Supplementary Materials:

Recognition of acids involve in Krebs cycle by 9-Anthrylmethyl-di(6-acetylamino-2-picolyl)amine: a case of selective fluorescence enhancement for maleic acid

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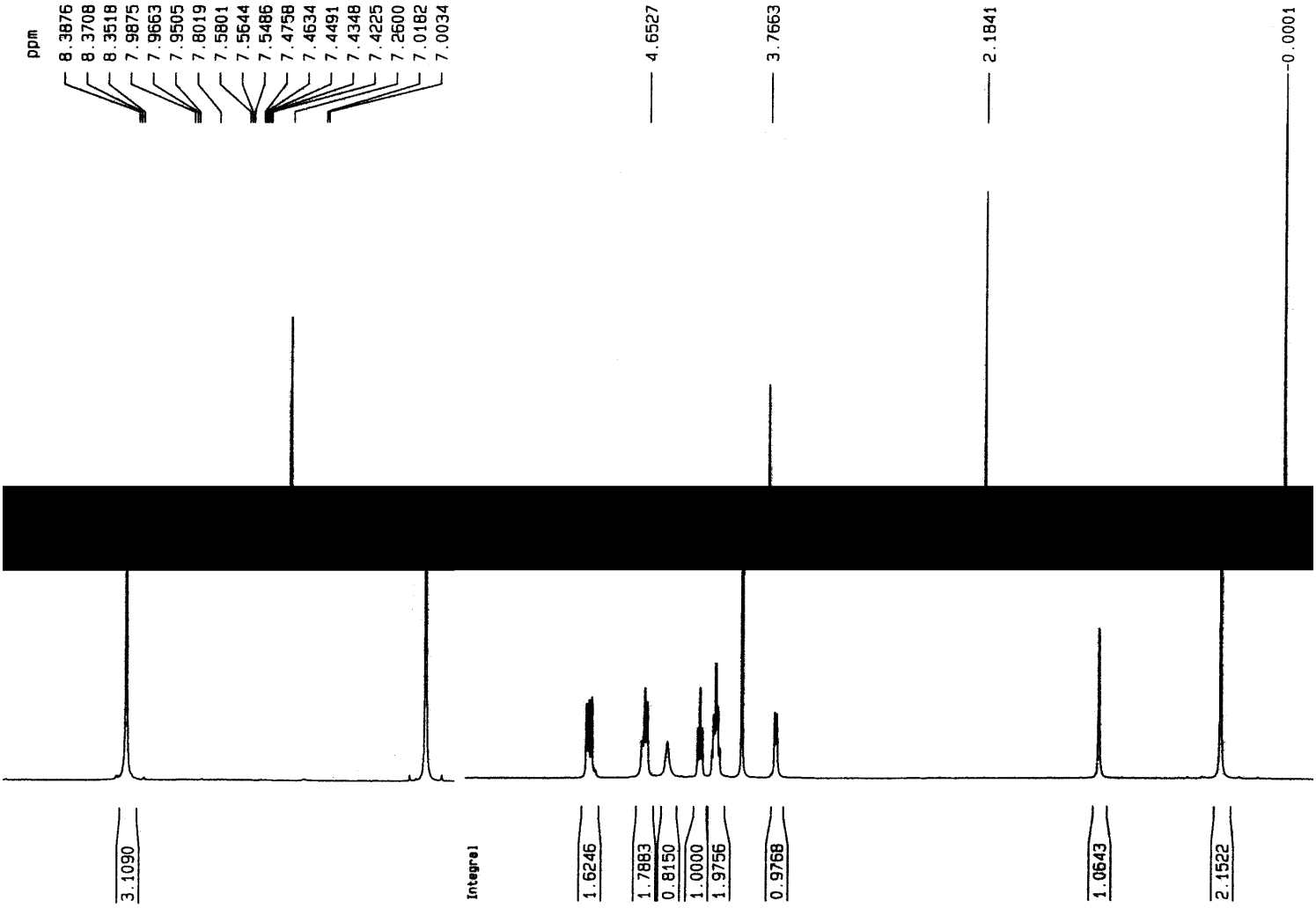
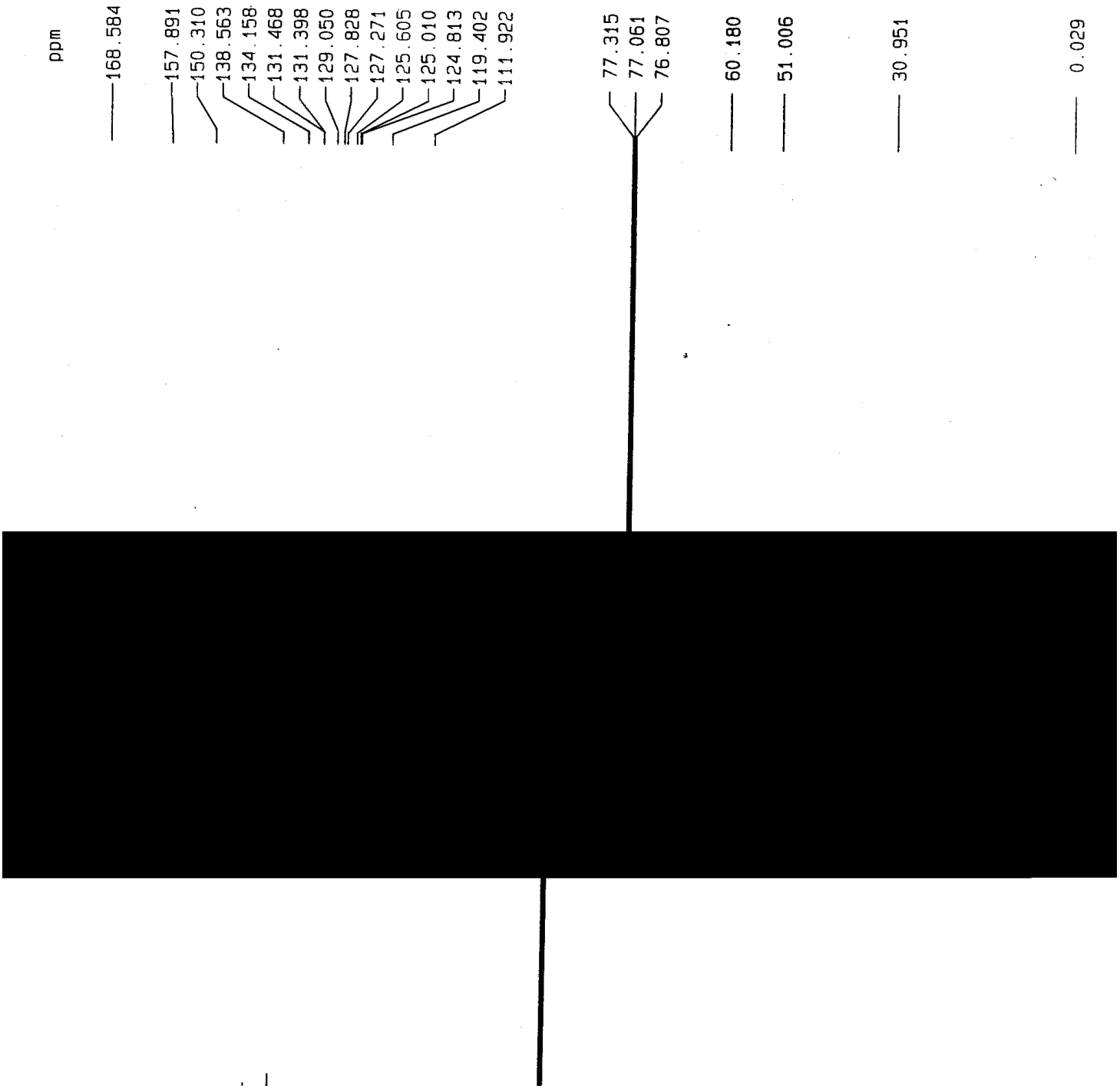
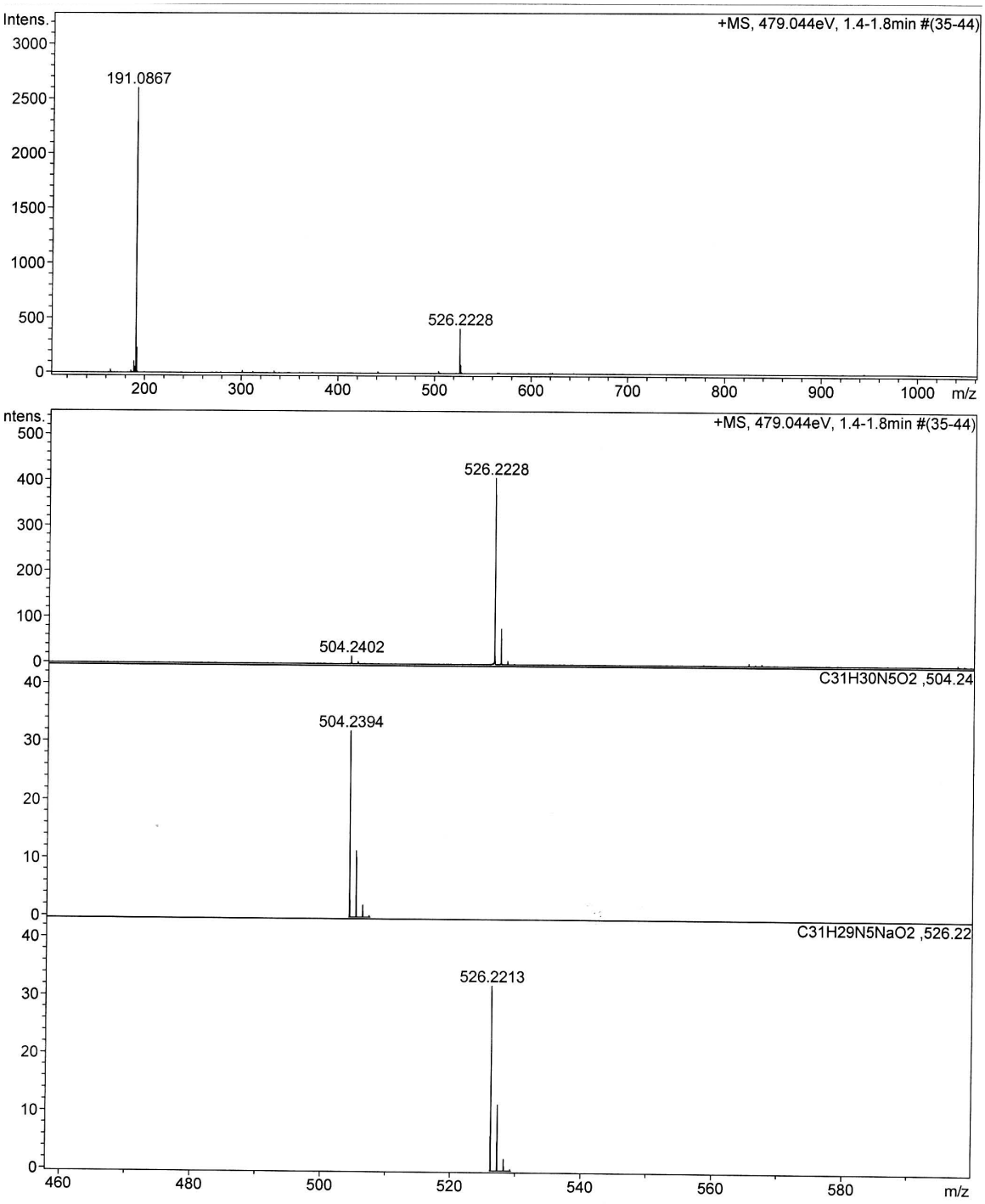
^{1}H NMR of **1**:



^{13}C NMR of **1:**



HRMS of **1**:



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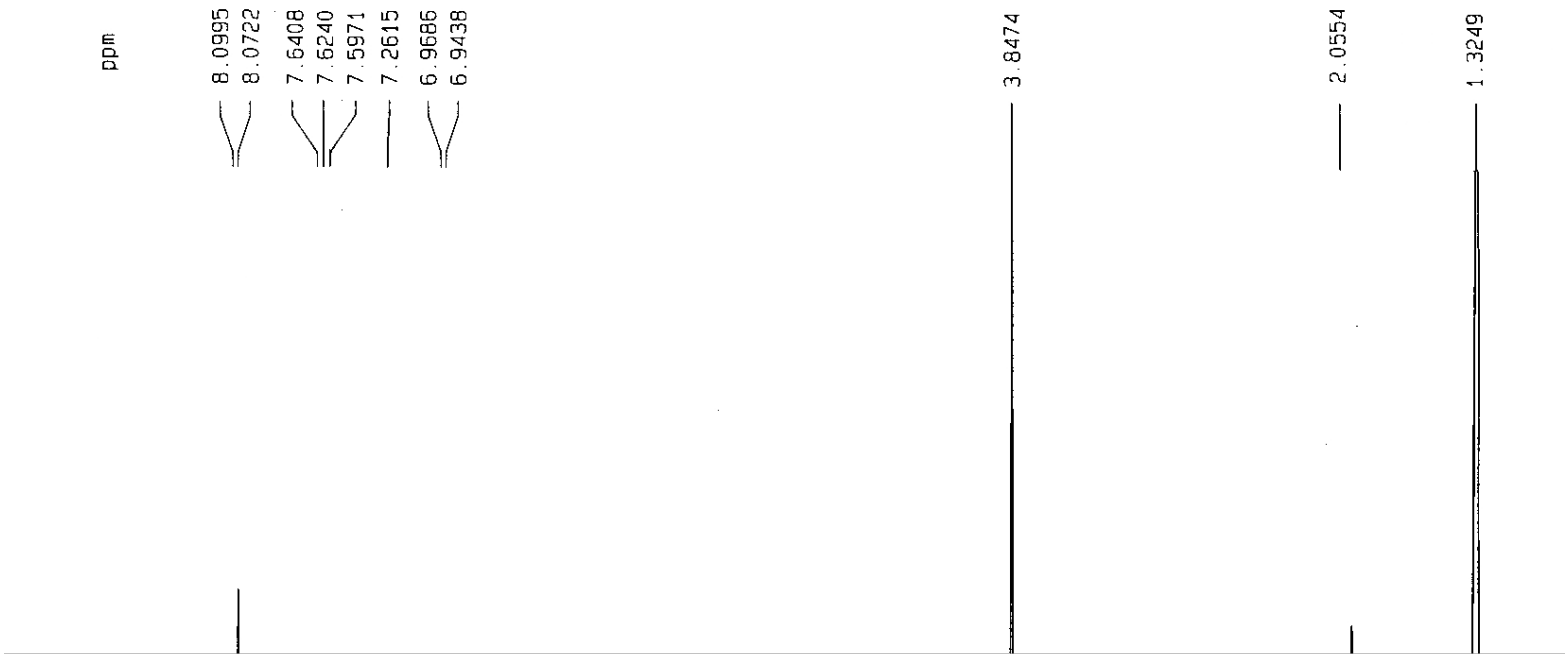
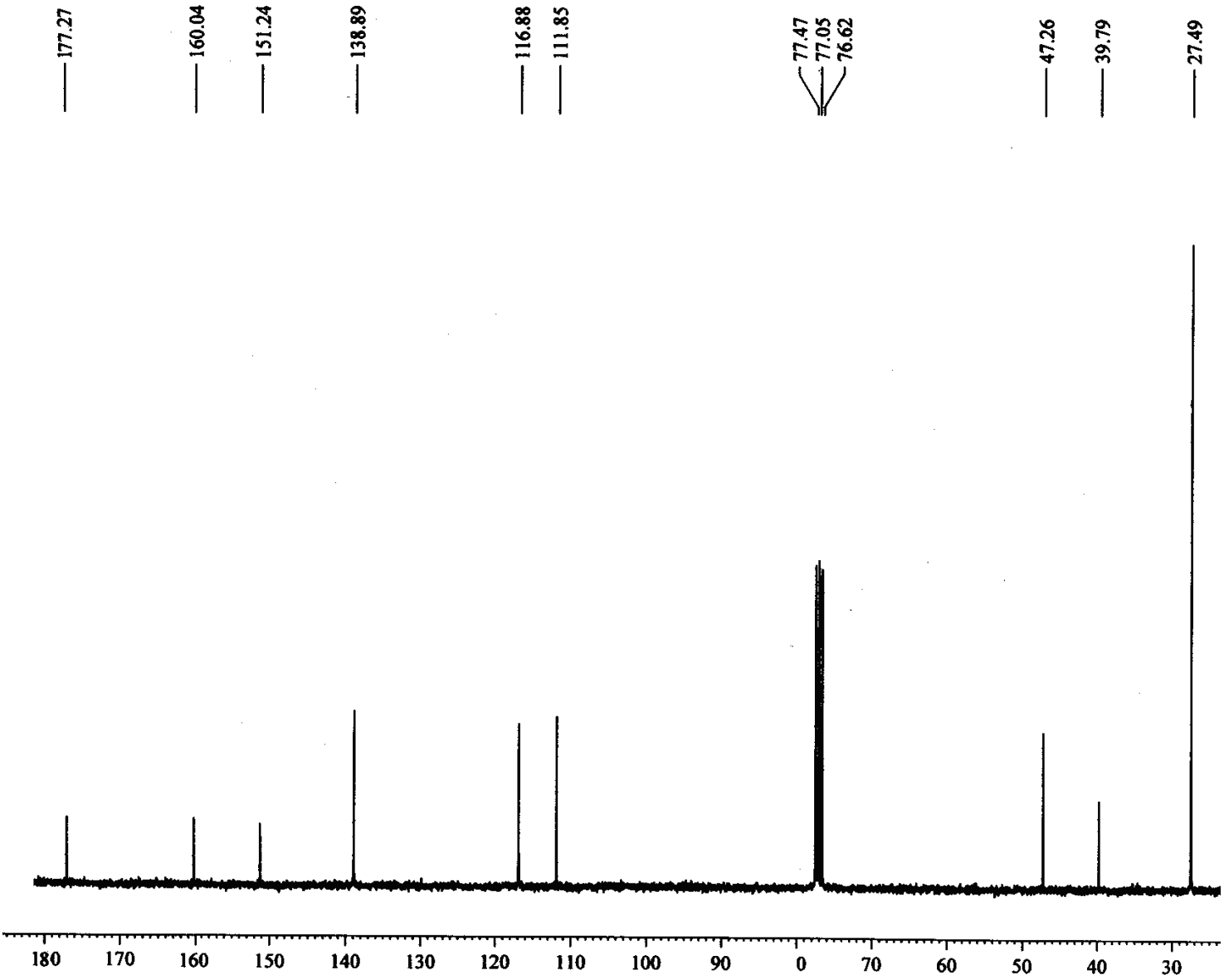
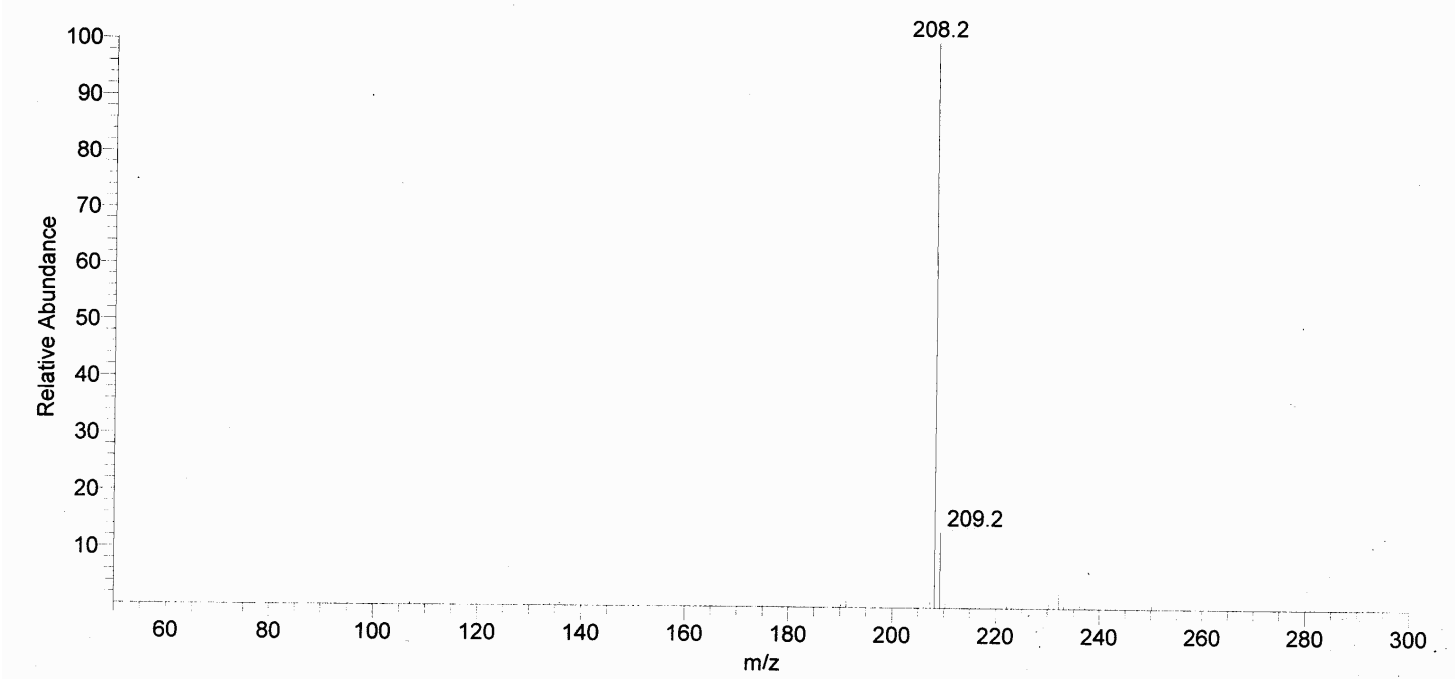


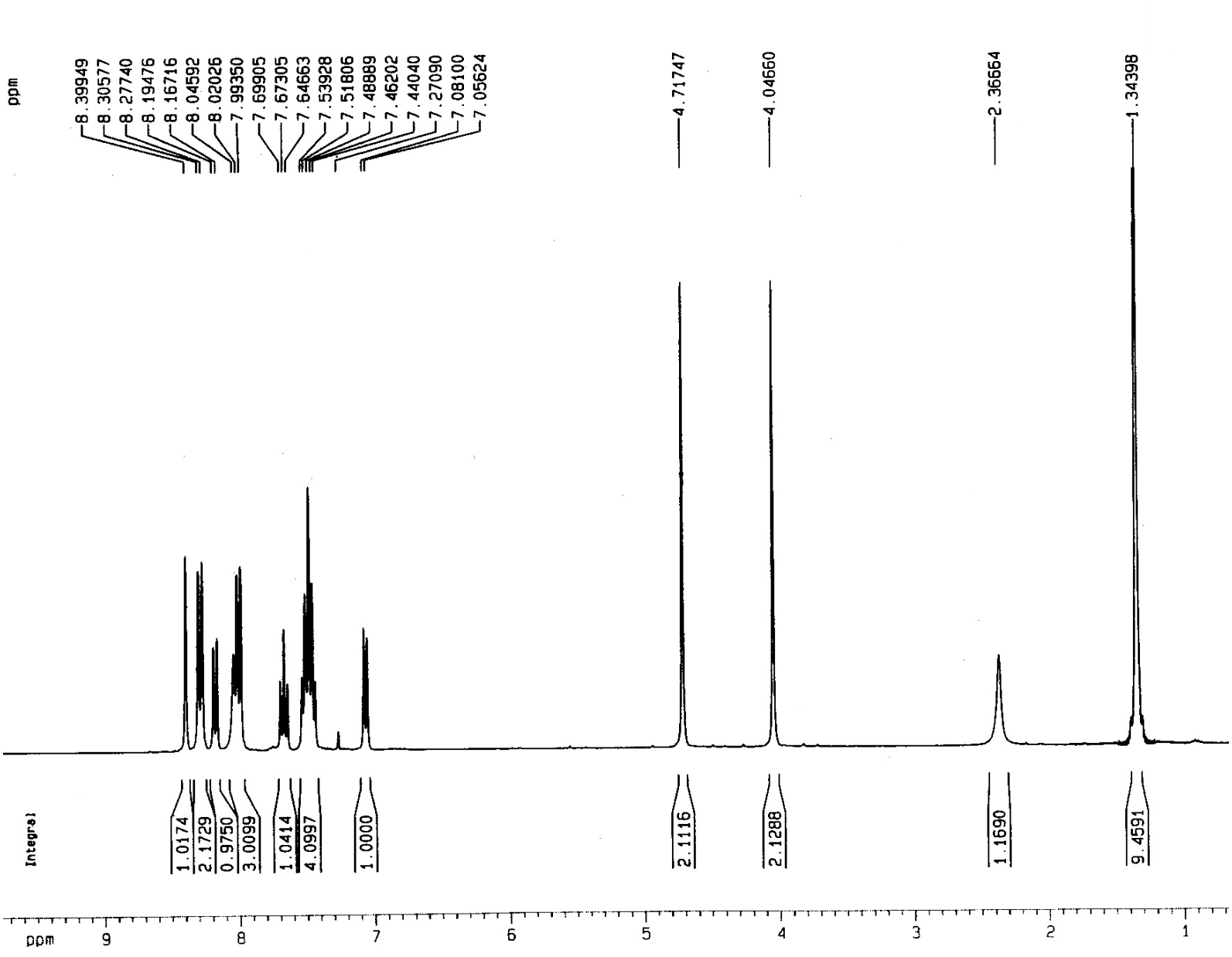
^{13}C-NMR of **4**:



ESI-MS of compound **4**:



^{1}H NMR or compound **5:**



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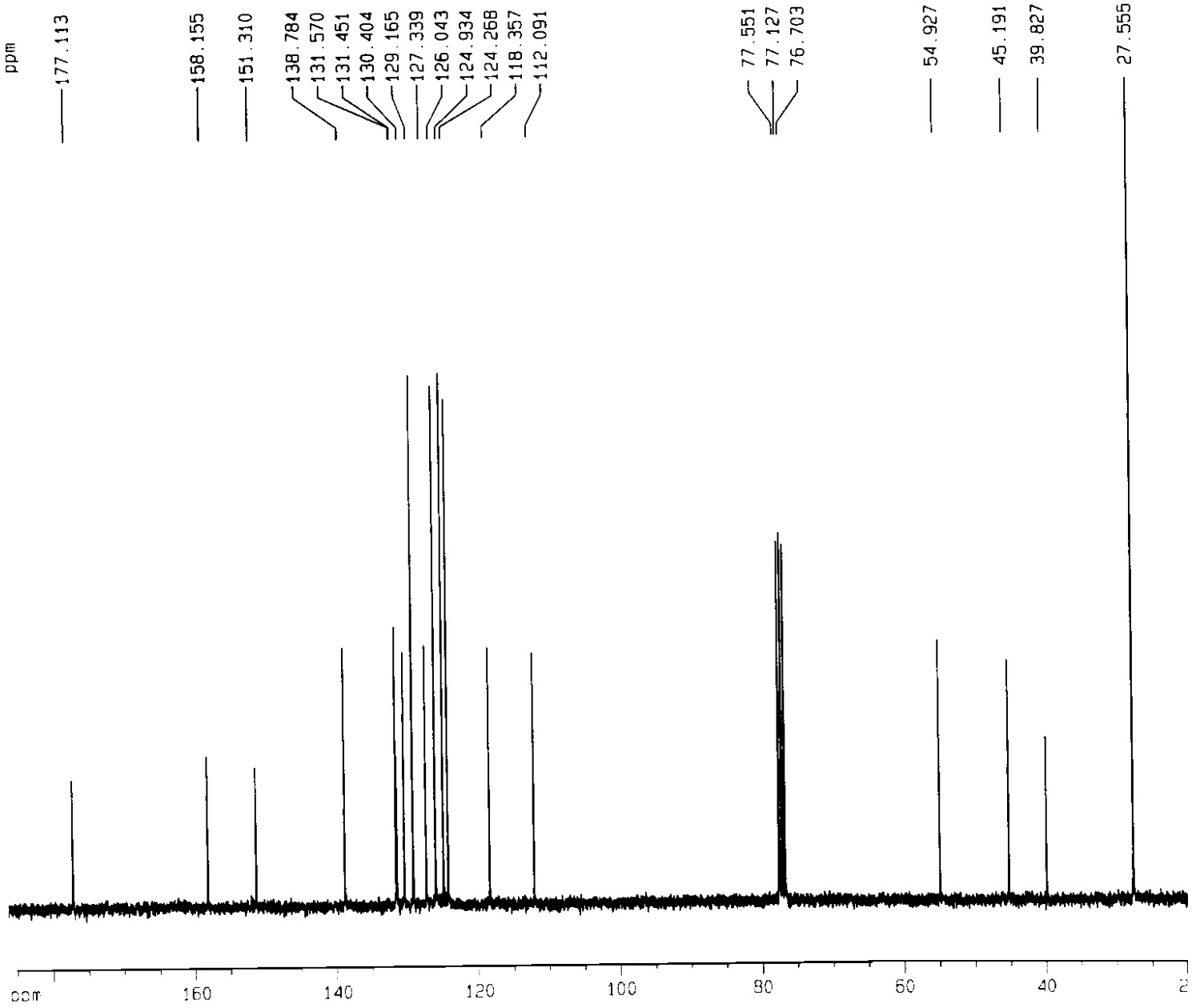
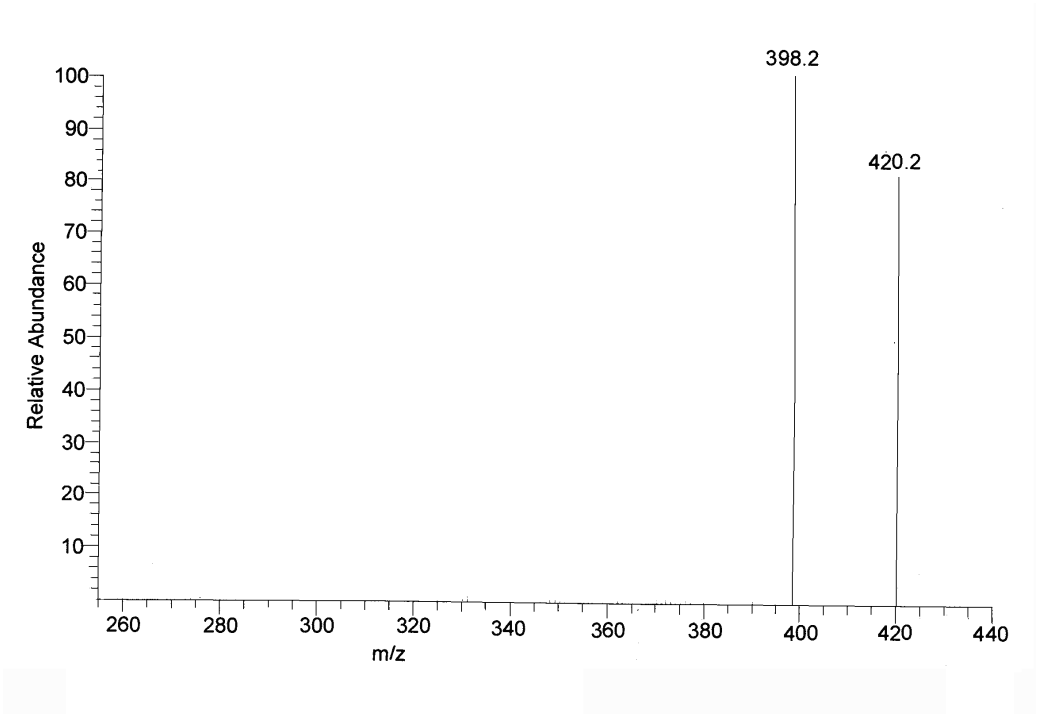


ESI-MS of compound **5**:



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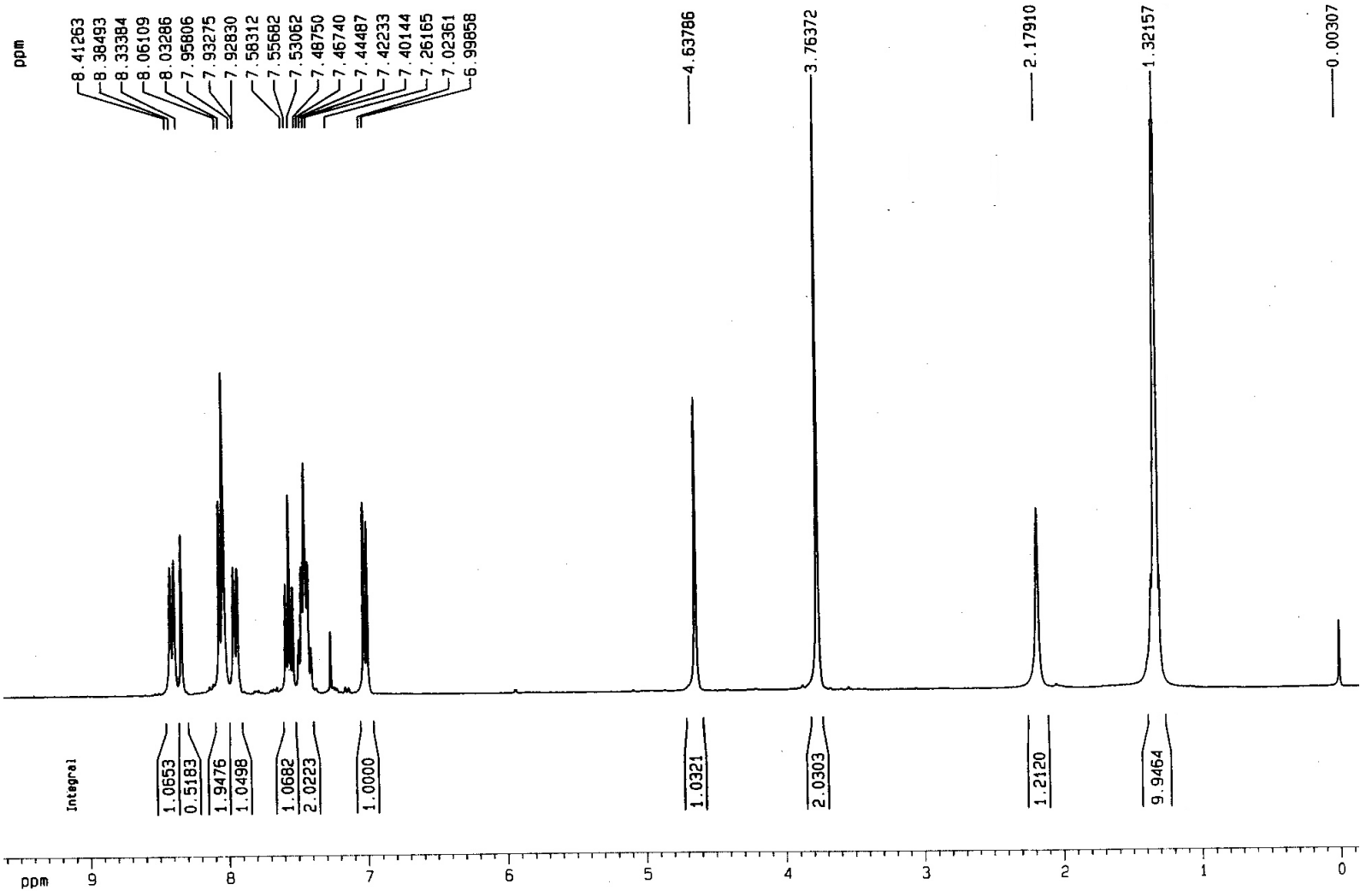
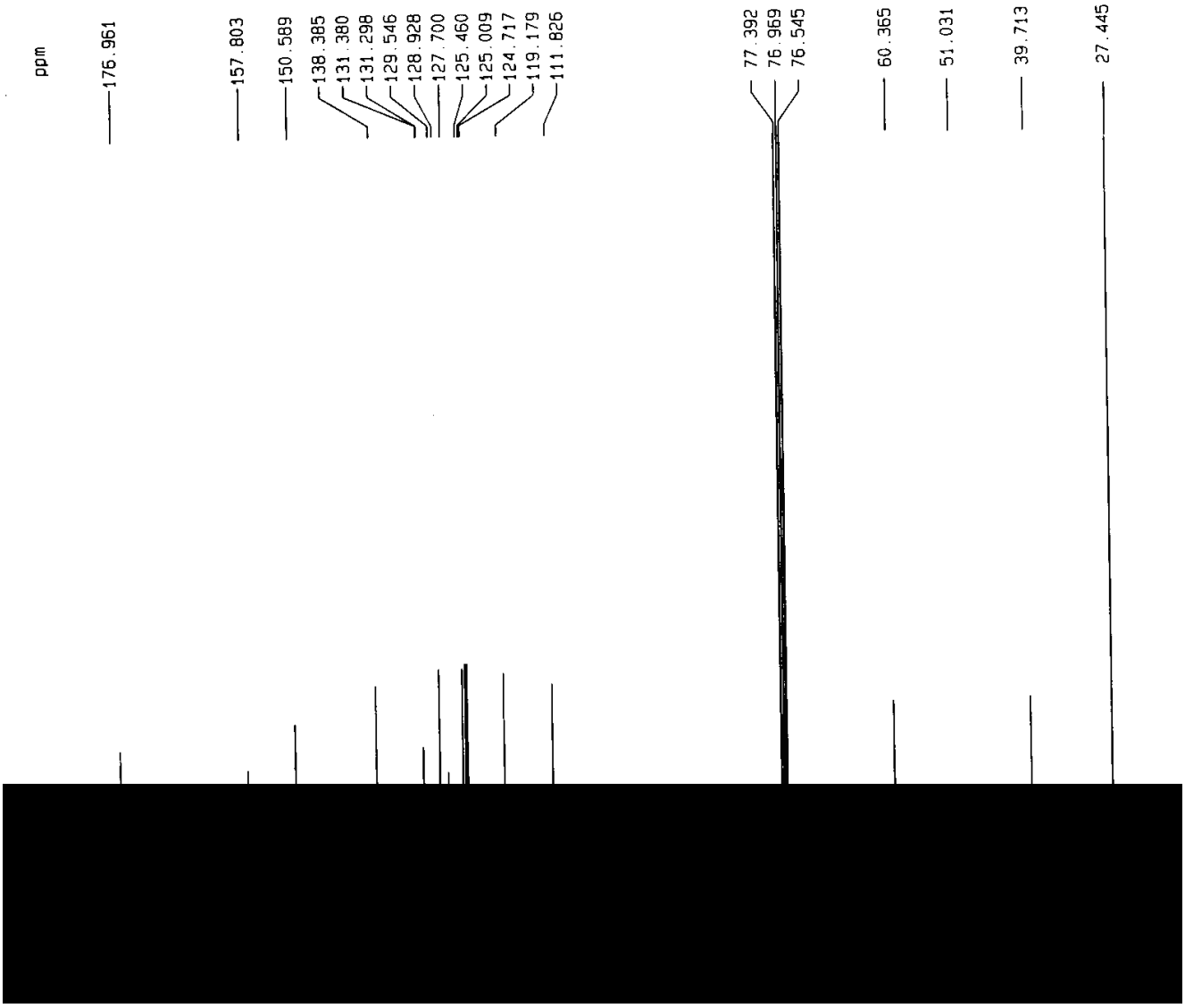
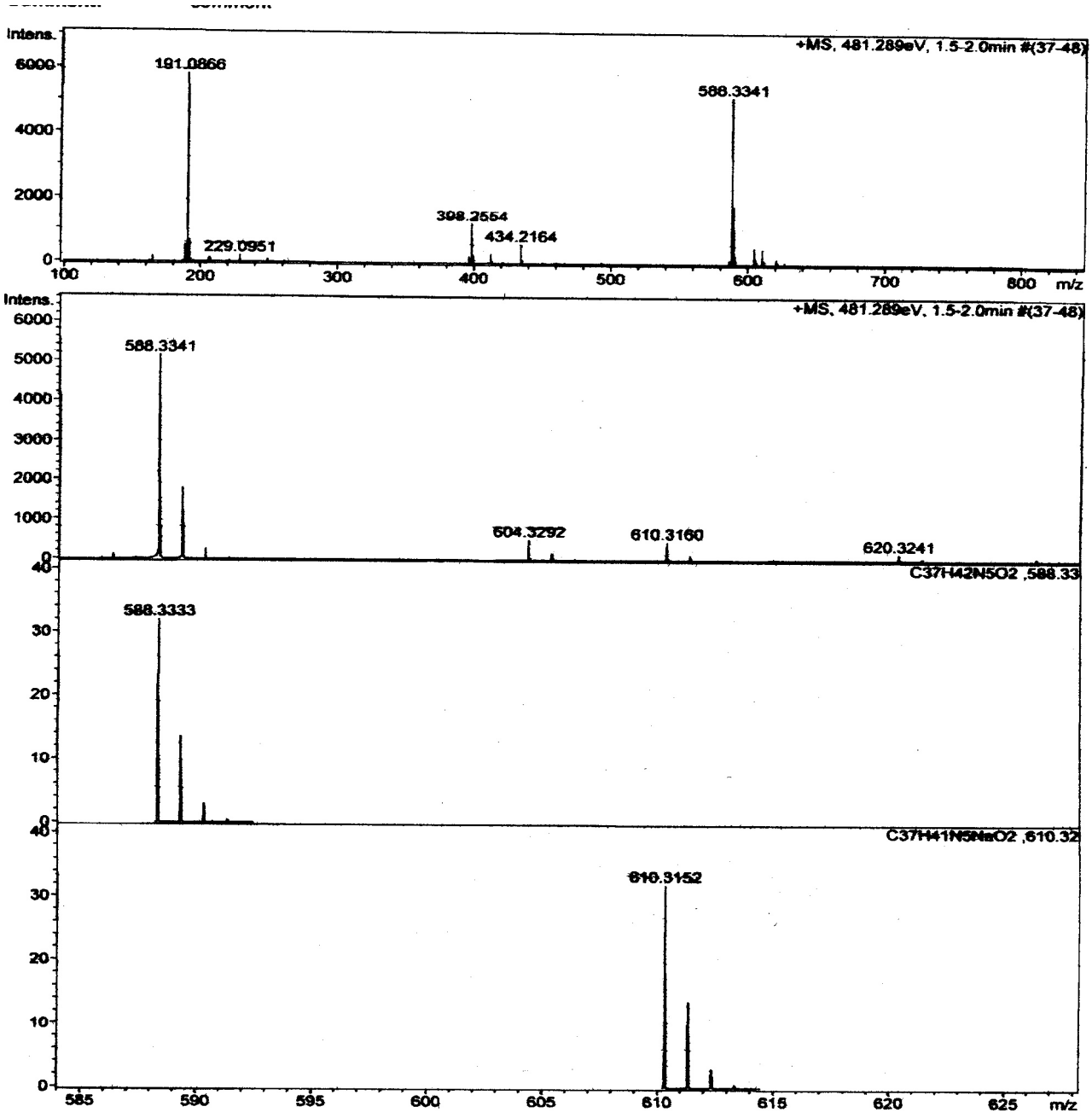


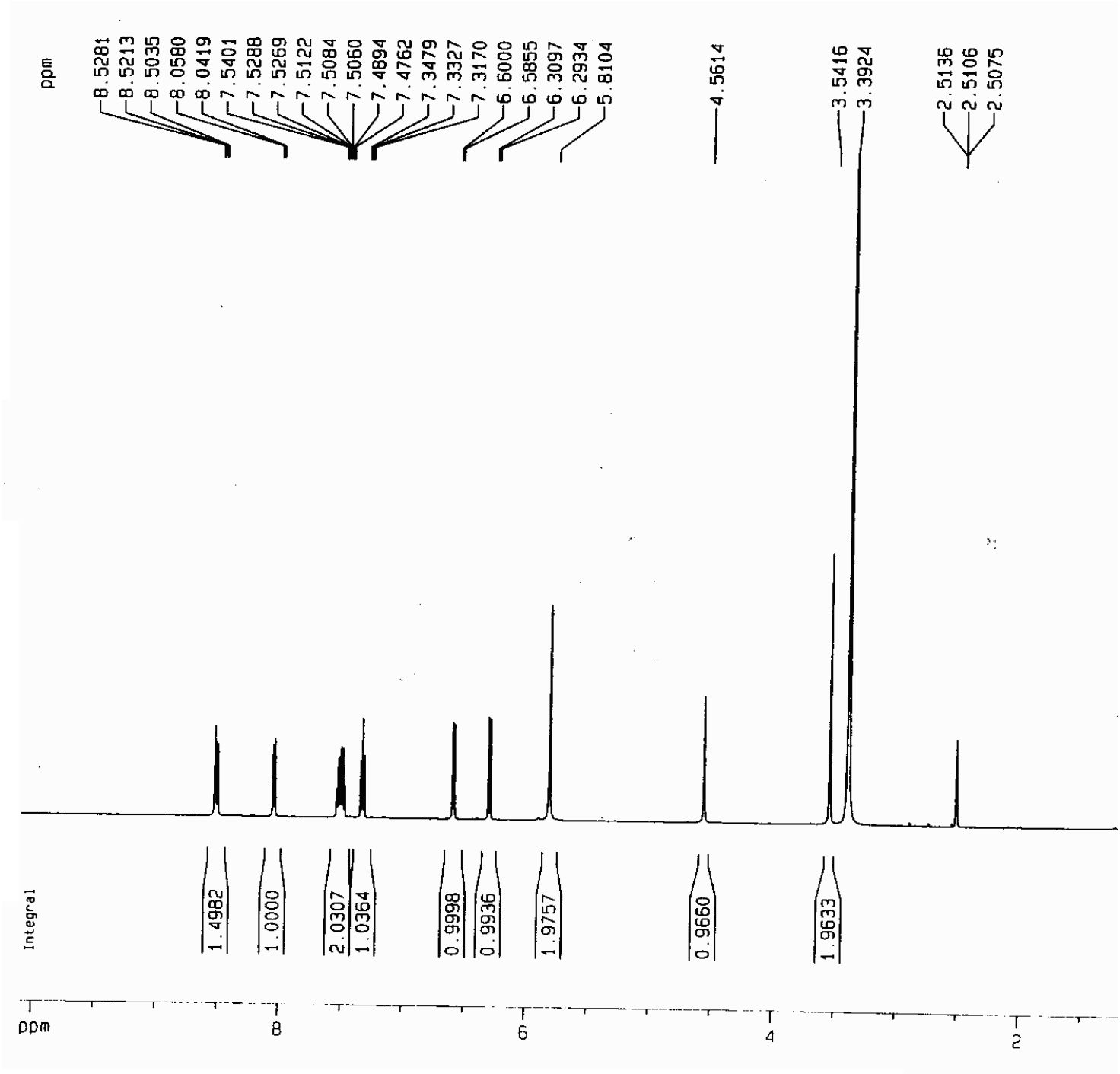
^{13}C NMR or compound **6**:



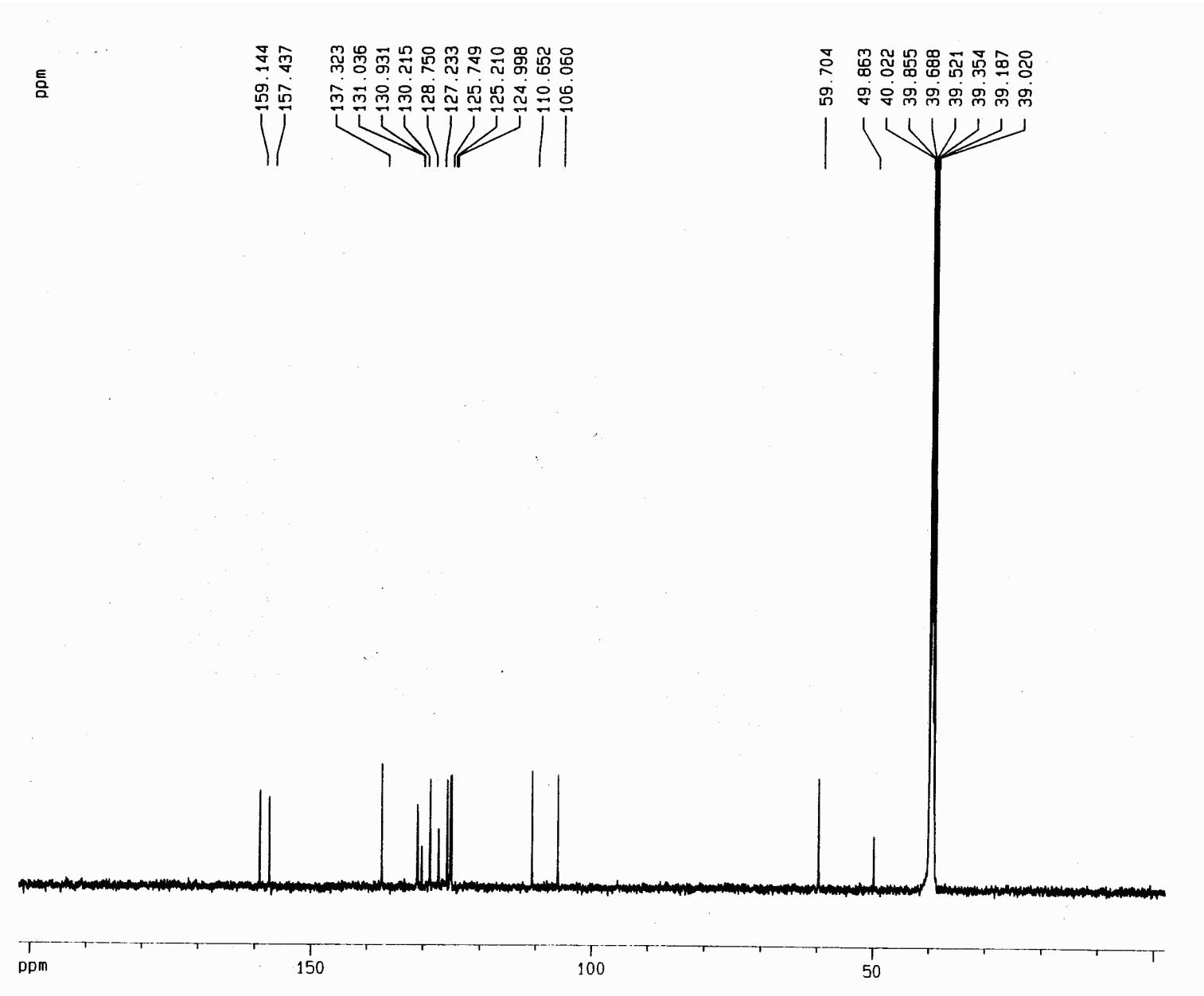
HRMS of compound **6**:



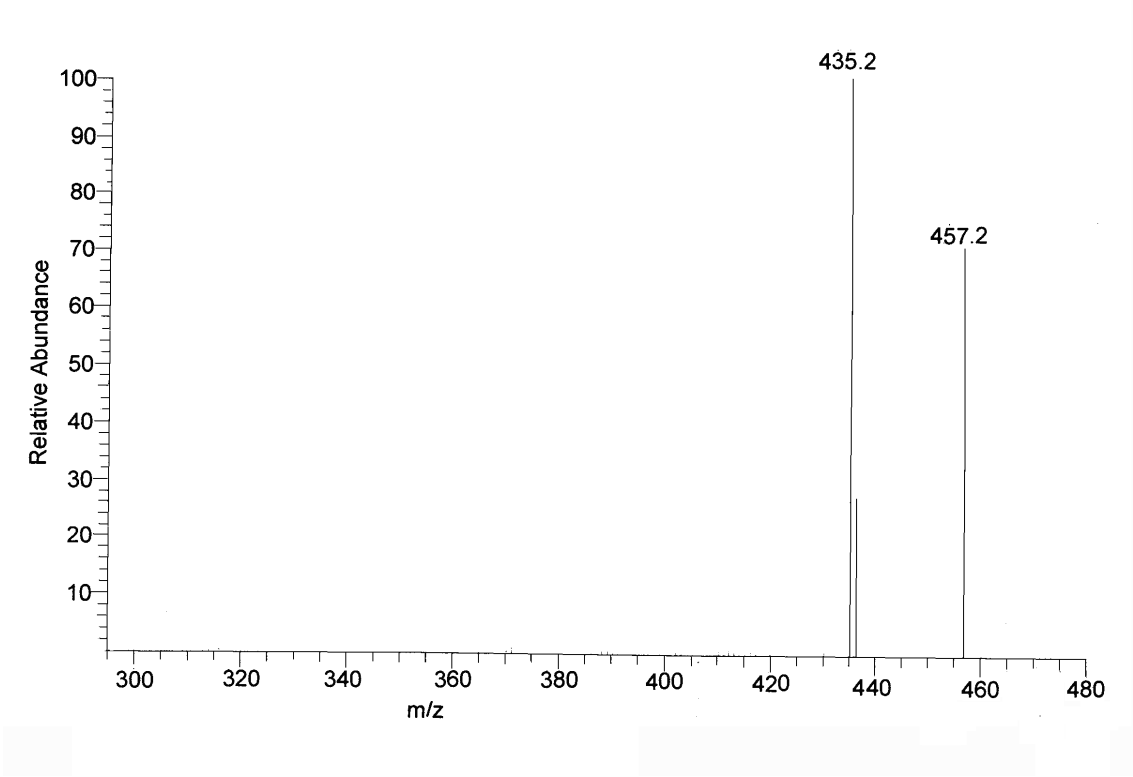
^{1}H-NMR of Compound **7**:



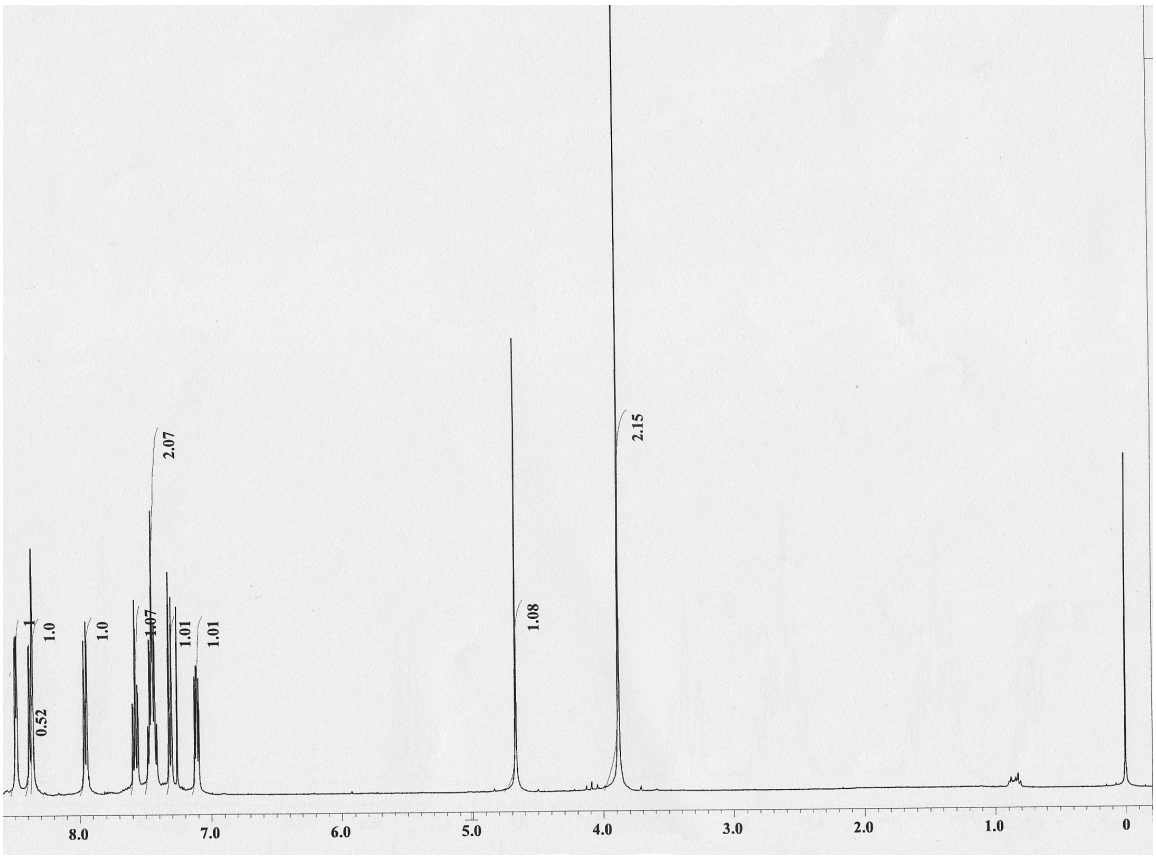
^{13}C-NMR of Compound **7**:



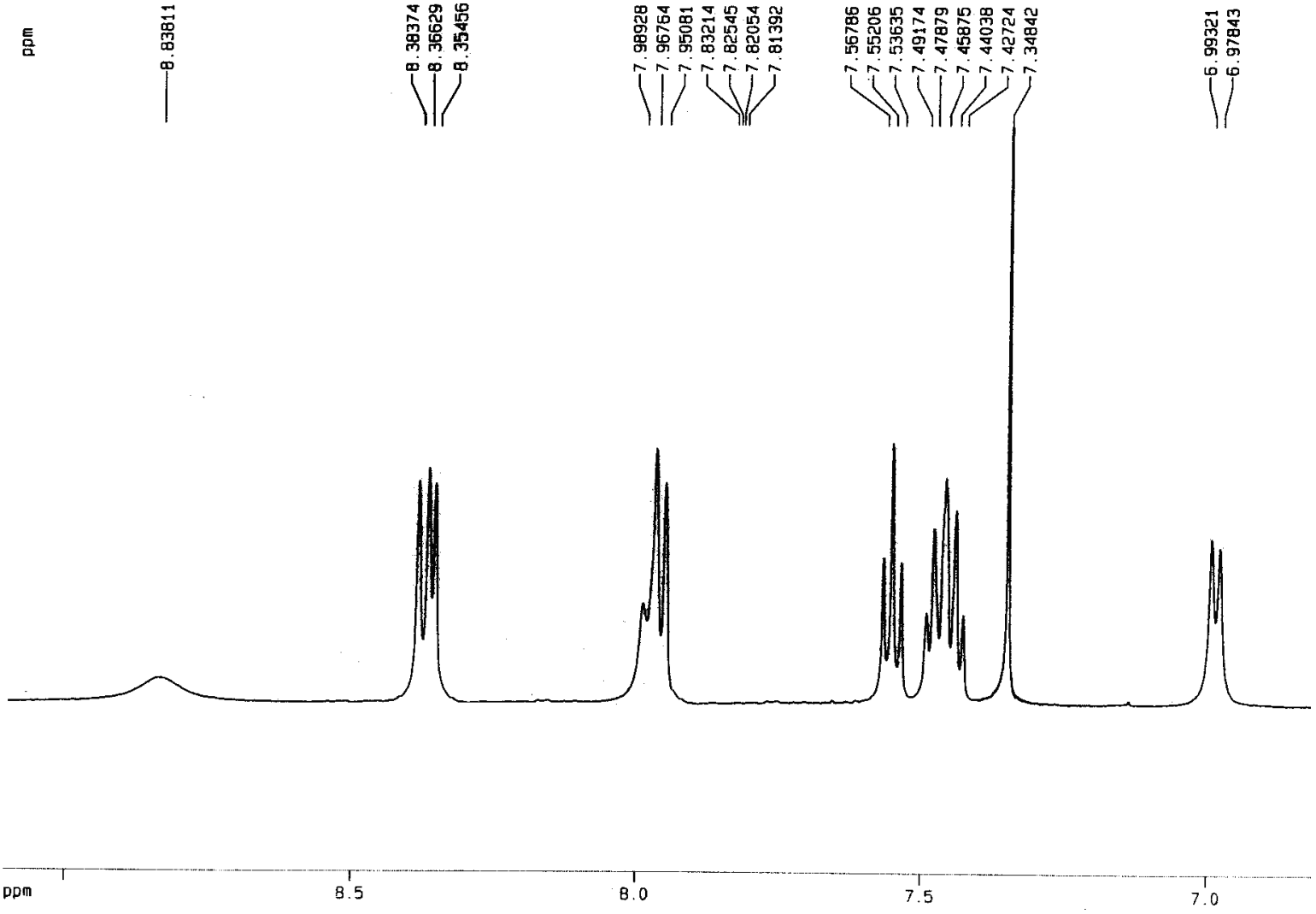
ESI-MS of compound **7**:



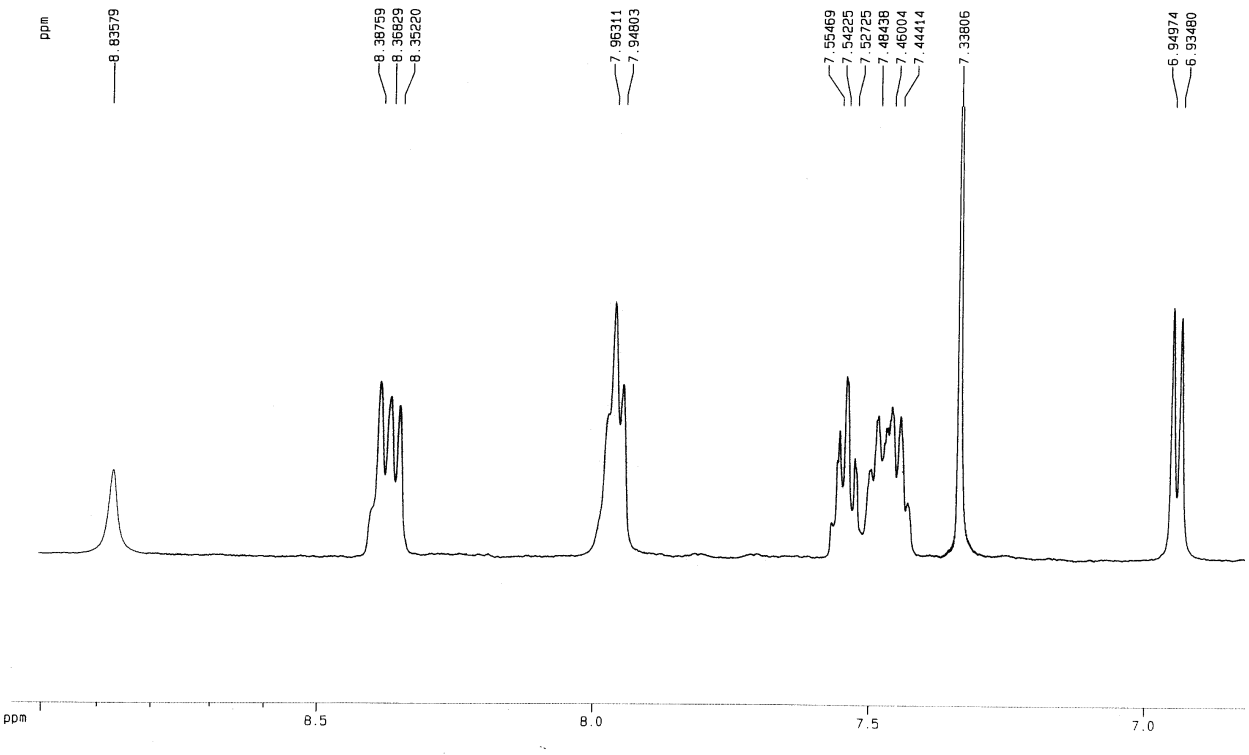
^{1}H-NMR of **2**:



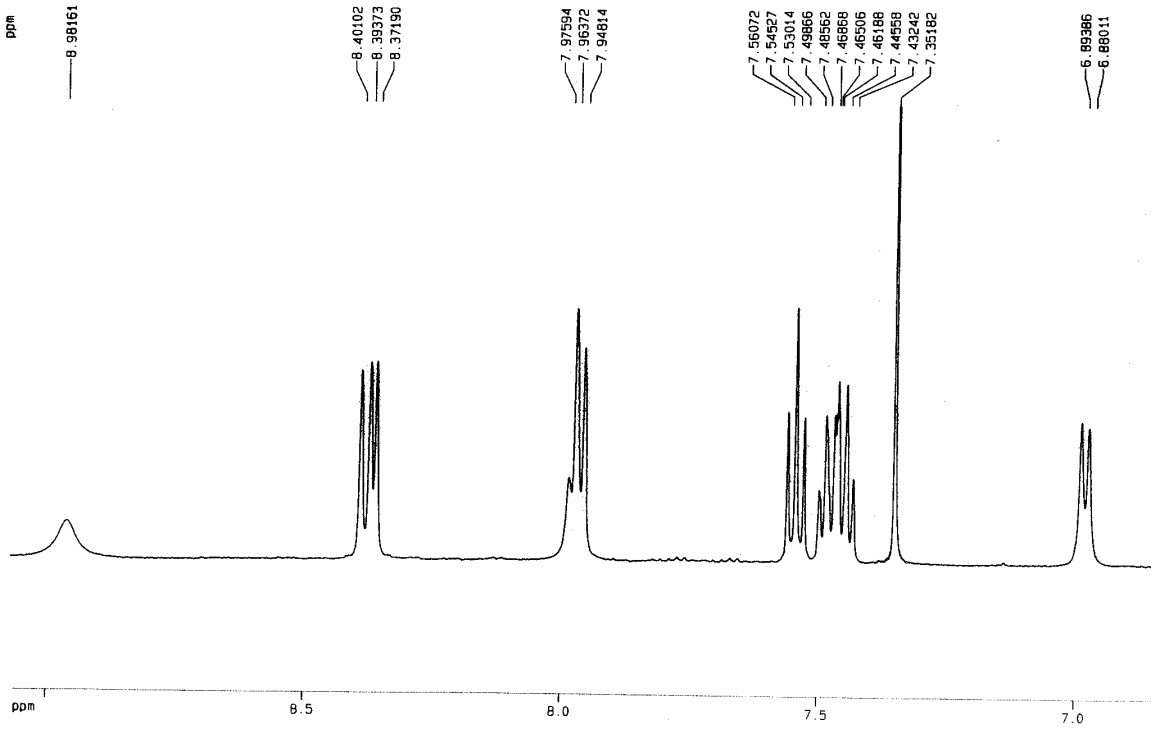
Partial ^{1}H NMR with succinic acid and **1**:



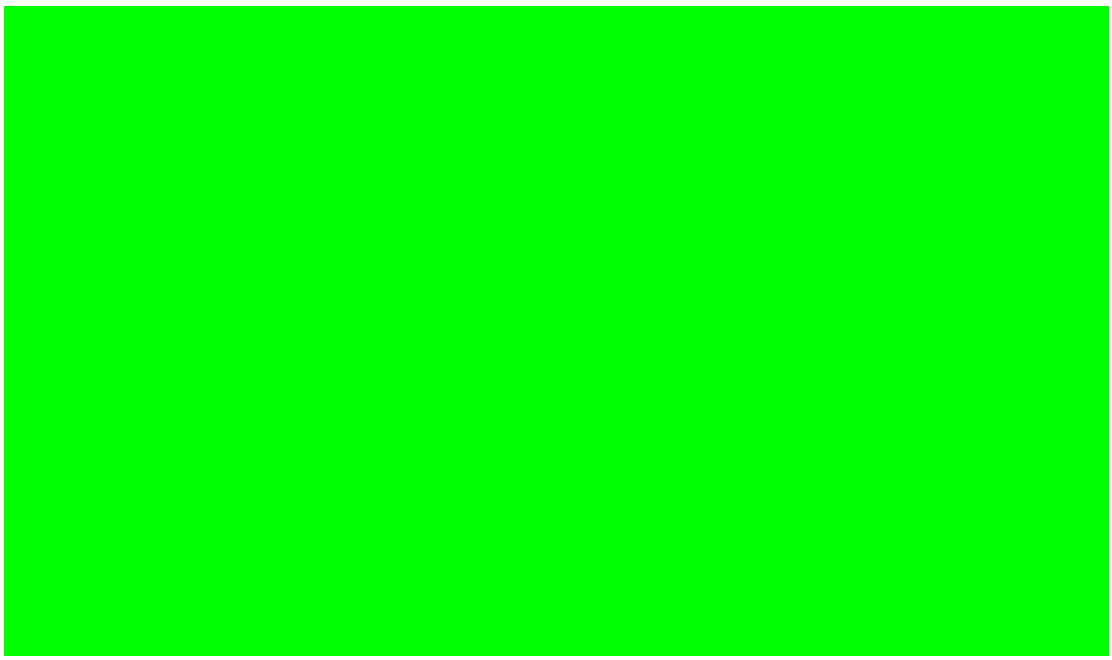
Partial ^{1}H NMR with *dl*-malic acid and **1**:



Partial ^{1}H NMR of **1** with fumaric acid:



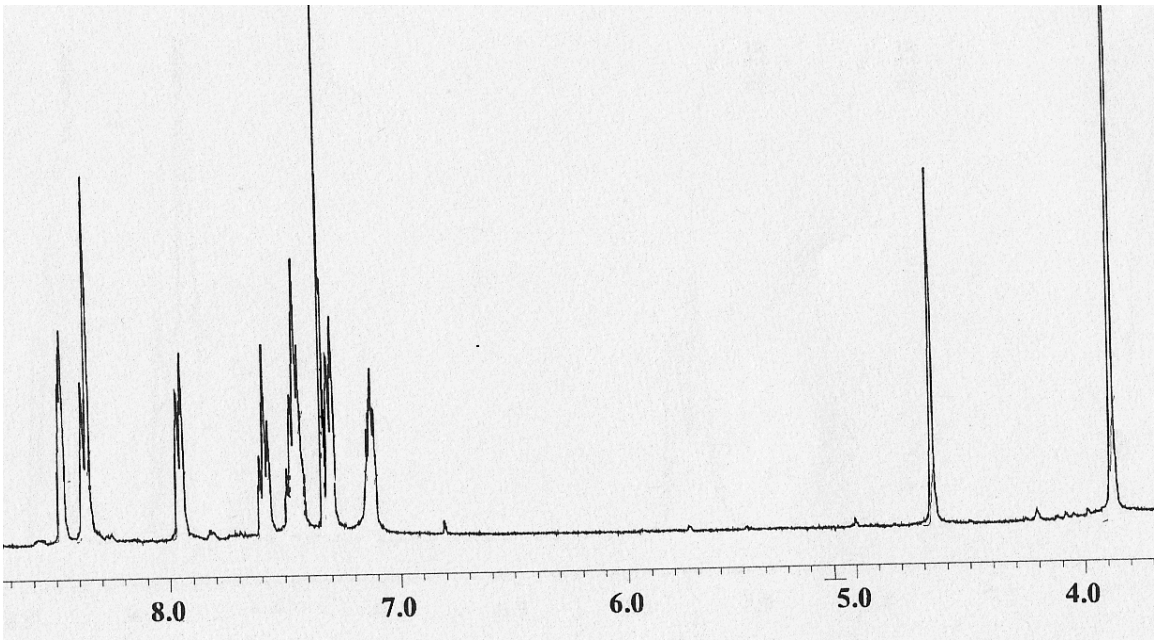
Partial 1H NMR with citric acid



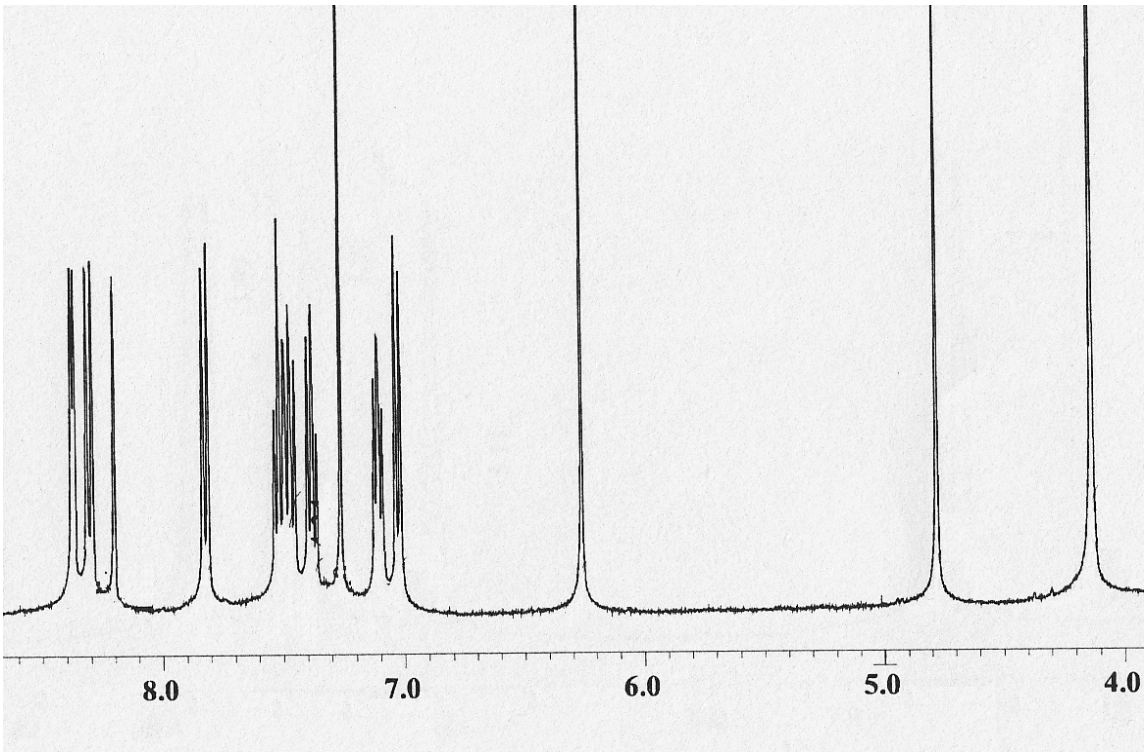
Partial ^{1}H NMR of **1** with TFA (1 equivalent):

Partial ^{1}H NMR of **1** with TFA (3 equivalent):

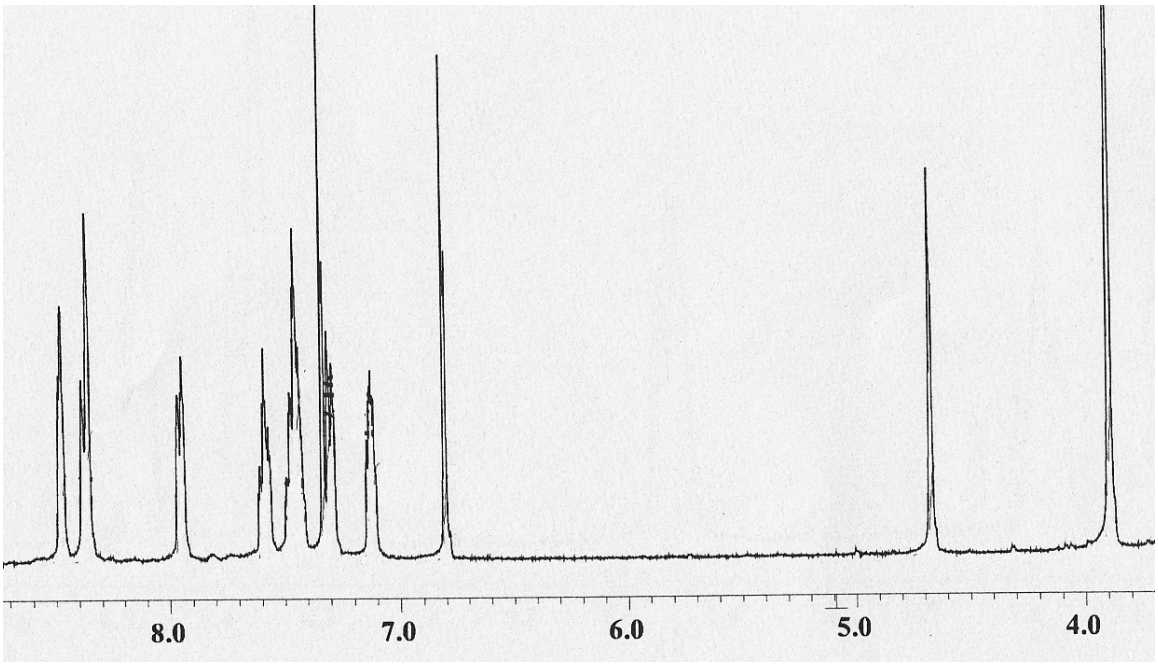
Partial ^{1}H-NMR of **2** with succinic acid:



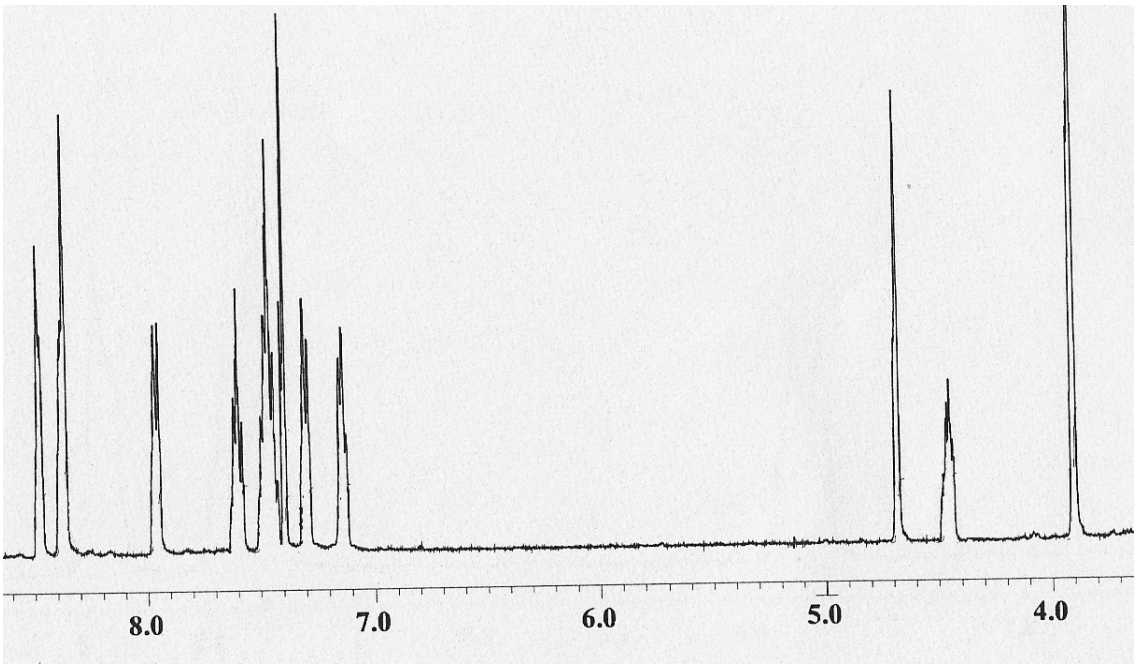
Partial ^{1}H-NMR of **2** with maleic acid:



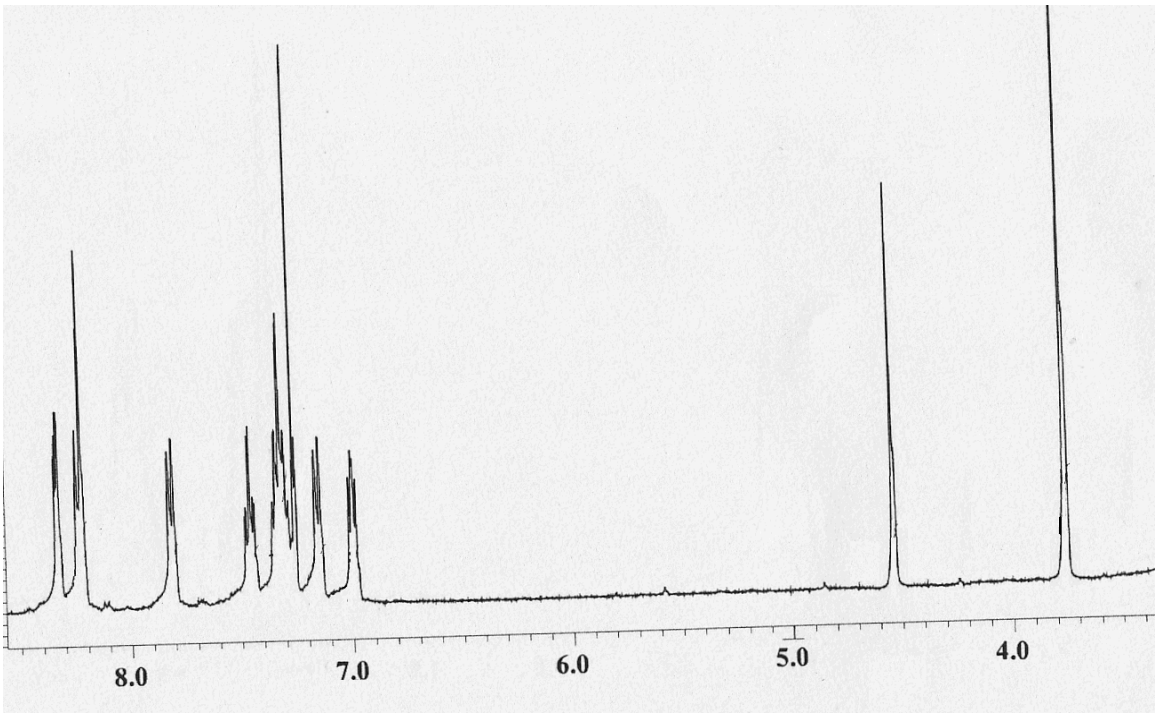
Partial ^{1}H-NMR of **2** with fumaric acid:



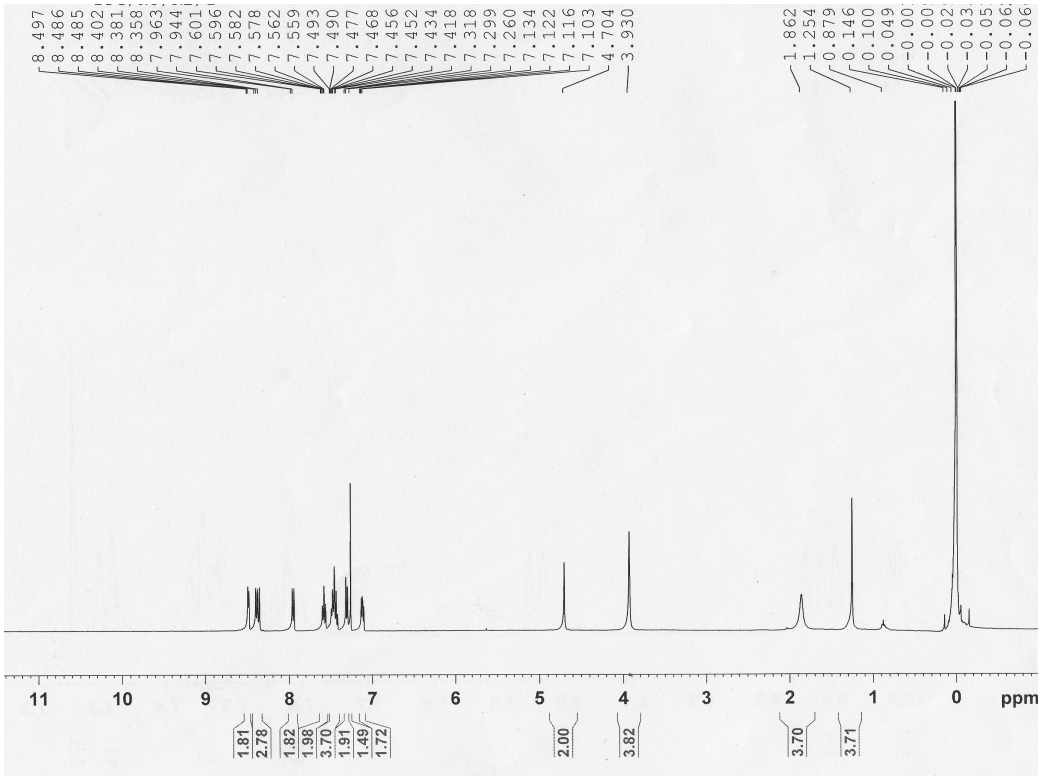
Partial ^{1}H-NMR of **2** with dl-malic acid:



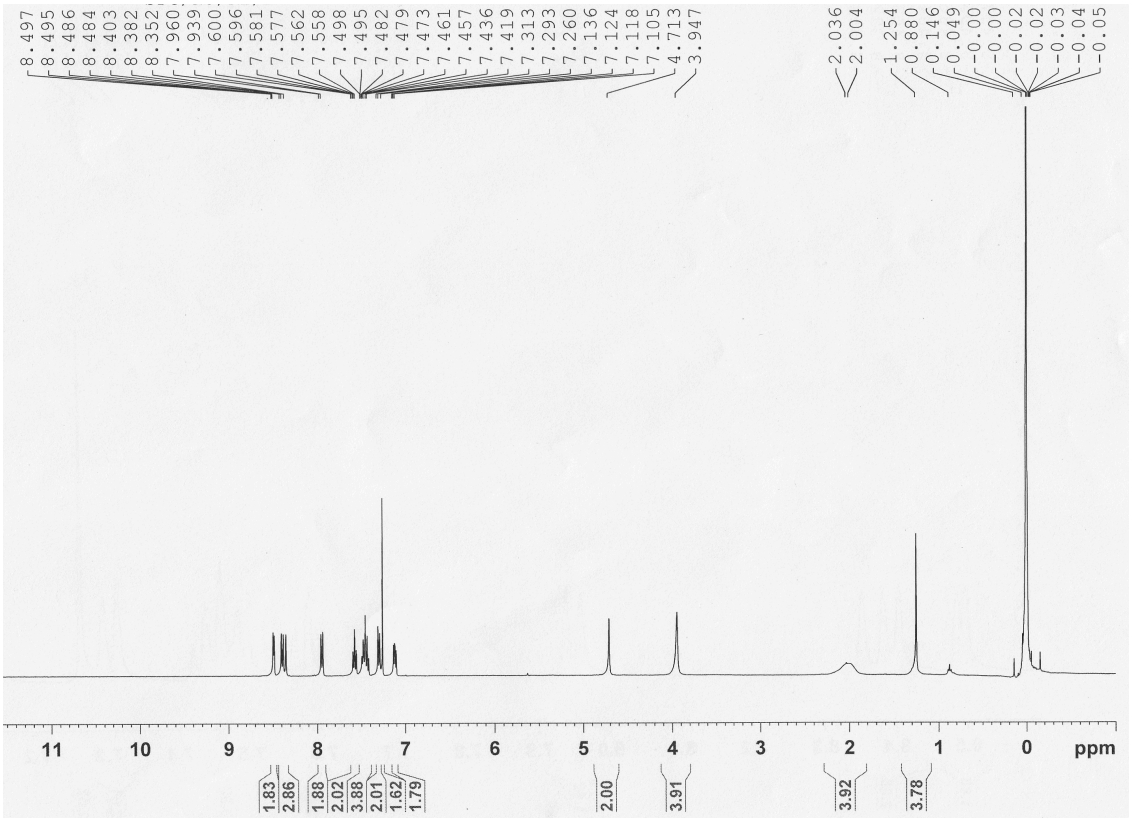
Partial ^{1}H-NMR of **2** with cirtic acid:



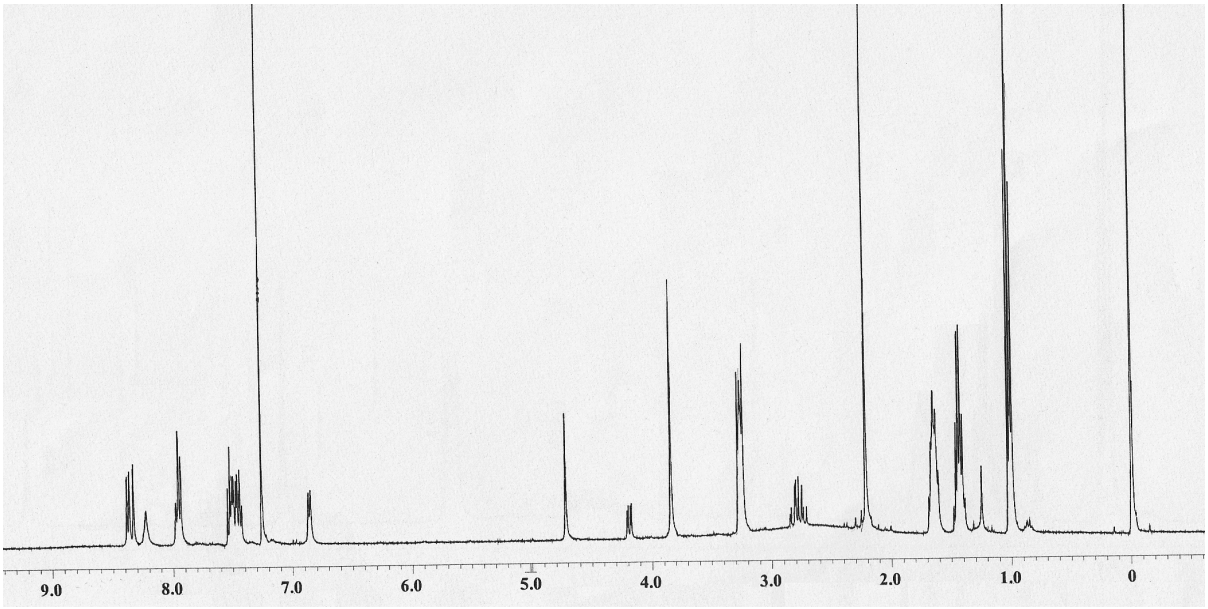
Partial ^{1}H-NMR of **2** with TFA (1:1):



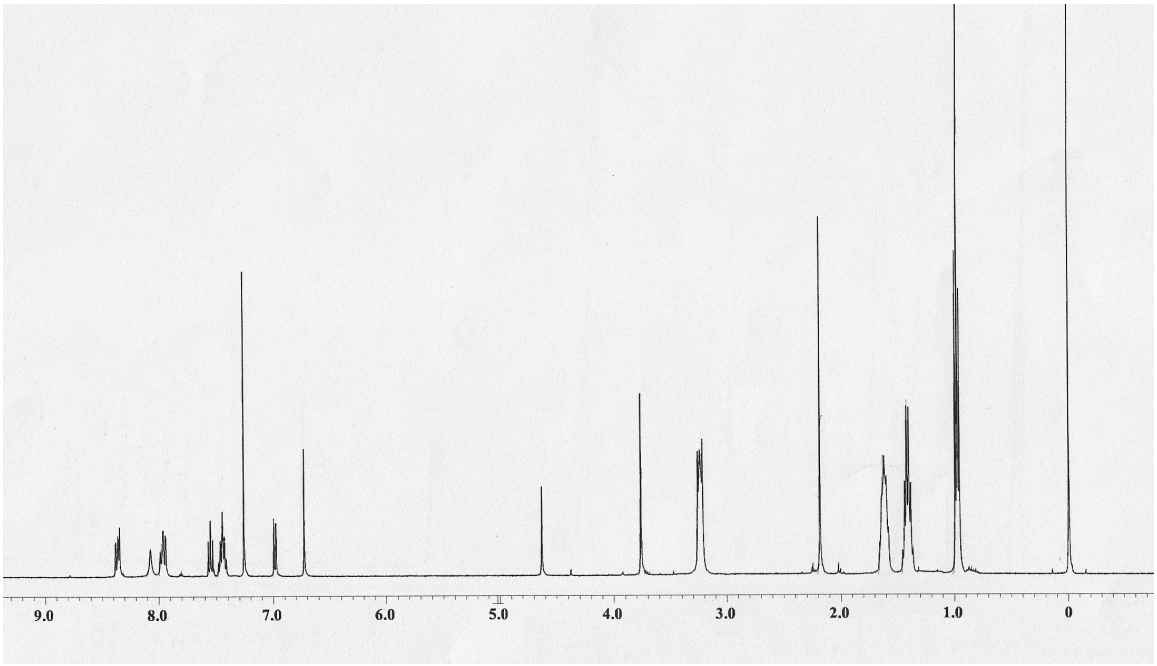
Partial ^{1}H-NMR of **2** with TFA (1:3):



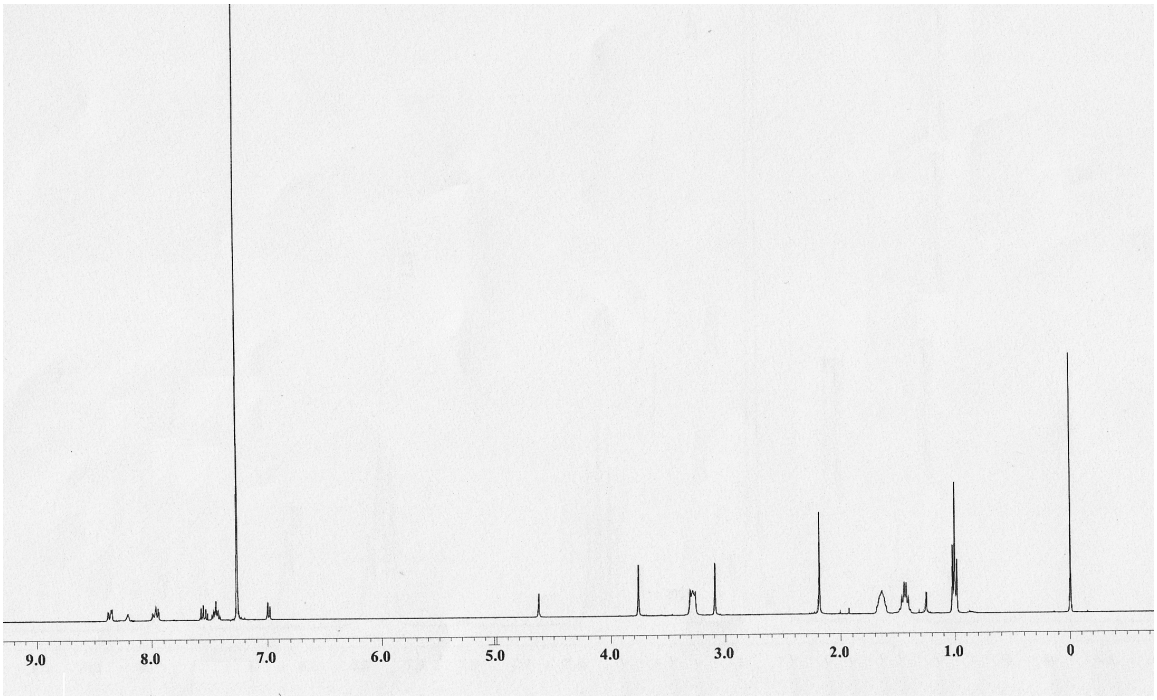
^{1}H NMR of **1** with dl-malate (1:1):



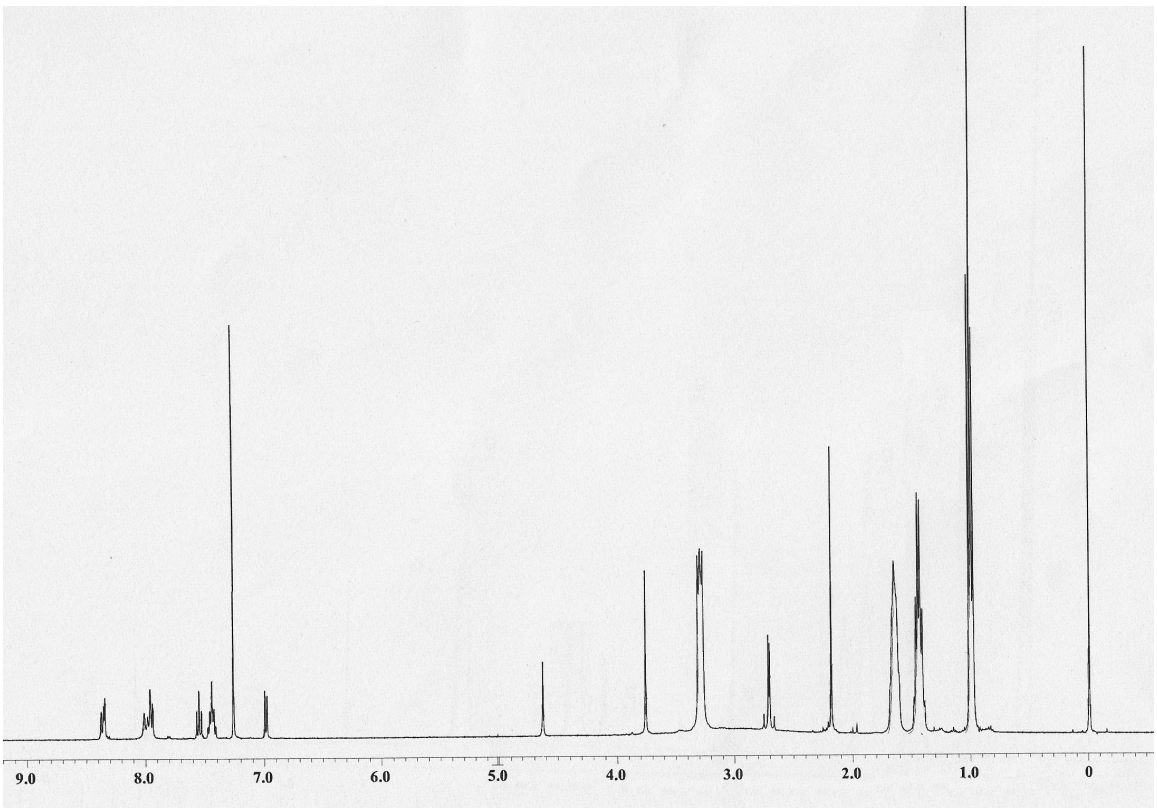
^{1}H NMR of **1** with fumarate (1:1):



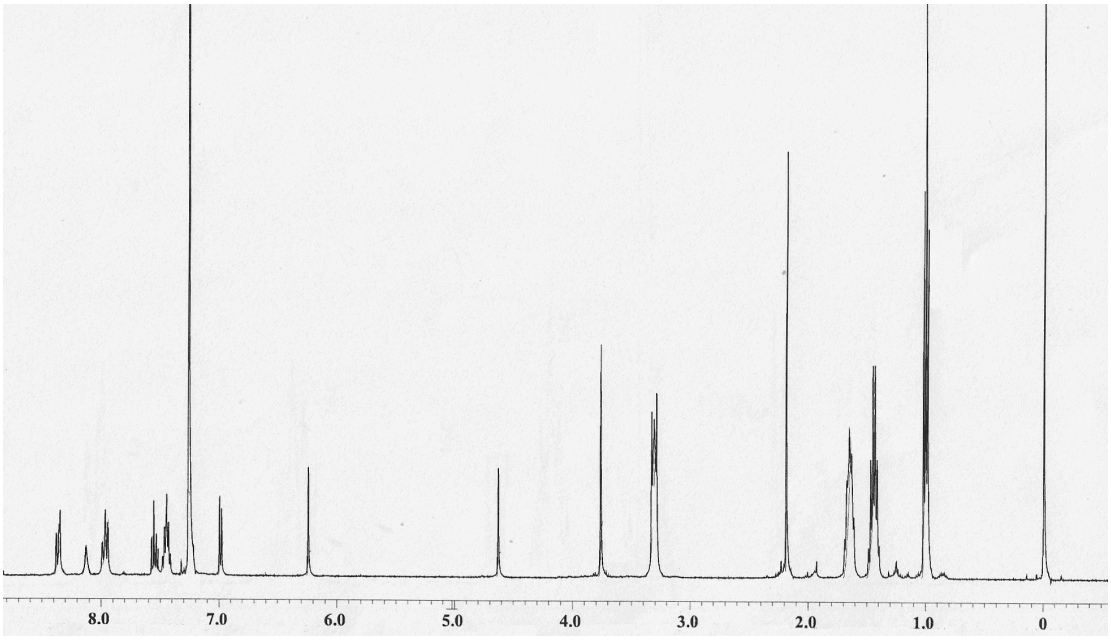
^{1}H NMR of **1** with succinate (1:1):



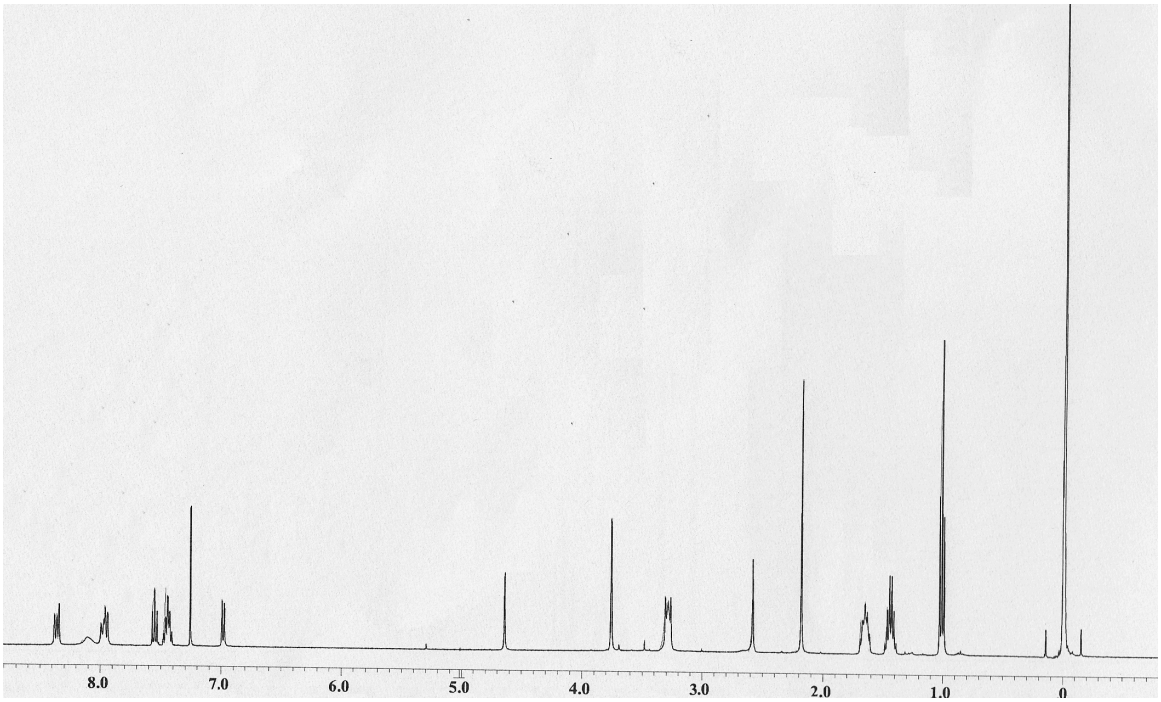
^{1}H NMR of **1** with citrate (1:1):



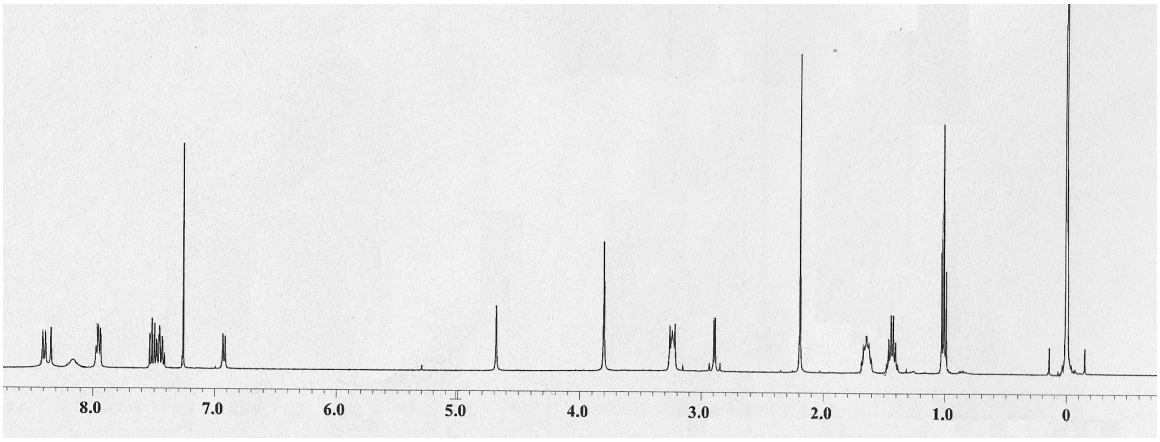
^{1}H NMR of **1** with maleate (1:1):



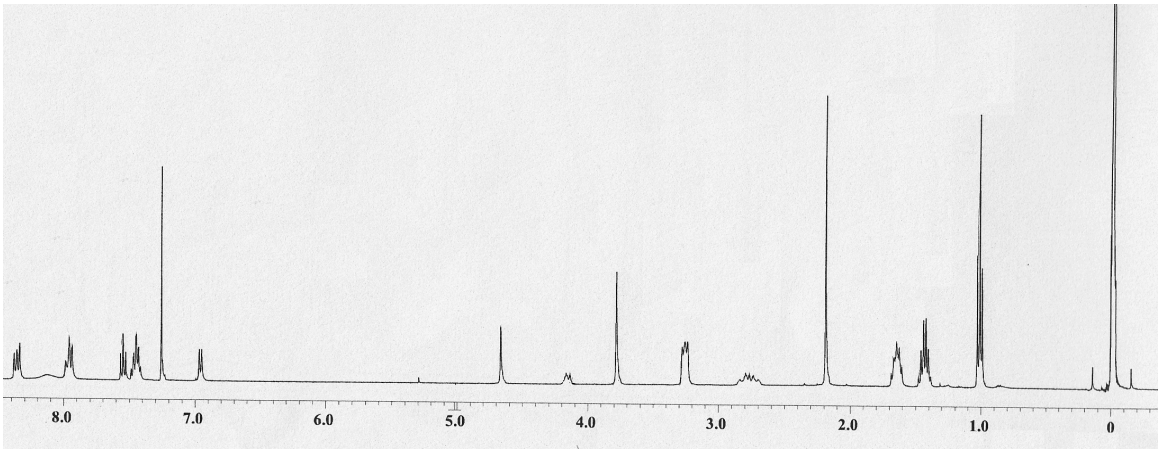
^{1}H-NMR of **1** with monotetrabutylamonium salt of succinic acid:



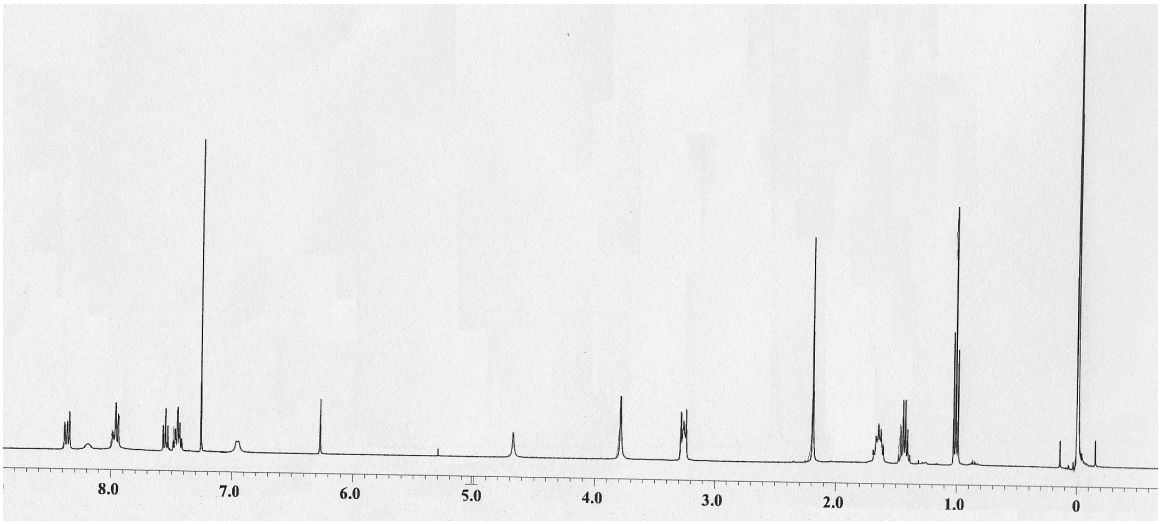
^{1}H-NMR of **1** with monotetrabutylamonium salt of citric acid:



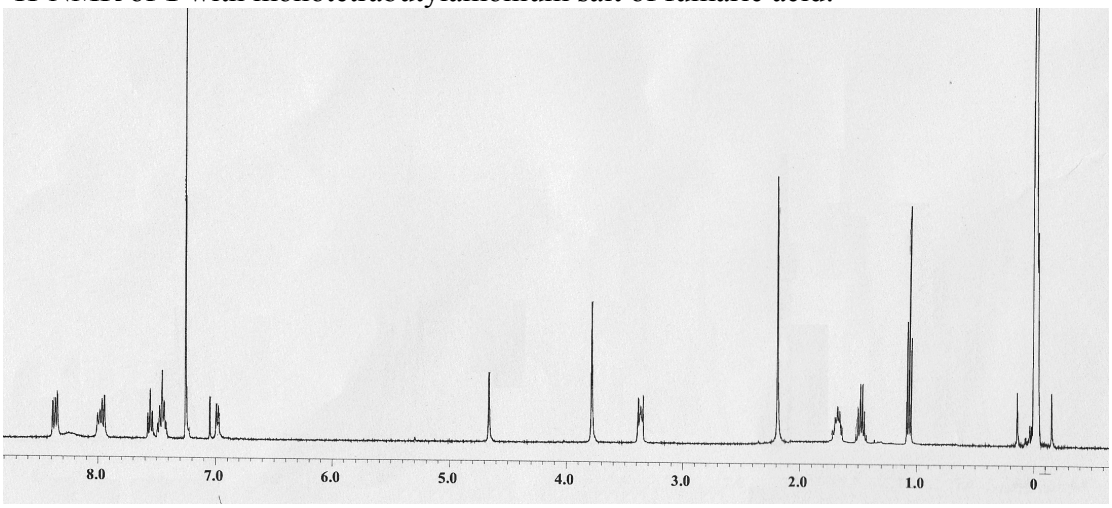
^{1}H-NMR of **1** with monotetrabutylamonium salt of dl-malic acid:



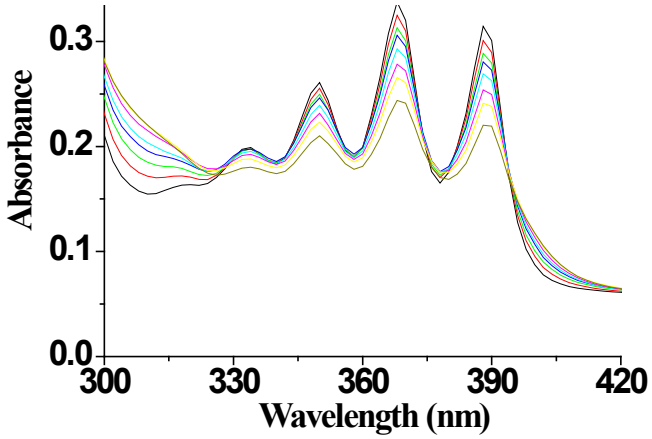
^{1}H-NMR of **1** with monotetrabutylamonium salt of maleic acid:



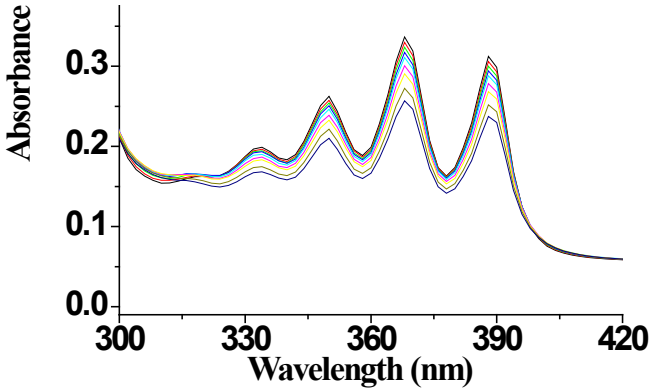
^{1}H-NMR of **1** with monotetrabutylamonium salt of fumaric acid:



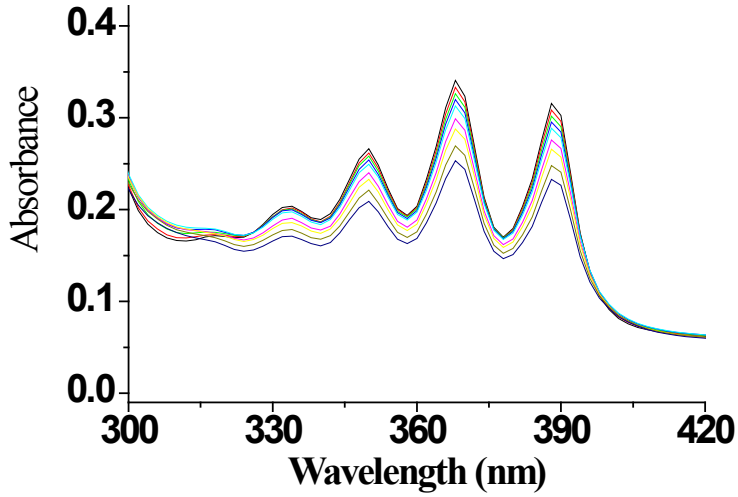
Change of absorbance upon addition of citric acid to **1** (c = 4.0 X 10^{-5} M).



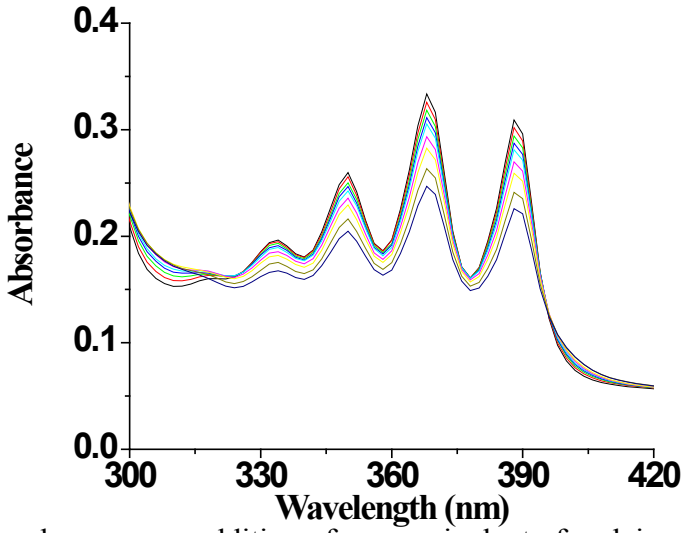
Change of absorbance upon addition of fumaric acid to **1** (c = 4.0 X 10^{-5} M).



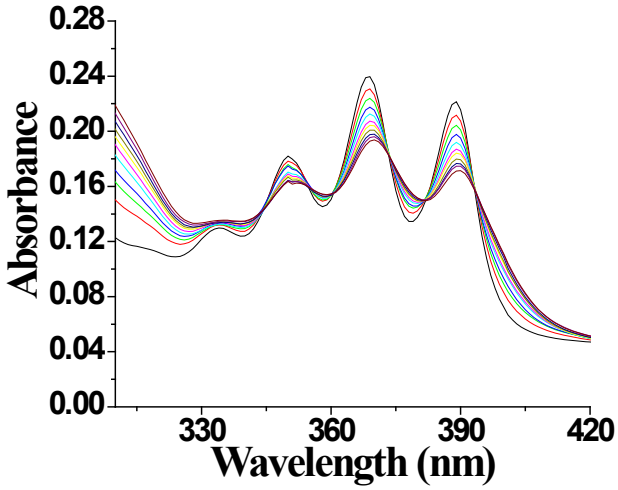
Change of absorbance upon addition of Succinic acid to **1** (c = 4.0 X 10^{-5} M).



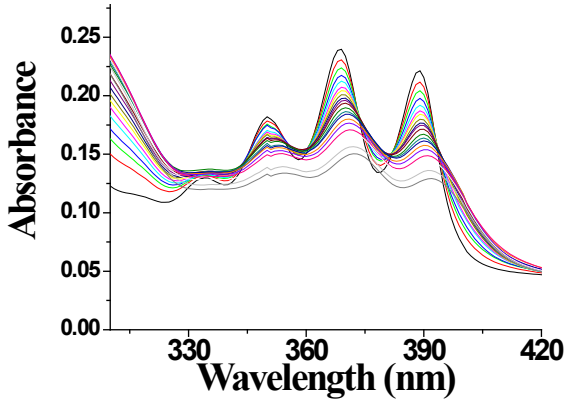
Change of absorbance upon addition of dl-malic acid to **1** (c = 4.0 X 10^{-5} M).



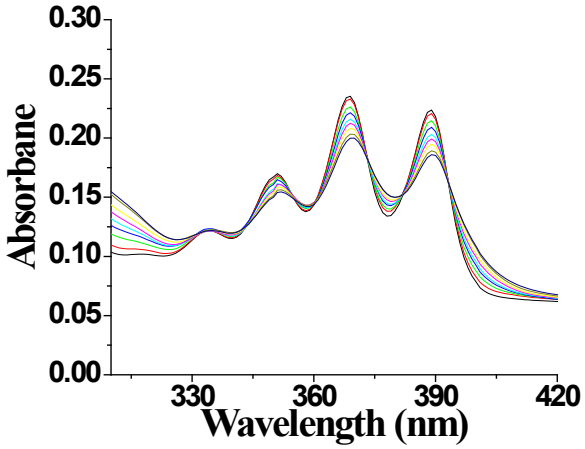
Change of absorbance upon addition of one equivalent of maleic acid to **1** (c = 4.0 X 10^{-5} M).



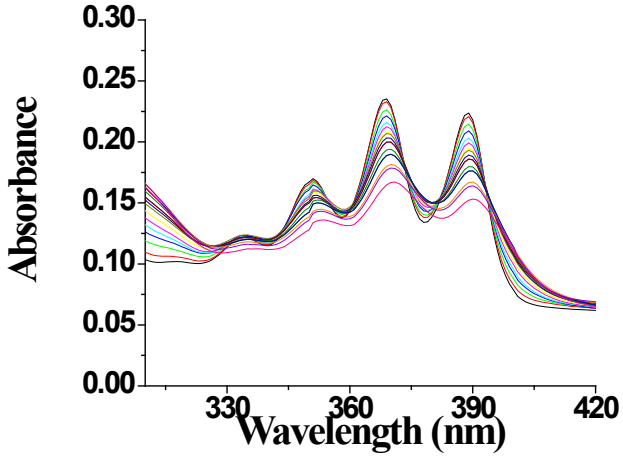
Change of absorbance upon addition of three equivalent of maleic acid to **1**(c = 4.0 X 10^{-5} M).



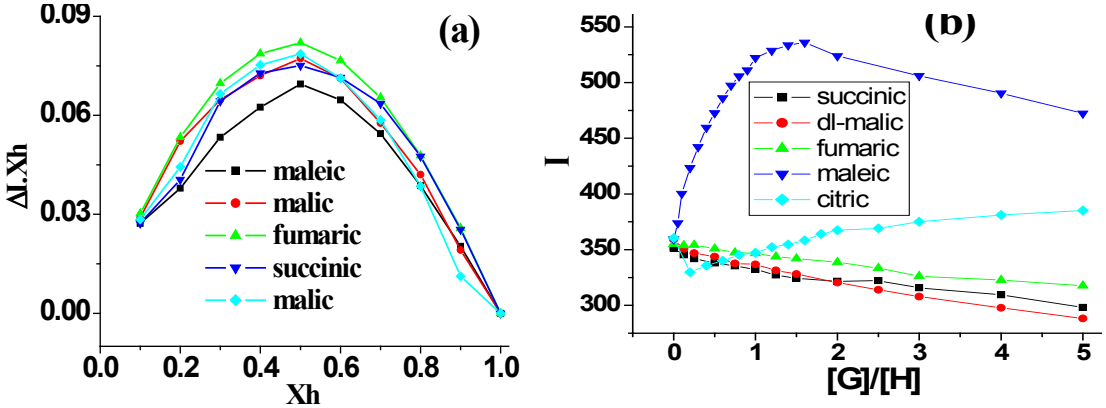
Change of UV upon addition of one equivalent TFA to **1**



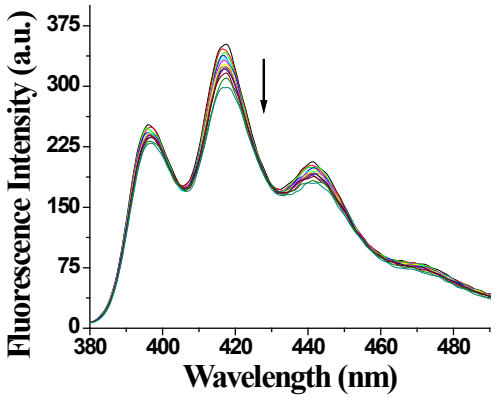
Change of UV upon addition of three equivalents TFA to **1**



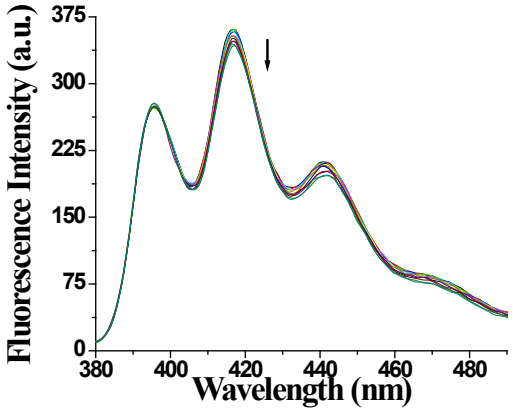
(a) Job plot and (b) fluorescence titration curve of **1** with acids



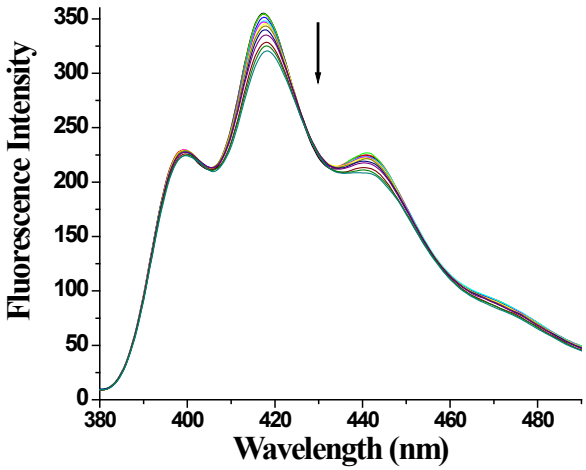
Fluorescence titration spectra of succinic acid with **1**:



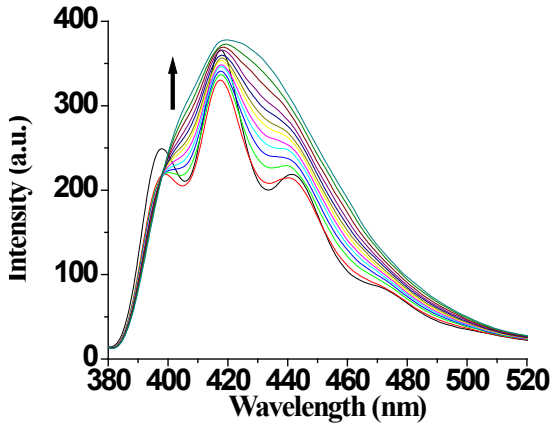
Fluorescence titration spectra of *dl*-malic acid with **1**:



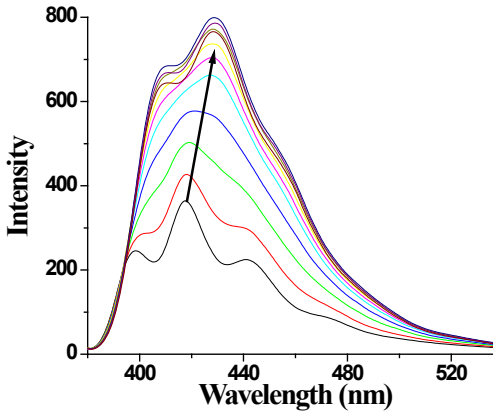
Fluorescence titration spectra of fumaric acid with **1**:



Fluorescence titration spectra of citric acid with **1**:



Fluorescence titration spectra of the sensor with TFA:



Linear Regression for **1** with succinic acid:
Y = A + B * X

Parameter	Value	Error
A	5.69351	0.68489
B	4.25681	0.14761

R	SD	N	P
0.99404	1.67561	12	<0.0001

Linear Regression for **1** with dl-malic acid:
Y = A + B * X

Parameter	Value	Error
A	2.74266	0.55066
B	4.34273	0.18958

R	SD	N	P
0.99246	1.04044	10	<0.0001

Linear Regression for **1** with fumaric acid:
Y = A + B * X

Parameter	Value	Error
A	-24.38899	4.8446
B	27.88158	1.09309

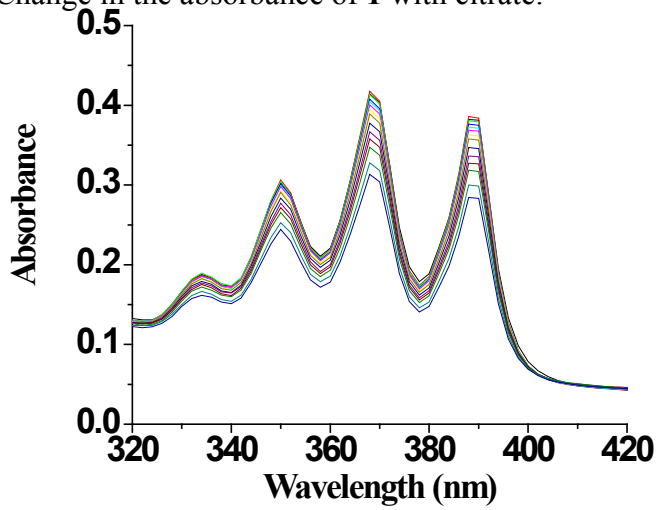
R	SD	N	P
0.99315	11.85242	11	<0.0001

Linear Regression for **1** with maleic acid:
Y = A + B * X

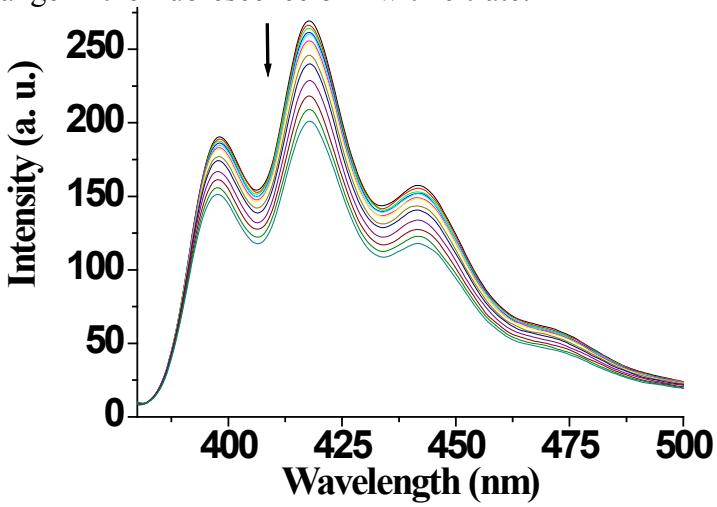
Parameter	Value	Error
A	1.55365	0.06063
B	0.29573	0.0071

R	SD	N	P
0.99656	0.16253	14	<0.0001

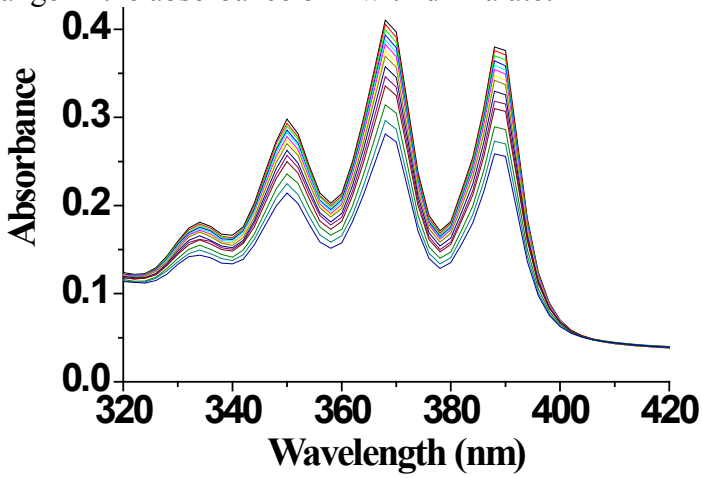
Change in the absorbance of **1** with citrate:



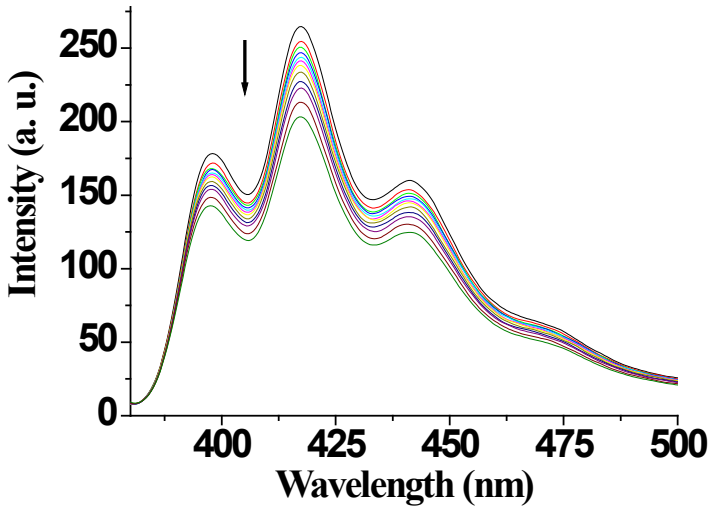
Change in the fluorescence of **1** with citrate:



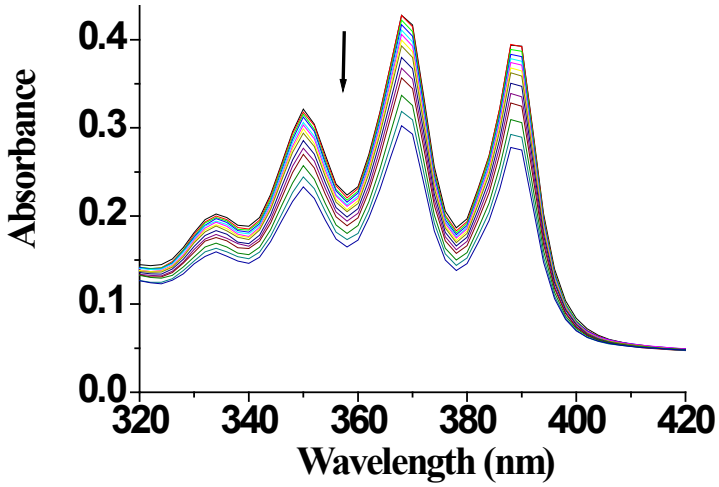
Change in the absorbance of **1** with dl-malate:



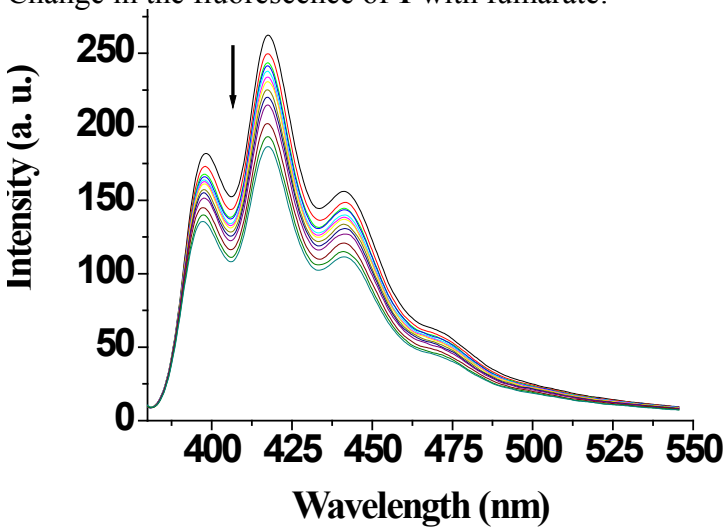
Change in the fluorescence of **1** with dl-malate:



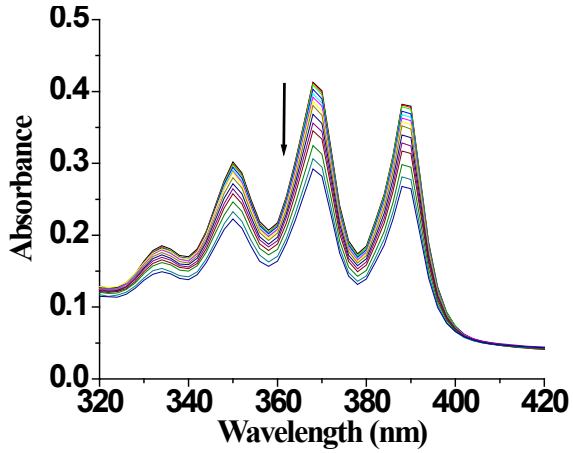
Change in the absorbance of **1** with fumarate:



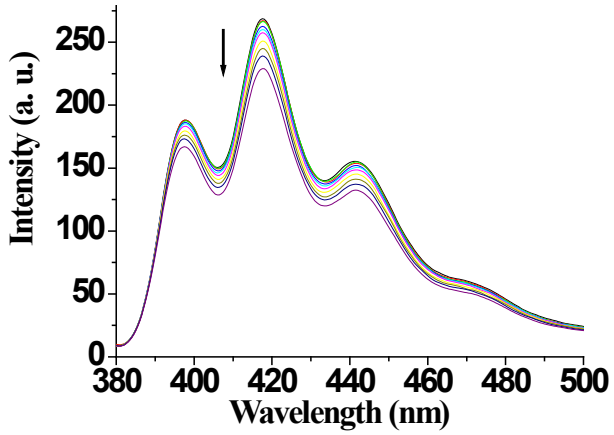
Change in the fluorescence of **1** with fumarate:



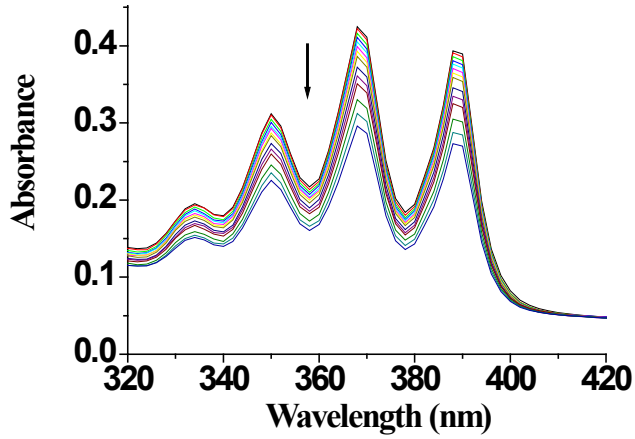
Change in the absorbance of **1** with maleate:



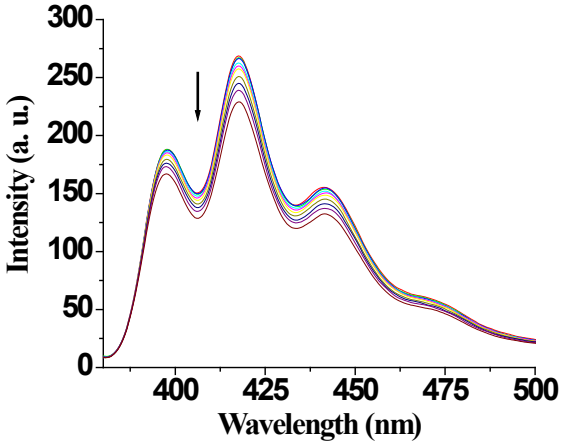
Change in the fluorescence of **1** with maleate:



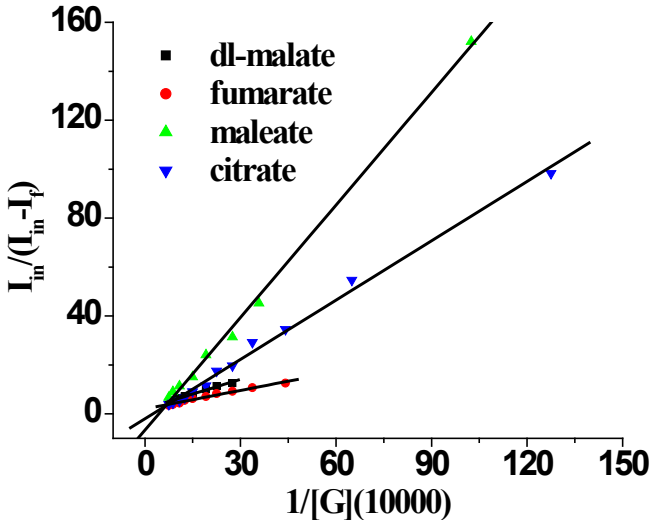
Change in the absorbance of **1** with succinate



Change in the fluorescence of **1** with succinate



Binding constant calculation plot of **1** with guest anions:



Linear Regression for **1** with dl-malate:

Y = A + B * X

Parameter	Value	Error
A	2.08096	0.44697
B	0.40062	0.02518

R	SD	N	P
0.99026	0.41721	7	<0.0001

Linear Regression for **1** with fumarate:

Y = A + B * X

Parameter	Value	Error
A	2.03198	0.29489
B	0.25159	0.01275

R	SD	N	P
0.98989	0.45682	10	<0.0001

Linear Regression for **1** with maleate:

Y = A + B * X

Parameter	Value	Error
A	-6.64544	1.32263
B	1.53292	0.03233

R	SD	N	P
0.99867	2.69395	8	<0.0001

Linear Regression for **1** with citrate:
Y = A + B * X

Parameter	Value	Error
A	-1.9575	0.94658
B	0.80795	0.0204

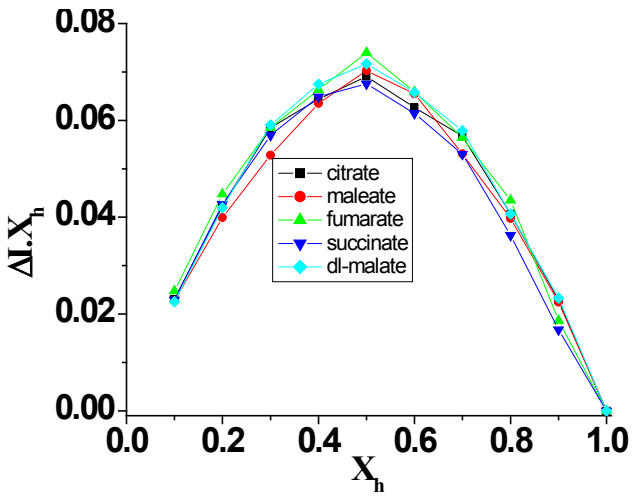
R	SD	N	P
0.99683	2.31584	12	<0.0001

Table 5: Binding constant of **1** with acids and their anions as tetrabutylammonium salt by fluorescence method

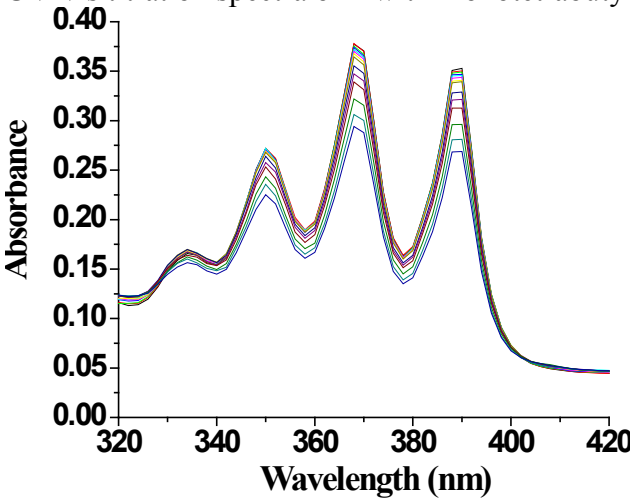
Entry	Acids	Binding constant of receptor **1** (M^{-1})	Anions of acids	Binding constant of receptor **1** (M^{-1})
1	succinic	$1.33X10^4$	succinic	-
2	dl-malic	$6.31X10^3$	dl-malic	$5.19X10^3$
3	fumaric	$8.75X10^3$	fumaric	$8.08X10^3$
4	maleic	$5.25X10^4$	maleic	$4.33X10^3$
5	citric	-	citric	$2.42X10^3$

'-' not determined due to irregular change

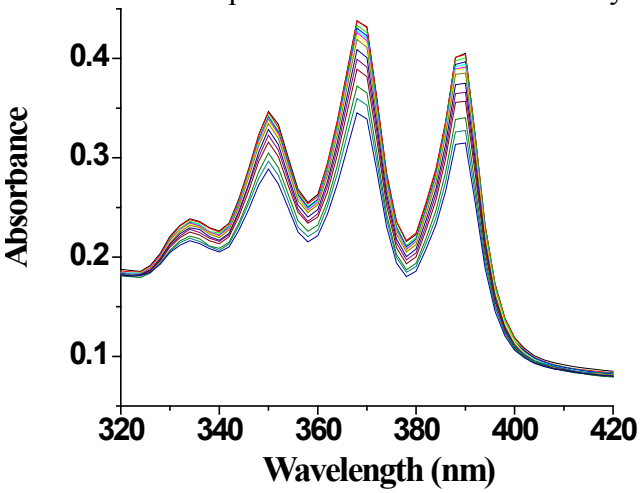
Job plot of **1** with corresponding carboxylates as their tetrabutylammonium salt:



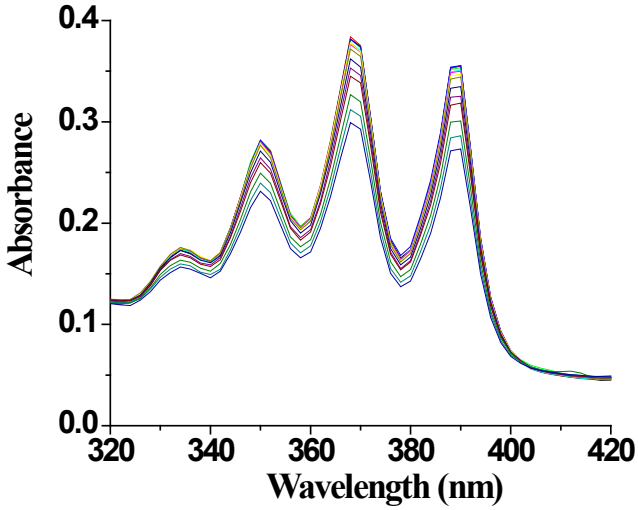
UV-vis titration spectra of **1** with monotetrabutylamonium salt of citric acid:



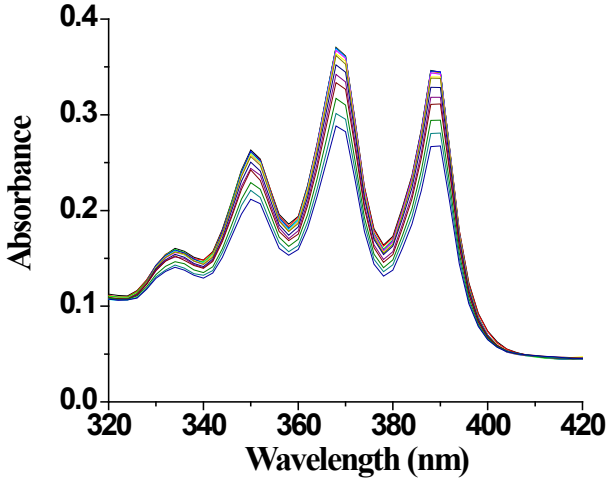
UV-vis titration spectra of **1** with monotetrabutylamonium salt of dl-malic acid:



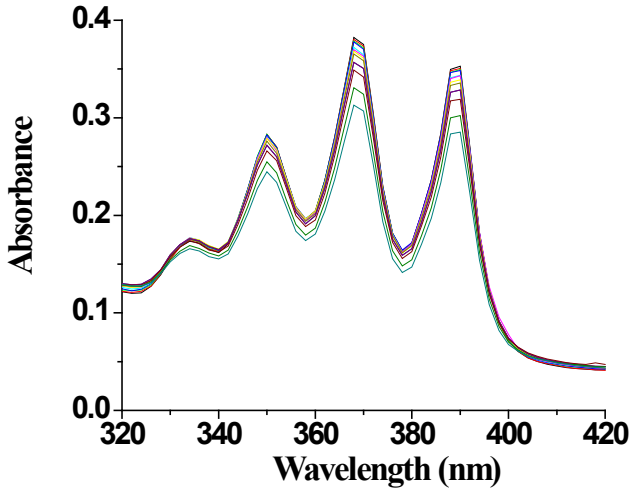
UV-vis titration spectra of **1** with monotetrabutylamonium salt of succinic acid:



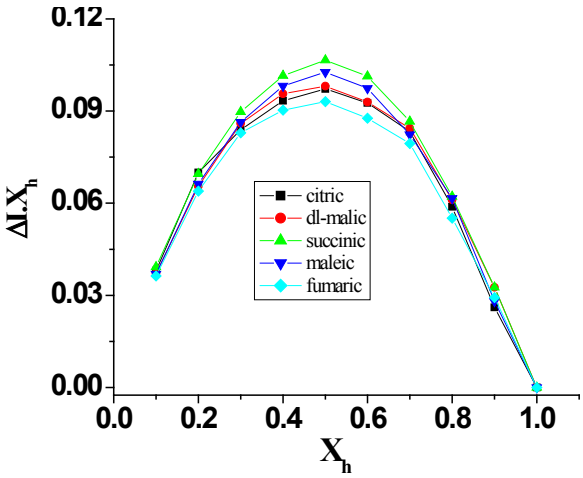
UV-vis titration spectra of **1** with monotetrabutylamonium salt of fumaric acid:



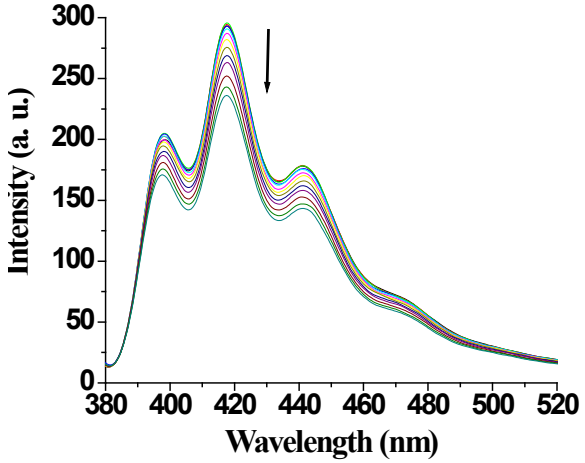
UV-vis titration spectra of **1** with monotetrabutylamonium salt of fumaric acid:



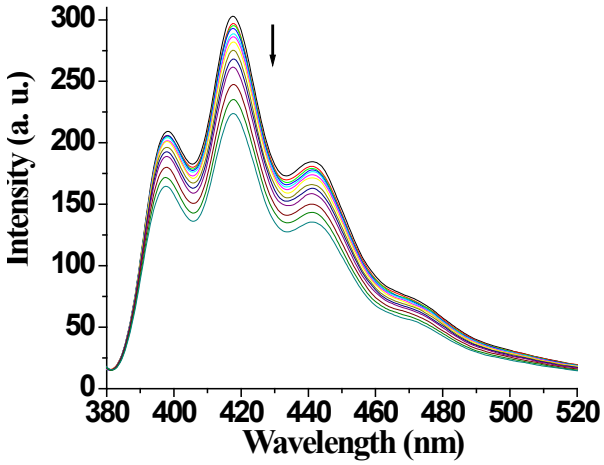
Job plot of receptor **1** with monotetrabutylamonium salts of guest acids:



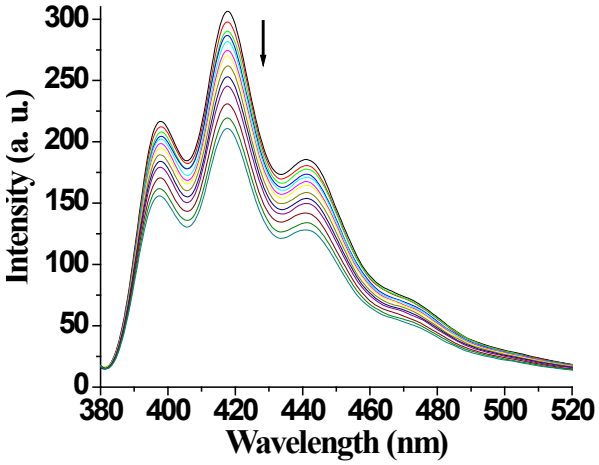
Fluorescence titration spectra of **1** with monotetrabutylamonium salt of citric acid:



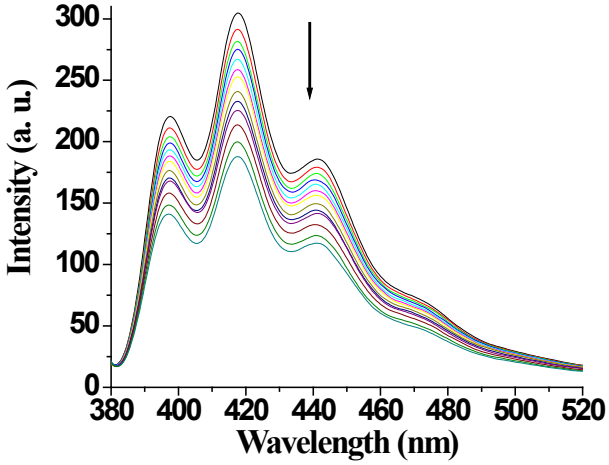
Fluorescence titration spectra of **1** with monotetrabutylamonium salt of dl-malic acid:



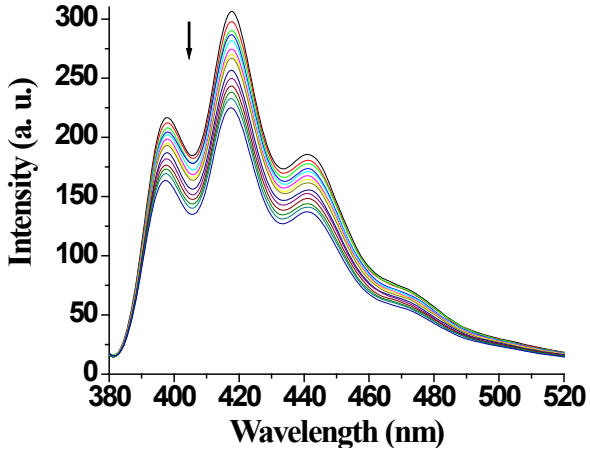
Fluorescence titration spectra of **1** with monotetrabutylamonium salt of succinic acid:



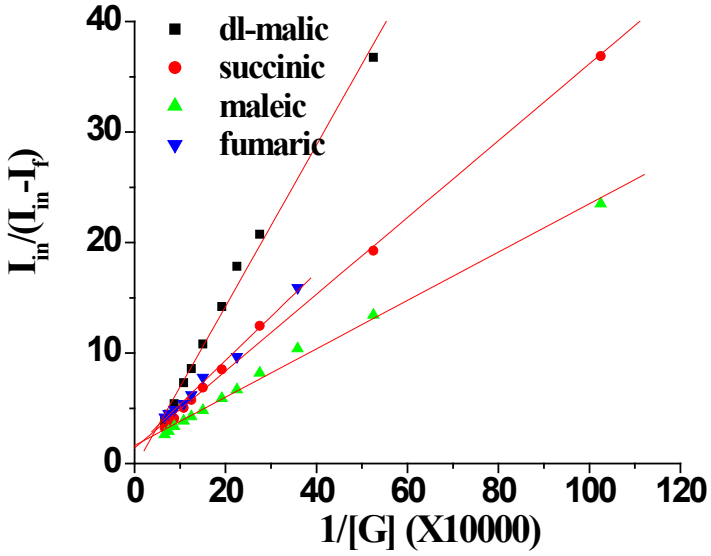
Fluorescence titration spectra of **1** with monotetrabutylamonium salt of maleic acid:



Fluorescence titration spectra of **1** with monotetrabutylamonium salt of fumaric acid:



Binding constant calculation curve of the **1** with monotetrabutylamonium salt of guest acids by fluorescence method:



Linear Regression for **1** with mono TBA salt of dl-malic acid:
Y = A + B * X

Parameter	Value	Error
A	-0.35555	0.52334
B	0.72958	0.02326

R	SD	N	P
0.99596	0.9636	10	<0.0001

Linear Regression for **1** with mono TBA salt of succinic acid:
Y = A + B * X

Parameter	Value	Error
A	1.56944	0.34762
B	0.34956	0.00928

R	SD	N	P
0.9965	0.84265	12	<0.0001

Linear Regression 1 with mono TBA salt of maleic acid:
Y = A + B * X

Parameter	Value	Error
A	1.63431	0.18366
B	0.21875	0.0049

R	SD	N	P
0.9975	0.44521	12	<0.0001

Linear Regression for **1** with mono TBA of fumaric acid:
Y = A + B * X

Parameter	Value	Error
A	2.20135	0.27955
B	0.34019	0.00746

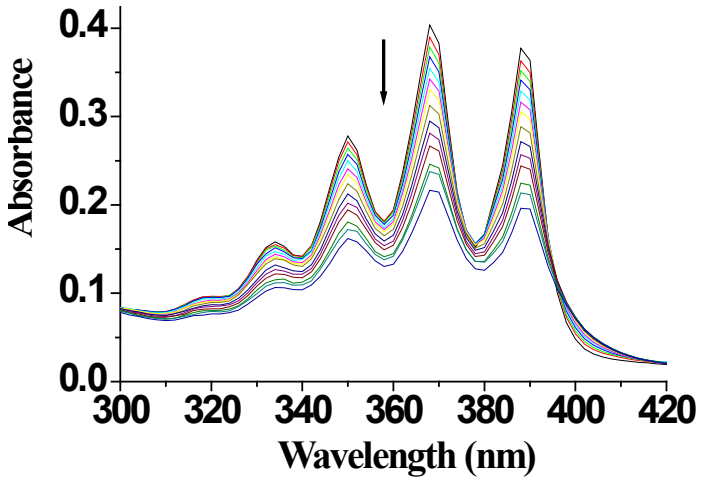
R	SD	N	P
0.9976	0.67766	12	<0.0001

Table 6: binding constants of **1** (c = $4X10^{-5}$ M) with monotetrabutylamonium salts of the guest acids at 25 °C:

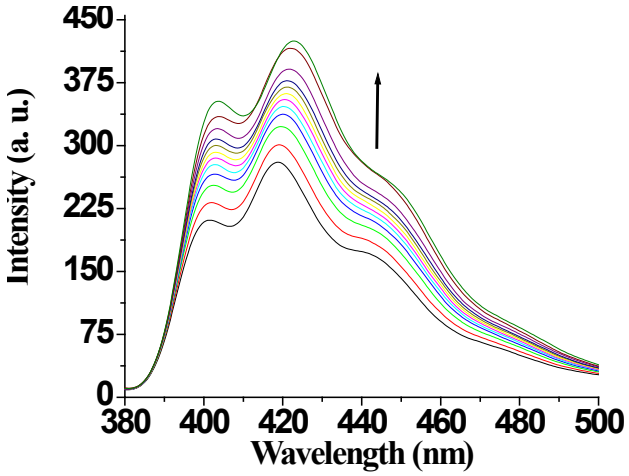
Entry	Name of the monotetrabutylamonium salt	Binding constants (M^{-1})
1	monotetrabutylamonium salt of citric acids	-
2	monotetrabutylamonium salt of dl-malic acids	$4.87X10^{4}$
3	monotetrabutylamonium salt of succinic acids	$4.49X10^{5}$
4	monotetrabutylamonium salt of maleic acids	$7.47X10^{5}$
5	monotetrabutylamonium salt of fumaric acids	$6.47X10^{5}$

'-' not determined due to irregular change

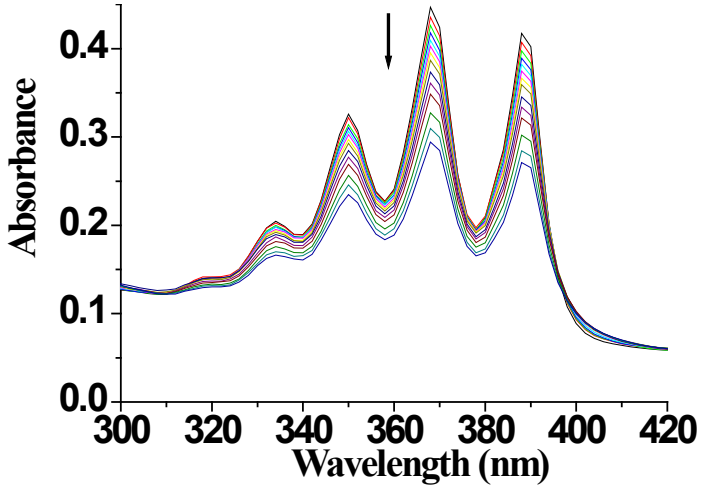
Change in the absorbance of **2** with succinic acid:



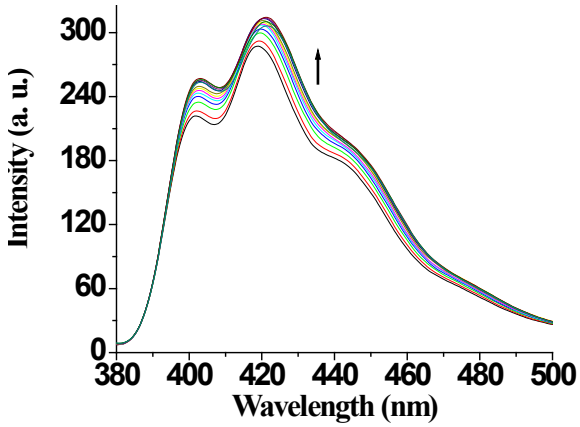
Change in the fluorescence of **2** with succinic acid:



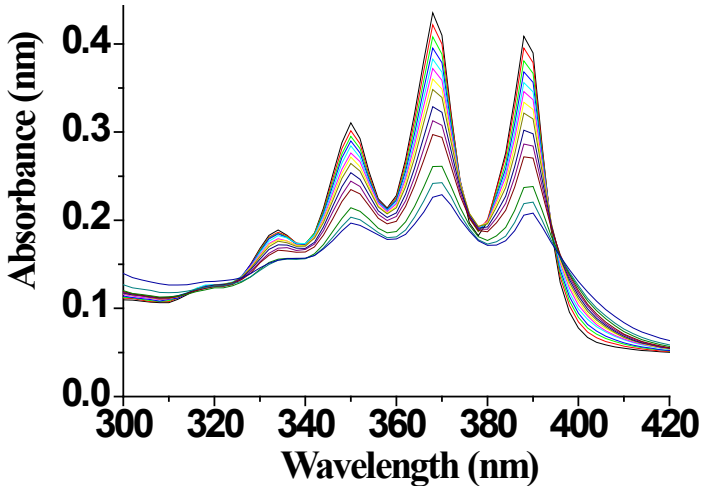
Change in the absorbance of **2** with fumaric acid:



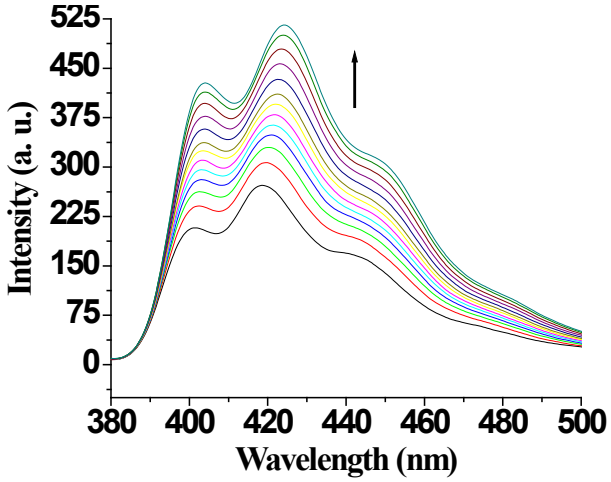
Change in the fluorescence of **2** with fumaric acid:



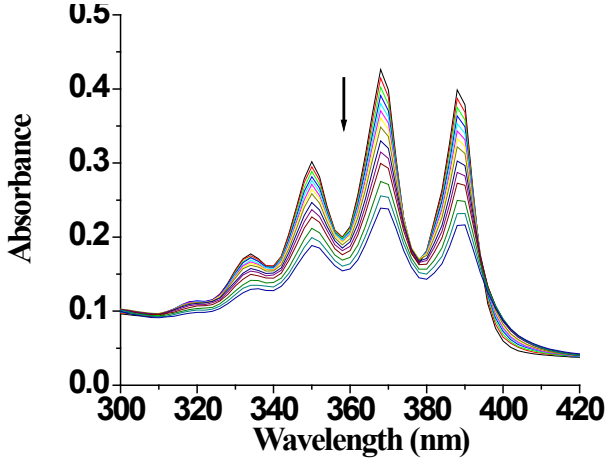
Change in the absorbance of **2** with citric acid:



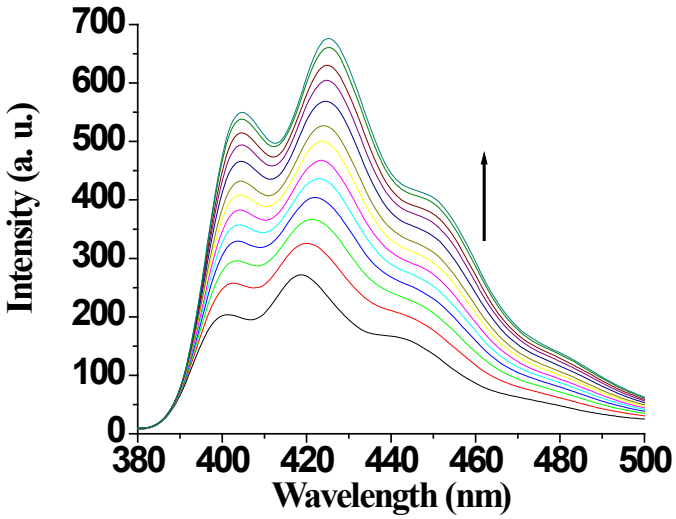
Change in the fluorescence of **2** with citric acid:



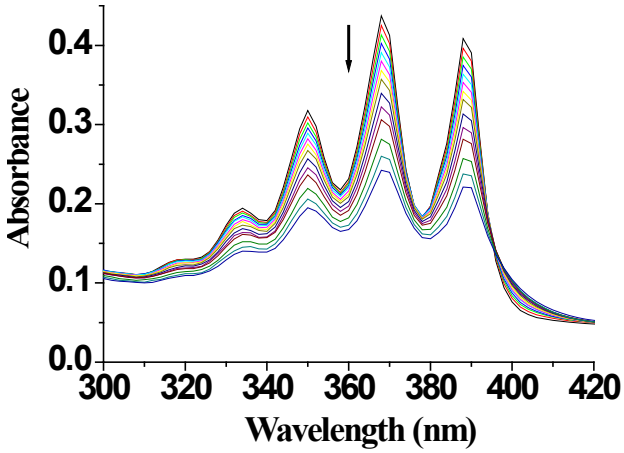
Change of absorbance of **2** with maleic acid:



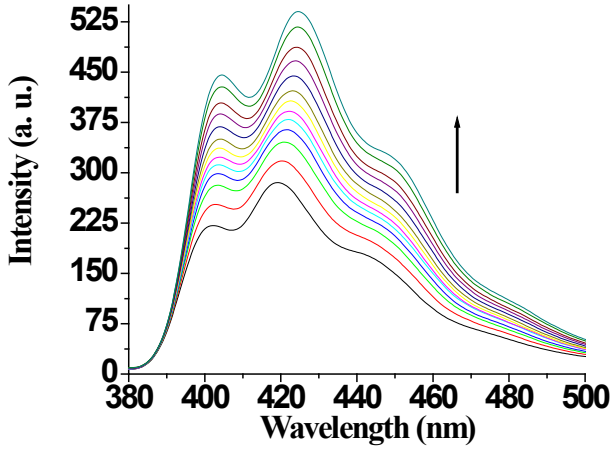
Change of fluorescence of **2** with maleic acid:



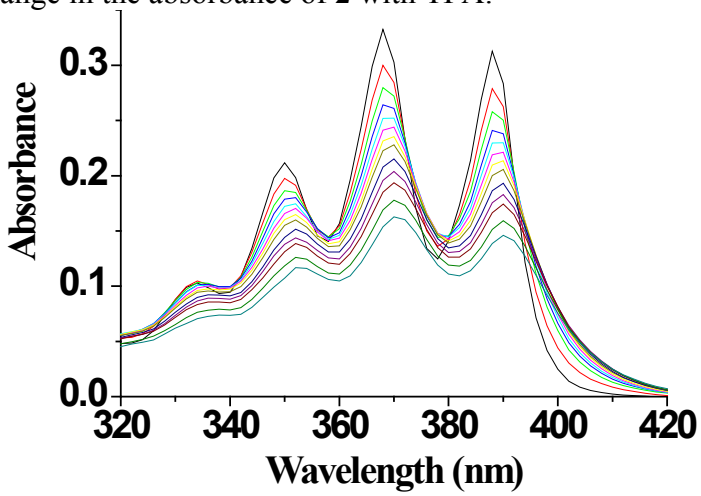
Change in the absorbance of **2** with dl-malic:



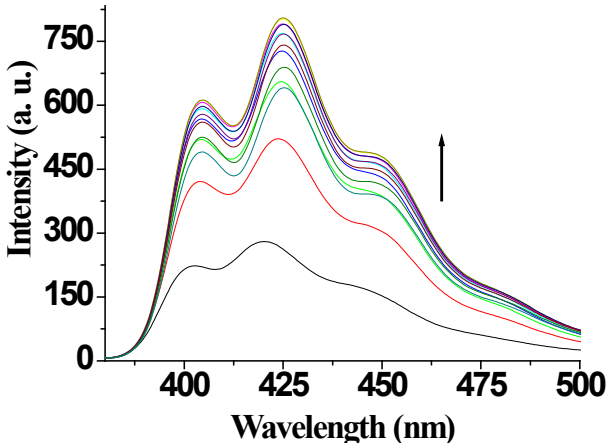
Change in the fluorescence of **2** with dl-malic:



Change in the absorbance of **2** with TFA:



Change in the fluorescence of **2** with TFA:



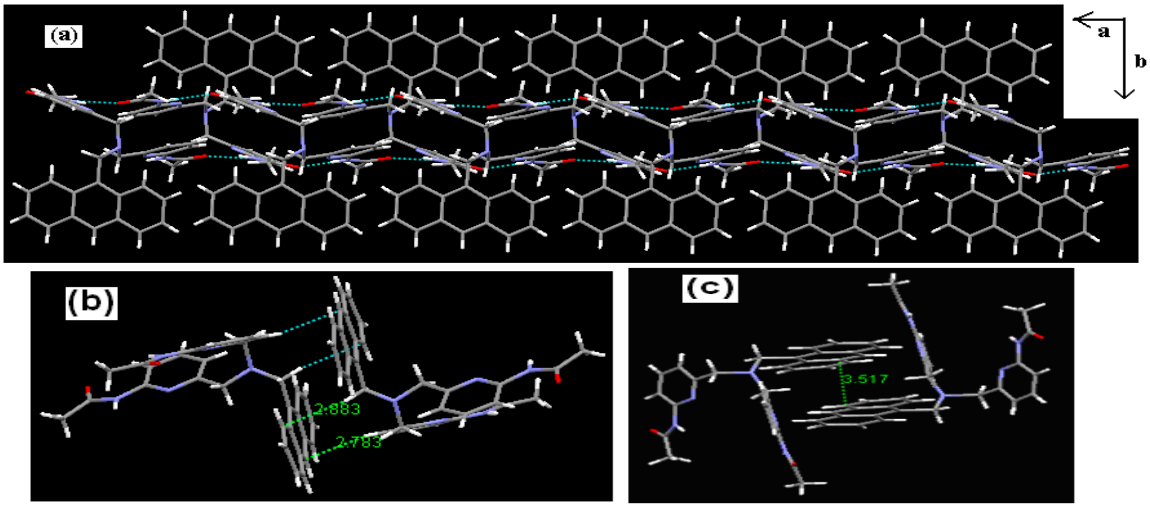


Figure 6: (a) Polymeric zigzag chains of **1** along the crystallography *a* axis, (b) showing two CH/ π stacking interactions with anthracene and (c) stacking between the anthracene rings in form **A**.

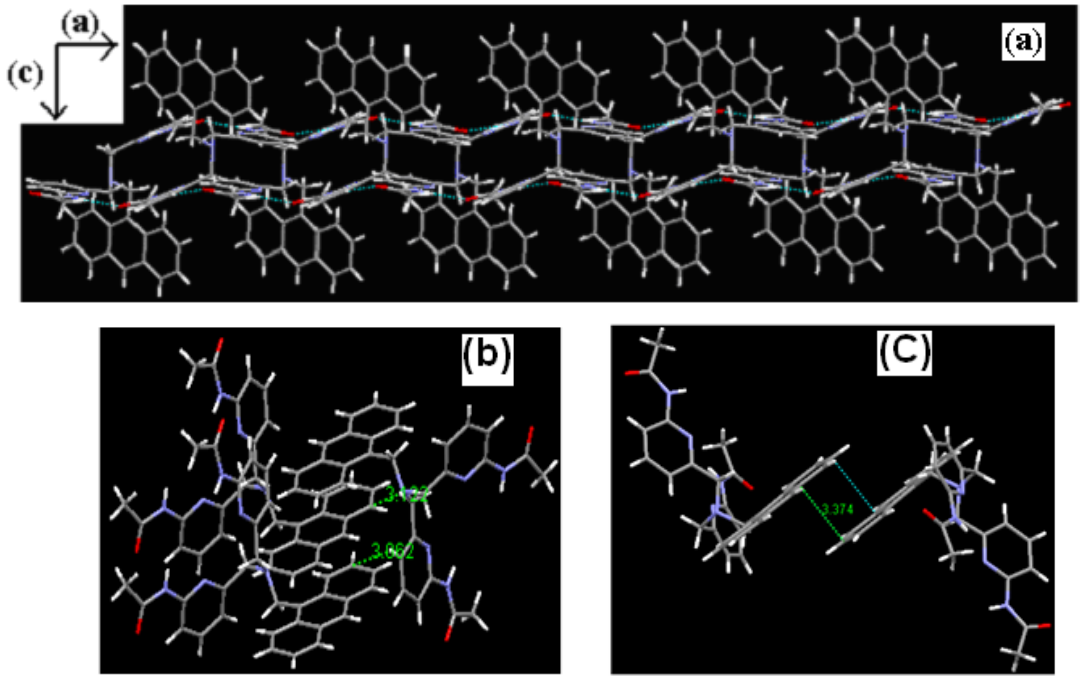


Figure 7: (a) Polymeric zigzag chains of the **1** along the crystallography *a* axis, (b) showing two CH/π interactions with anthracene and (c) stacking between the anthracene rings in form **B**.

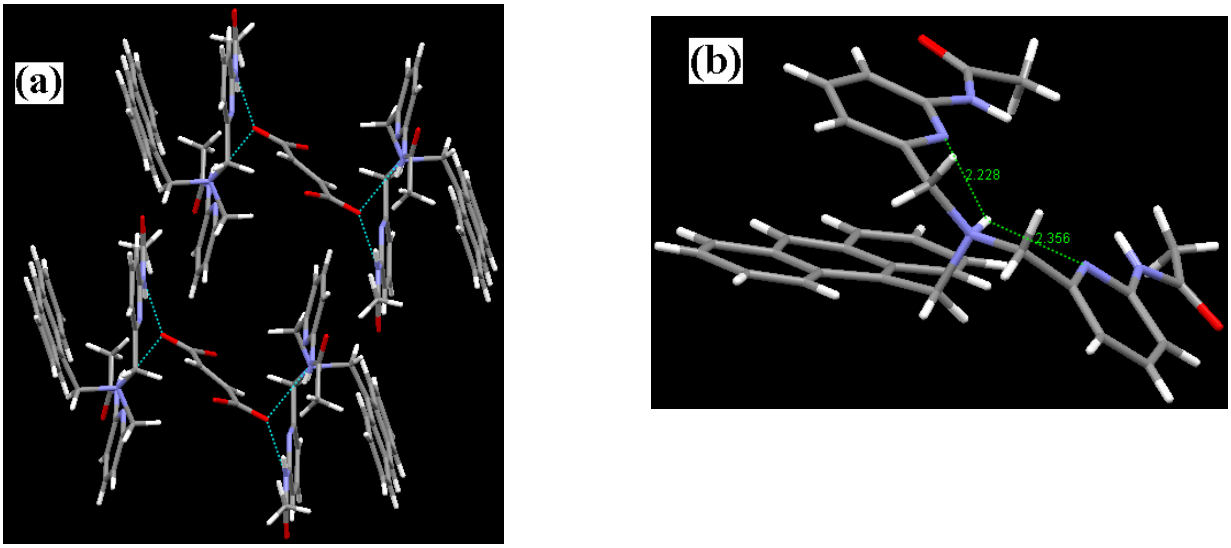


Figure 8: Illustration of crystal structure of co-crystal **A** (a) viewed down the crystallography *a* axis and (b) distances between the tertiary nitrogen proton and nitrogen of pyridine rings and

Table 7: Crystallographic data and structure refinement parameters of **1** in form **A**, form **B** and **co-crystal A**.

Compound	**Form A**	**Form B**	**Co-crystal A**
CCDC No.	CCDC 780314	CCDC 780315	CCDC 789746
Empirical Formula	$C_{31}H_{29}N_5O_2$	$C_{31}H_{29}N_5O_2$	$(C_{31}H_{30}N_5O_2)_2$, $(C_4H_4O_4)_4$, $(C_4H_2O_4)$, $(H_2O)_2$
Formula weight	503.59	503.59	811.79
Crystal system	Triclinic	Triclinic	Monoclinic
Space group	P-1 (No. 2)	P-1 (No. 2)	P21/c (No. 14)
T [K]	296	296	100
a [Å]	9.4245 (2)	9.3680 (2)	13.9724 (4)
b [Å]	9.9653 (2)	9.5760 (3)	32.9044(9)
c [Å]	13.9428 (3)	16.6792 (5)	8.6563 (2)
α [deg]	84.353 (1)	79.700 (2)	90
β [deg]	88.257 (1)	77.133 (2)	101.086(2)
γ [deg]	89.524 (1)	66.303 (2)	90
Z	2	2	4
V [Å^3]	1302.50 (5)	1328.99 (6)	3905.50(18)
Ï [Å]	0.71073	0.71073	0.71073
D_{calc} [g/cm^3]	1.284	1.258	1.381
F [000]	532	532	1704
Crystal size [mm]	0.10 x 0.37 x 0.59	0.11 x 0.27 x 0.29	0.09 x 0.17 x 0.62
Theta min-max [deg]	1.5, 32.5	1.3, 26.0	1.9, 30.0
μ [mm^{-1}]	0.083	0.081	0.104
Index ranges	$-14 \le h \le 14$ $-14 \le k \le 15$ $-21 \le l \le 21$	$-11 \le h \le 11$ $-10 \le k \le 11$ $-19 \le l \le 20$	$-19 \le h \le 12$ $-45 \le k \le 45$ $-12 \le l \le 12$
Reflections collected	38829	20782	49877
Unique reflections	9347	5206	11309
Observed reflections [I > 2.0 sigma(I)]	5742	3539	6798
R_1 [$I > 2\sigma(I)$]	0.0551	0.0538	0.0878
wR2	0.1579	0.1453	0.2596
GOF	1.06	1.05	1.05

Table 8: Hydrogen-bond parameters (Å, °) of **1** in form **A**.

D-H….A	D-H	H….A	D….A	D-H….A
N1-H1N1....O2^{i}	0.854 (16)	2.090 (16)	2.9300 (15)	167.8 (14)
N5-H1N5....O1^{ii}	0.847 (16)	2.155 (16)	2.9802 (15)	164.6 (14)
Intra C12-H12A....N3	0.93	2.62	3.1551 (18)	117
Intra C20-H20A....O1	0.93	2.32	2.8787 (18)	119
C23-H23B....O2^{i}	0.96	2.44	3.3092 (19)	150
Intra C24-H24B....N2	0.97	2.48	3.1644 (16)	128
Intra C26-H26A....N3	0.93	2.55	2.8650 (17)	100
Intra C28-H28A....O2	0.93	2.25	2.8315 (18)	120
C31-H31D....O1^{ii}	0.96	2.59	3.2254 (18)	124

Symmetry codes: (i) 2-x, 2-y,-z, (ii) 1-x, 2-y,-z

Table 9: Hydrogen-bond parameters (Å, °) of **1** in form **B**.

D-H….A	D-H	H….A	D….A	D-H….A
N1-H1N1....O2^{i}	0.86 (3)	2.06 (3)	2.899 (3)	164 (2)
N5-H1N5....O1^{ii}	0.88 (2)	2.10 (3)	2.974 (3)	170.1 (19)
Intra C20-H20A....O1	0.93	2.28	2.860 (3)	120
C23-H23A....N4^{ii}	0.96	2.53	3.453 (4)	161
Intra C24-H24A....N2	0.97	2.48	3.188 (3)	130
Intra C28-H28A....O2	0.93	2.29	2.865 (3)	120
C31-H31B....O1^{ii}	0.96	2.52	3.375 (4)	148

Symmetry codes: (i) 1-x,1-y,1-z, (ii) 2-x,1-y,1-z

Table 10: pKa values of different diacids.

Serial No.	Name of acid	First pKa	Second pKa	Third pKa
1	Succinic	4.2	5.6	-
2	dl-Malic	3.4	5.1	-
3	Fumaric	3.5	4.5	-
4	Maleic	1.8	6.5	-
5	Citric	3.1	4.7	5.4

Experimental

General

Melting points (mp) were recorded on an A. D. and Co. hot-coil stage melting point apparatus and are uncorrected. NMR spectra were recorded in dry $CDCl_3$ unless otherwise mentioned with TMS as the internal standard with Bruker AM 500 MHz NMR and Bruker AVANCE DPX 300 MHz instruments (mentioned case wise). Chemical shifts are given in δ (ppm) scale and *J* values in Hz. IR spectra were measured in KBr disk with a JASCO FT/IR-460 plus spectrometer. UV–vis spectra were recorded on a JASCO V-530. Fluorescence spectra were recorded on Perkin Elmer LS-55. HRMS of **1** and compound **3** were recorded on a Qtof Micro YA263 instrument. Mass spectra of other compounds are done by Micromass Quattro II (LC- ESI-MS)**.** All solvents were dried prior to use by common methods. Silica gel 100–200 mesh was used for all chromatographic purifications. Starting materials are commercially available (purchased from Fluka and Aldrich).

General procedure for UV-vis. and Fluorescence titration

Stock solution of sensor **1** and **2** was prepared at a concentration of $4X10^{-5}$ mol/dm^3 in dry $CHCl_3$. Acids were dissolved in 2% DMSO in $CHCl_3$ in $4X10^{-4}$ mol/dm^3 concentration. DMSO (2%) was added to make a homogeneous solution. Solutions of receptors (2 mL) were taken in the cell and guest solutions were then added to solutions and continuous changes in absorbance and fluorescence intensity were recorded each time. The chloroform solution of guest anions were taken for titration as their tetrabutylammonium salts which are easily soluble in $CHCl_3$. Association constants were calculated by plotting I_0/I_0-I *vs* 1/[G] (I_0 and I are the initial and final intensity of the solution after each addition during titration).

Procedure for Job plot:

Solutions of **1** and **2** were prepared in a concentration $4X10^{-5}$ mol/dm^3. Guest solutions were also made in the concentration $4X10^{-5}$ mol/dm^3. The solutions of host and the guest were added in 1.8:0.2, 1.6:0.4, 1.4:0.6, 1.2:0.8, 1:1, 0.8:1.2, 0.6:1.4, 0.4:1.6, 0.2:1.8 ratio and the absorbances were recorded. Plot of Xh Vs ΔI.Xh give curves with breaking at 0.5 with mole fraction of host replies a 1:1 stoichiometry.

General Procedure for the synthesis of monotetrabutylamonium salt of the guest acids:

Mononeutralization reactions of the corresponding diacids by tetrabutylamonium hydroxide were carried out for the preparation of the monotetrabutylamonium ammonium salts of the guest acids according to literature procedure (M. Giffard, G. Mabon, N. Mercies, P. Molinié, T.P. Nguyen, A. Riou and M. Vautrin, *Synthetic Metals,* 1999**, 102**, 1766). Elemental analysis was performed for their characterization.

X-ray crystallography

Intensity data of all the compounds were collected with the Bruker SMART APEXII CCD area-detector diffractometer (Mo Kα radiation, λ = 0.7107Å) using the APEX2 software [18]. The low temperature data for complex **A** were collected using the Oxford Cryosystem Cobra low-temperature attachment (Both data were collected at room temperature). Data reductions were performed using SAINT [18]. Absorption corrections were performed using SADABS [17]. The structures were solved by direct methods. The non-hydrogen atoms were refined anisotropically. The Ortep23 figures were plotted using SHELXTL [17]**.**