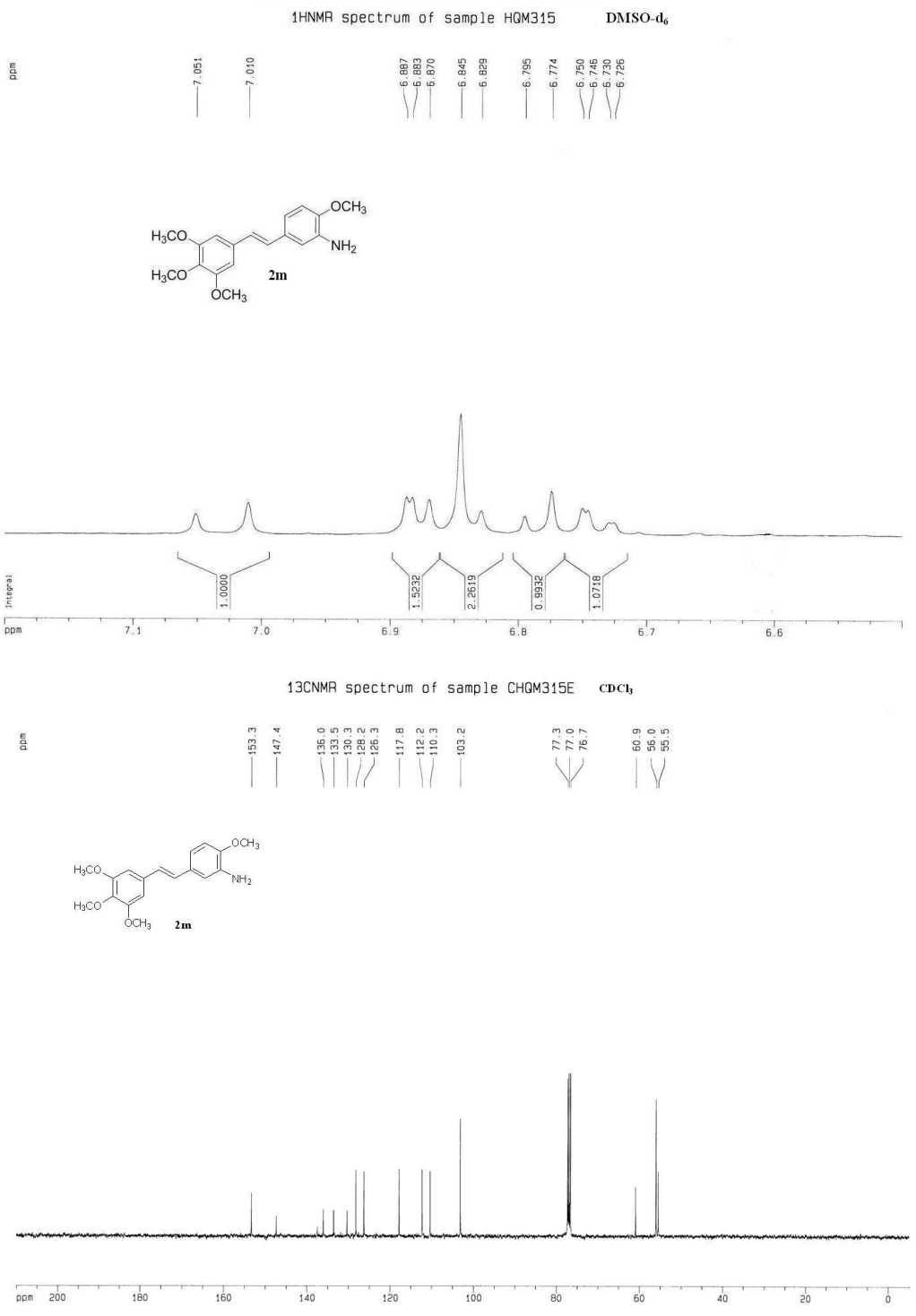

1HNMR spectrum of sample HQM300A CDCl3
ppm
7.240
7.223
7.202
6.779
6.774
6.762
6.757
6.507
6.485
6.476
6.414
6.384
H3CO
H3CO
OCH3
OCH3
2l
Integral
2.0000
2.1627
4.4454
ppm 7.50 7.25 7.00 6.75 6.50 6.25

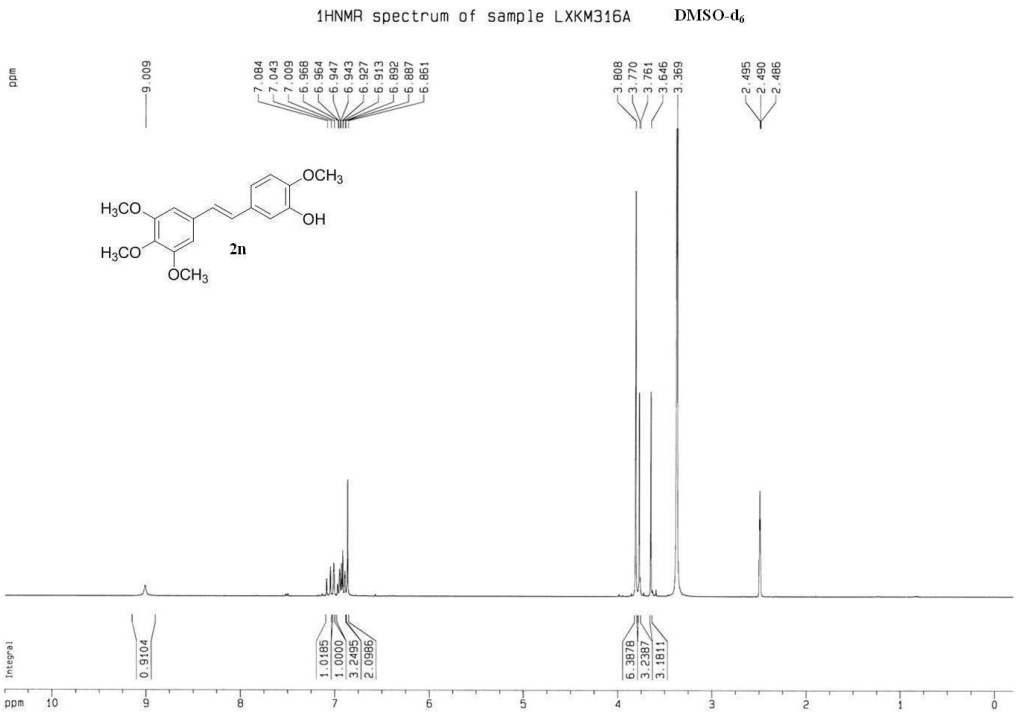


Z-3,4,4',5-tetramethoxystilbene (2l)





E-3’-animo-3,4,4’,5-tetramethoxystilbene (2m)


1HNMR spectrum of sample LXKM316A DMSO-d6
ppm
9.009
7.084
7.043
7.009
6.968
6.964
6.947
6.943
6.927
6.913
6.892
6.887
6.861
3.808
3.770
3.761
3.646
3.369
2.495
2.490
2.486
OCH3
OH
H3CO
H3CO
OCH3
2n
Integral
0.9104
1.0185
1.0000
3.2495
2.0986
6.3878
3.2387
3.1811
ppm
10
9
8
7
6
5
4
3
2
1
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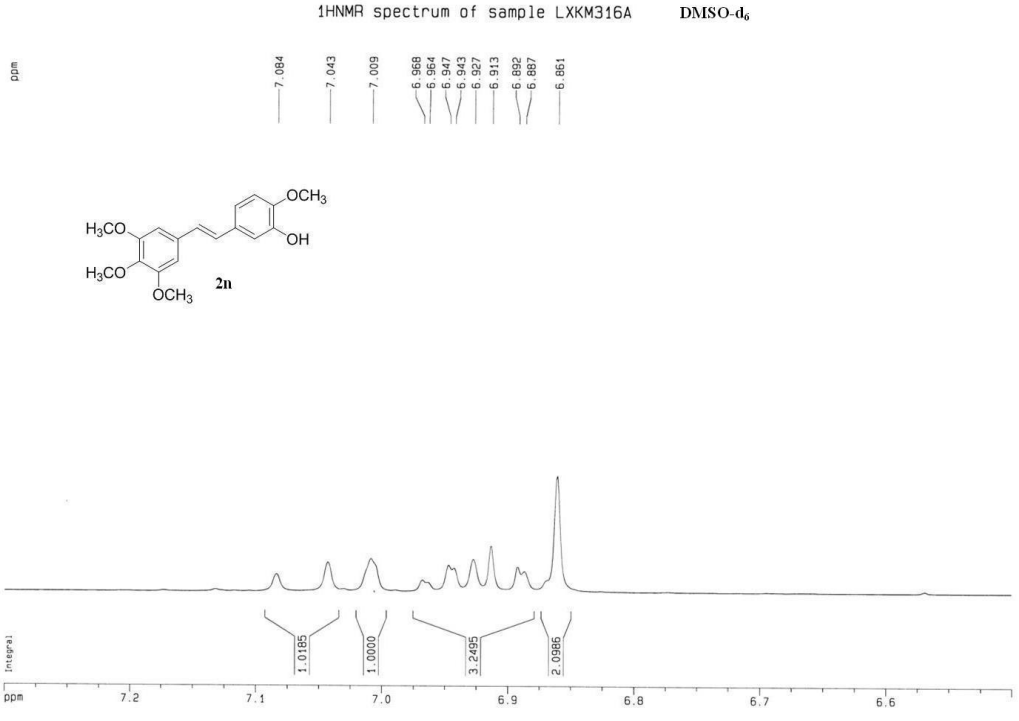
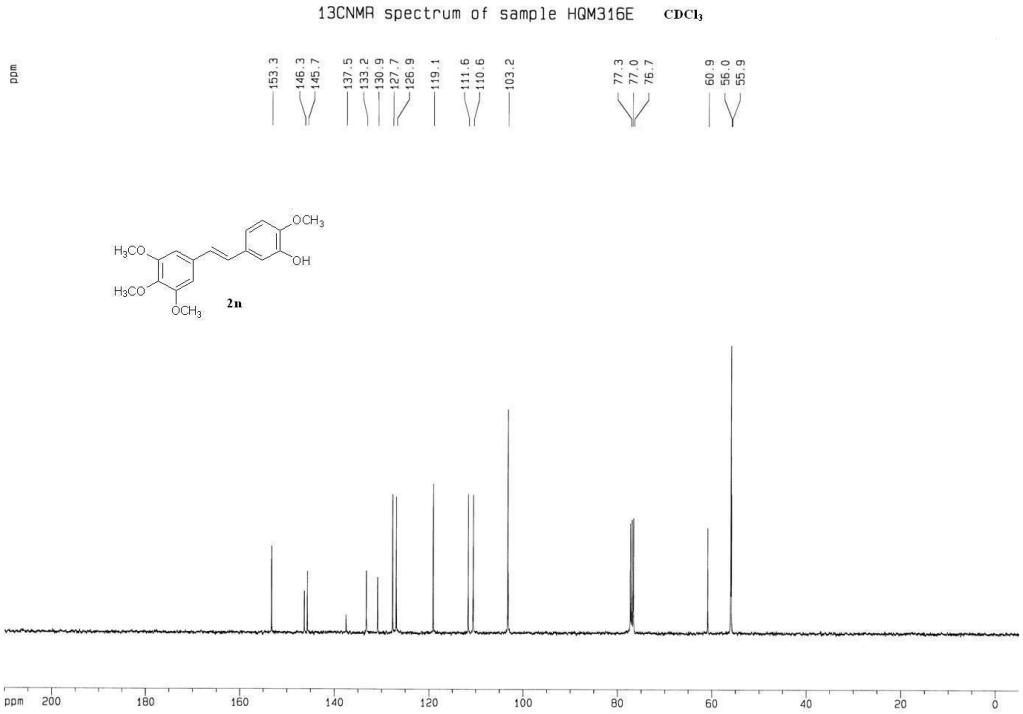
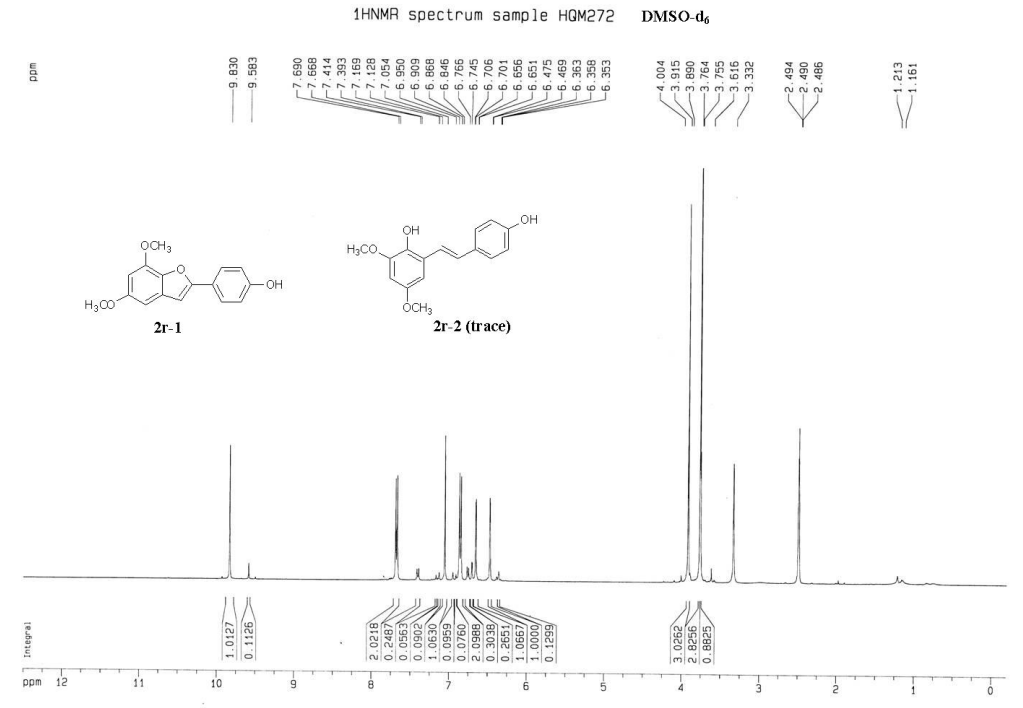
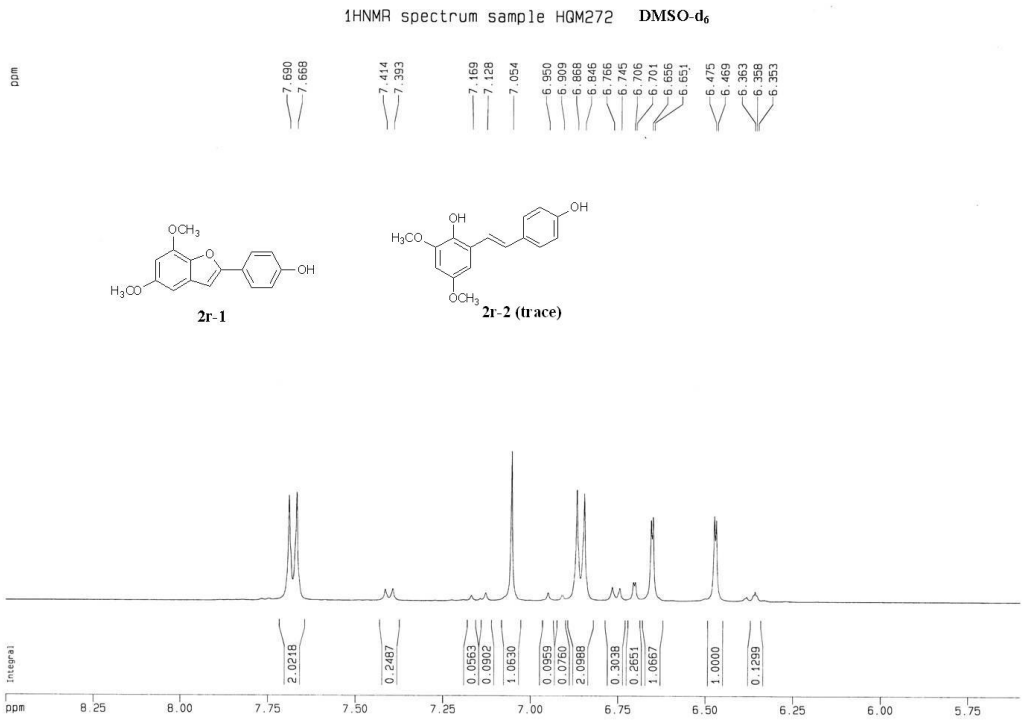
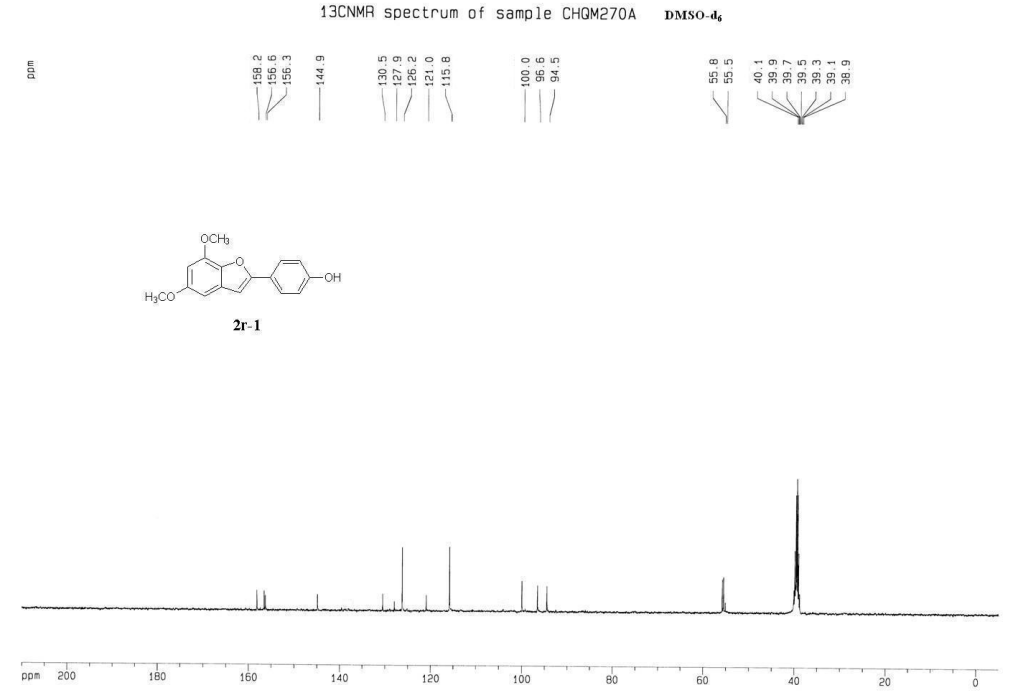

1HNMR spectrum of sample LXKM316A DMSO-d6
ppm
7.084
7.043
7.009
6.968
6.964
6.947
6.943
6.927
6.913
6.892
6.887
6.861
OCH3
OH
H3CO
H3CO
OCH3
2n
Integral
1.0185
1.0000
3.2495
2.0986
ppm
7.2
7.1
7.0
6.9
6.8
6.7
6.6



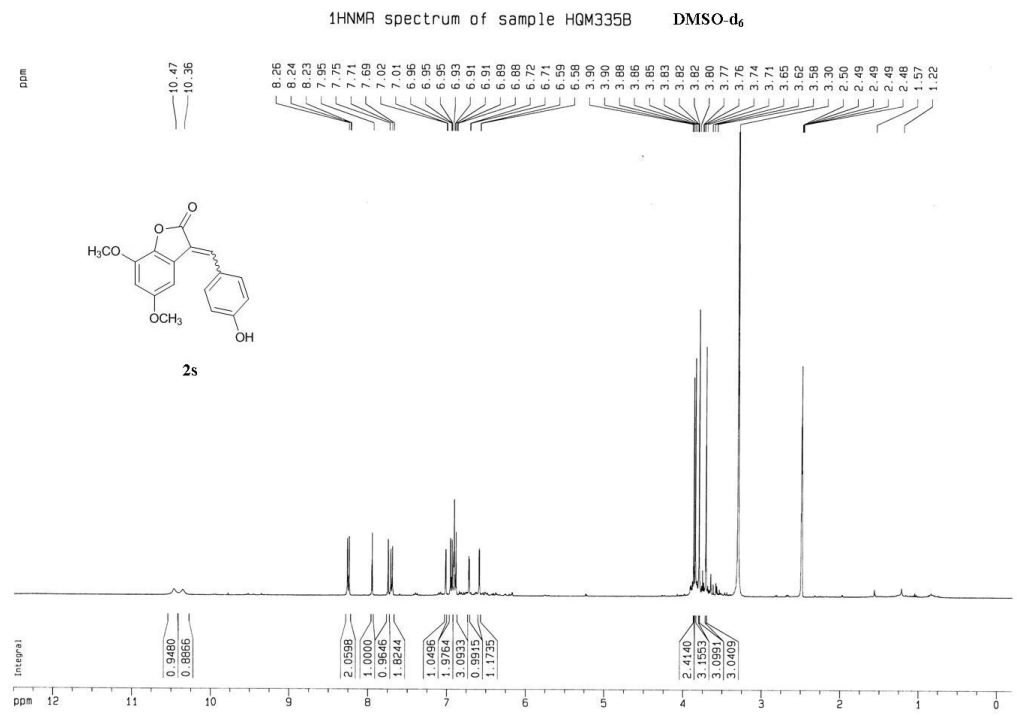
E-3'-hydroxyl-3,4,4',5-tetramethoxystilbene (2n)

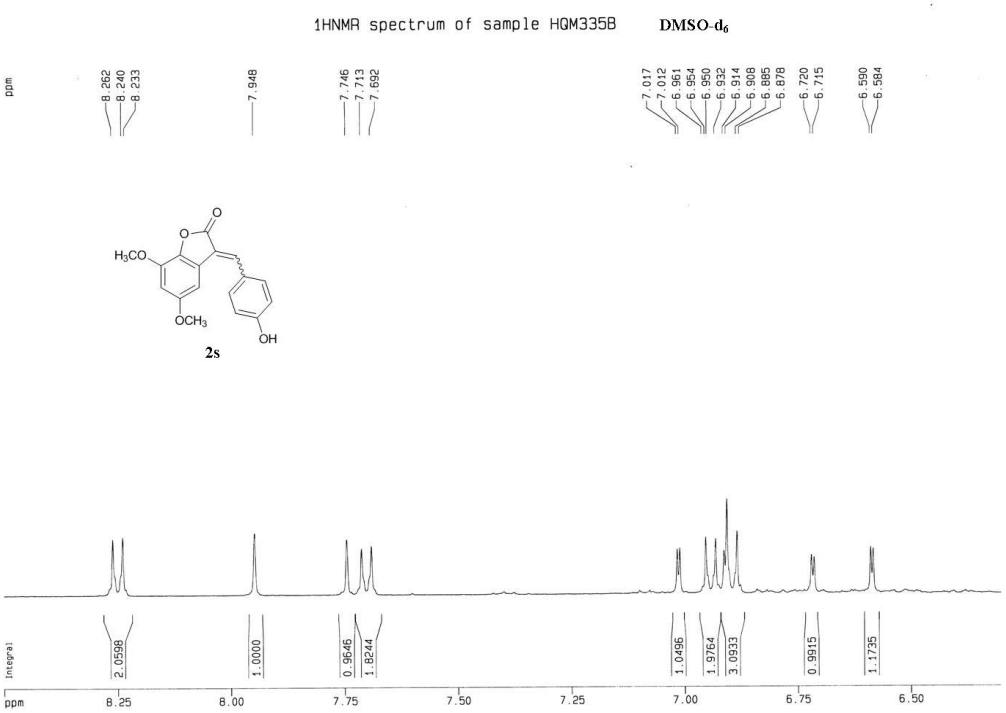






4-(5,7-dimethoxybenzofuran-2-yl)phenol (2r-1)





3-(4-hydroxybenzylidene)-5,7-dimethoxybenzofuran-2(3H)-one (2s)