Self-assembled blue-light emitting materials for their liquid crystalline and OLED applications: from a simple molecular design to supramolecular materials

Vinay S. Sharma[a*], Anuj S.Sharma[b], Nikhil K. Agarwal[a], Priyanka A. Shah[b], Pranav S. Shrivastav[b]

[a]: *Department of Chemistry, Faculty of Basic and Applied Science, Madhav University, Sirohi, Rajasthan, India.*

[b]: *Department of Chemistry, School of Science, Gujarat University, Ahmedabad, Gujarat, India.*

Email address of corresponding author[a]: vinaysharma3836@gmail.com

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1. Synthesis and Characterization

All chemicals were used without further purification and purchased from the available sources while the solvents were dried and purified in the usual manner. Melting points were taken on Opti-Melt (Automated melting point system). The FT-IR spectra were recorded as KBr pellet on Shimadzu in the range of 3800-600 cm^{-1}. Microanalysis was performed on Perkin-Elmer PE 2400 CHN analyser. The texture images were studied on a trinocular optical polarising microscope (POM) equipped with a heating stage. ^{1}H NMR spectra and ^{13}C NMR was recorded on a 400 MHz in Bruker Advance 400 in the range of 0.5 ppm-16 ppm using $CDCl_3$ solvent. Thermo gravimetric analysis (TGA) was performed using a Perkin Elmer-STA 6000 apparatus under high purity nitrogen. Mass Spectrometry was carried out using High Resolution Mass Spectrometer. The phase transition temperatures were measured using Shimadzu DSC-50 at heating and cooling rates of 10°C min^{-1}. The samples were heated from room temperature to 550˚C at 10˚C/min. X-ray diffraction (XRD) measurements were performed on a Rigaku-Ultima IV powder diffractometer equipped with a Cu kα source (λ = 1.5418 A˚ and 1.6 kW, X-ray tube with applied voltage and current values as 40 kV and 30 mA power) and also Philips X'PERT MPD. The absorption spectra were studied by using Jasco V-570 UV-Vis recording spectrophotometer with a variable wavelength between 200 and 800 nm. The fluorescence spectra were recorded on a Jasco FP-6500 spectrofluorometer. Cyclic voltammetry (CV) experiments were performed on a CH Instruments electrochemical workstation. The reference electrode was calibrated with the ferrocene/ferrocenium (Fc/Fc^{+}) redox couple (absolute energy level of – 4.80 eV to vacuum).

Preparation of 3, 4-dibutloxy acetanilide (1a)

3,4-dibutyloxy acetanilide (**1a**) was synthesized by refluxing the reaction mixture of 3,4-dihydroxy acetanilide (1 equiv.) with butyl bromide (2 equiv.) and anhydrous K_2CO_3 (2.4 equiv.) in dry acetone for 2 hr.[1] Yield 88 %, FT-IR (KBr) in cm^{-1}: 3319, 3068, 2970, 2866, 1684, 1240. ^1H NMR $CDCl_3$ (400 MHz): 0.88 (t, 3H), 1.46 (q, 4H), 1.71 (p, 4H), 4.06 (t, 4H), 7.24-7.03 (d, 4H, Ar), 6.71 (s, 1H, Ar), 8.41 (s, 1H). ^{13}C NMR: 166.8, 131.6, 143.3, 113.8, 105.4, 68.6, 31.6.

Preparation of 3, 4-dihexyloxy acetanilide (1b)

3,4-dibutyloxy acetanilide (**1a**) was synthesized by refluxing the reaction mixture of 3,4-dihydroxy acetanilide (1 equiv.) with hexyl bromide (2 equiv.) and anhydrous K_2CO_3 (2.4 equiv.) in dry acetone for 2 hr.[1] Yield 89 %, FT-IR (KBr) in cm^{-1}: 3321, 3068, 2970, 2861, 1664, 1234. ^1H NMR $CDCl_3$ (400 MHz): 0.88 (t, 3H), 1.46 (q, 4H), 1.71 (p, 4H), 1.26 (m, 4H), 4.06 (t, 4H), 7.24-7.03 (d, 4H, Ar), 6.71 (s, 1H, Ar), 8.43 (s, 1H). ^{13}C NMR: 166.7, 132.6, 141.3, 113.8, 105.4, 68.6, 31.6, 19.6.

Preparation of 3,4-decyloxy acetanilide (1c)

3,4-dibutyloxy acetanilide (**1a**) was synthesized by refluxing the reaction mixture of 3,4-dihydroxy acetanilide (1 equiv.) with decyl bromide (2 equiv.) and anhydrous K_2CO_3 (2.4 equiv.) in dry acetone for 2 hr.[1] Yield 84 %, FT-IR (KBr) in cm^{-1}: 3321, 3068, 2976, 2861, 1684, 1241. ^1H NMR $CDCl_3$ (400 MHz): 0.88 (t, 3H), 1.46 (q, 4H), 1.24 (m, 8H), 1.71 (p, 4H), 4.06 (t, 4H), 7.24-7.03 (d, 4H, Ar), 6.71 (s, 1H, Ar), 8.41 (s, 1H). ^{13}C NMR: 164.8, 130.6, 142.3, 113.8, 105.4, 68.6, 31.7.

Preparation of 3,4-dihexadecyloxy acetanilide (1d)

3,4-dibutyloxy acetanilide (**1a**) was synthesized by refluxing the reaction mixture of 3,4-dihydroxy acetanilide (1 equiv.) with hexadecyl bromide (2 equiv.) and anhydrous K_2CO_3 (2.4

equiv.) in dry acetone for 2 hr.[1] Yield 86 %, FT-IR (KBr) in cm^{-1}: 3321, 3040, 2973, 2864, 1654, 1231. ^{1}H NMR $CDCl_3$ (400 MHz): 0.88 (t, 3H), 1.46 (q, 4H), 1.71 (p, 4H), 1.24-1.26 (m, 16H), 4.06 (t, 4H), 7.24-7.03 (d, 4H, Ar), 6.71 (s, 1H, Ar), 8.41 (s, 1H). ^{13}C NMR: 166.8, 133.6, 143.3, 113.8, 104.4, 67.6, 31.4.

Preparation of *N*-(3, 4-dibutyloxy phenyl)-*N*-phenyl acetamide (2a)

N-(3, 4-dibutyloxy phenyl)-*N*-phenyl acetamide (**2a**) is synthesised from the mixture of 3,4-dibutyloxy acetanilide (**1a**, 1 equiv.) in presence of bromo benzene in presence of anhydrous K_2CO_3 and catalytic amount of CuI in nitrobenzene, the whole reaction mixture is reflux for 15 hr.[2] Yield: 76 %; FT-IR (KBr pellet) in cm-1: 3103, 2940, 2866, 1640, 1507, 1430, 1234, 713, 686, 563;^{1}H NMR ($CDCl_3$, 400 MHz): 0.88 (t, 3H), 1.46 (q, 4H), 1.71 (p, 4H), 4.06 (t, 4H), 7.24-7.03 (d, 4H, Ar), 6.71 (s, 1H, Ar), 7.47-7.54 (d, 4H). ^{13}C NMR: 167.6, 141.8, 133.5, 129.1, 126.5, 114.4, 77.4, 77.0, 68.7, 31.8, 22.6.

Preparation of *N*-(3, 4-dihexyloxy phenyl)-*N*-phenyl acetamide (2b)

N-(3, 4-dihexyloxy phenyl)-*N*-phenyl acetamide (**2b**) is synthesised from the mixture of 3,4-dibutyloxy acetanilide (**1b**, 1 equiv.) in presence of bromo benzene in presence of anhydrous K_2CO_3 and catalytic amount of CuI in nitrobenzene, the whole reaction mixture is reflux for 15 hr.[2] Yield: 72 %; FT-IR (KBr pellet) in cm-1: 3103, 2940, 2860, 1660, 1510, 1450, 1234, 713, 636, 561; ^{1}H NMR ($CDCl_3$, 400 MHz): 0.88 (t, 3H), 1.46 (q, 4H), 1.26 (m, 4H), 1.71 (p, 4H), 4.06 (t, 4H), 7.24-7.03 (d, 4H, Ar), 6.70 (s, 1H, Ar), 7.46 -7.54 (d, 4H). ^{13}C NMR: 167.6, 143.8, 133.5, 129.1, 126.5, 114.4, 77.4, 77.6, 68.7, 31.8, 22.1.

Preparation of *N*-(3, 4-didecyloxy phenyl)-*N*-phenyl acetamide (2c)

N-(3, 4-didecyloxy phenyl)-*N*-phenyl acetamide (**2c**) is synthesised from the mixture of 3,4-didecyloxy acetanilide (**1c**, 1 equiv.) in presence of bromo benzene in presence of anhydrous K_2CO_3 and catalytic amount of CuI in nitrobenzene, the whole reaction mixture is reflux for 15 hr.[2] Yield: 71 %; FT-IR (KBr pellet) in cm-1: 3101, 2950, 2860, 1660, 1510, 1440, 1230, 781, 691, 582; ^{1}H NMR $CDCl_3$ (400 MHz): 0.88 (t, 3H), 1.46 (q, 4H), 1.24 (m, 8H), 1.71 (p, 4H), 4.06 (t, 4H), 7.24-7.03 (d, 4H, Ar), 6.71 (s, 1H, Ar). ^{13}C NMR: 167.6, 141.8, 133.5, 129.1, 126.5, 114.4, 77.6, 77.3, 68.7, 31.8, 22.1.

Preparation of *N*-(3, 4-dihexadecyloxy phenyl)-*N*-phenyl acetamide (2d)

N-(3, 4-dihexadecyloxy phenyl)-*N*-phenyl acetamide (**2d**) is synthesised from the mixture of 3,4-dihexadecyloxy acetanilide (**1d**, 1 equiv.) in presence of bromo benzene in presence of anhydrous K_2CO_3 and catalytic amount of CuI in nitrobenzene, the whole reaction mixture is reflux for 15 hr.[2] Yield: 69 %; FT-IR (KBr pellet) in cm-1: 3103, 2950, 2843, 1640, 1542, 1450, 1234, 768, 636, 560; ^{1}H NMR $CDCl_3$ (400 MHz): 0.88 (t, 3H), 1.46 (q, 4H), 1.71 (p, 4H), 1.24-1.26 (m, 16H), 4.06 (t, 4H), 7.24-7.03 (d, 4H, Ar), 6.71 (s, 1H, Ar). ^{13}C NMR: 167.6, 141.8, 133.5, 129.3, 126.5, 114.8, 77.4, 77.1, 68.7, 31.8, 22.6.

(*E*)-*N*-(3,4-dibutyloxy)-3-(4-hydroxy phenyl)-*N*-phenyl acrylamide (3a)

Compound (**3a**) was prepared by the reported method in literature.[3] Yield: 64 %; IR (KBr pellet) in cm^{-1}: 3101, 2940, 2880, 1640, 1554, 1457, 1341, 1321, 1212, 869, 781, 637. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.49 (d, 4H, *J* = 6 Hz, Ar), 7.29 (d, 4H, *J* = 6 Hz, Ar), 7.47 (d, 4H, *J* = 8Hz, Ar), 6.34 (d, 1H, *J* = 15.1 Hz, -CH=CH-), 7.31 (d, 1H, *J* = 15.1 Hz, -CH=CH-), 1.81 (p, 4H, -OC_4H_9), 1.47 (q, 4H, -OC_4H_9), 0.90 (t, 4H, -OC_4H_9).^{13}C NMR: 159.3, 157.1, 144.6, 141.7, 131.7, 130.6, 129.5, 128.5, 121.4, 102.9, 77.1, 76.9, 68.7, 31.8, 14.6.

(*E*)-*N*-(3,4-dihexyloxy)-3-(4-hydroxy phenyl)-*N*-phenyl acrylamide (3b)

Compound (**3b**) was prepared by the reported method in literature.[3] Yield: 68 %; IR (KBr pellet) in cm^{-1}: 3142, 2943, 2880, 1670, 1544, 1457, 1354, 1320, 1232, 1143, 870, 776, 642. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.47 (d, 4H, *J* = 6 Hz, Ar), 7.28 (d, 4H, *J* = 6 Hz, Ar), 7.47 (d, 4H, *J* = 8Hz, Ar), 6.32 (d, 1H, *J* = 15.1 Hz, -CH=CH-), 7.36 (d, 1H, *J* = 15.1 Hz, -CH=CH-), 1.81 (p, 4H, $-OC_6H_{13}$), 1.47 (q, 4H, $-OC_6H_{13}$), 1.26 (m, 4H, $-OC_6H_{13}$), 0.90 (t, 4H, $-OC_6H_{13}$). ^{13}C NMR: 159.3, 157.6, 144.6, 141.7, 131.7, 130.6, 129.5, 128.5, 127.8, 121.4, 102.9, 77.6, 72.9, 68.7, 31.8, 14.3.

(*E*)-*N*-(3,4-didecyloxy)-3-(4-hydroxy phenyl)-*N*-phenyl acrylamide (3c)

Compound (**3c**) was prepared by the reported method in literature.[3] Yield: 71 %; IR (KBr pellet) in cm^{-1}: 3167, 2946, 2880, 1630, 1548, 1457, 1341, 1321, 1230, 1146, 870, 781, 536. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.43 (d, 4H, *J* = 6 Hz, Ar), 7.21 (d, 4H, *J* = 6 Hz, Ar), 7.47 (d, 4H, *J* = 8 Hz, Ar), 6.41 (d, 1H, *J* = 15.6 Hz, -CH=CH-), 7.36 (d, 1H, *J* = 15.1 Hz, -CH=CH-), 1.81 (p, 4H, $-OC_{10}H_{21}$), 1.47 (q, 4H, $-OC_{10}H_{21}$), 1.26 (m, 8H, $-OC_{10}H_{21}$), 0.90 (t, 4H, $-OC_{10}H_{21}$). ^{13}C NMR: 160.3, 157.1, 144.6, 141.7, 131.7, 130.6, 129.5, 128.5, 121.4, 102.9, 77.1, 76.5, 68.7, 31.8, 14.6.

(*Z*)-*N*-(3,4-dihexadecyloxy)-3-(4-hydroxy phenyl)-*N*-phenyl acrylamide (3d)

Compound (**3d**) was prepared by the reported method in literature.[3] Yield: 62 %; IR (KBr pellet) in cm^{-1}: 3142, 2940, 2880, 1650, 1540, 1457, 1341, 1321, 1240, 1140, 870, 780, 637. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.43 (d, 4H, *J* = 6 Hz, Ar), 7.21 (d, 4H, *J* = 6 Hz, Ar), 7.47 (d, 4H, *J* = 8 Hz, Ar), 6.41 (d, 1H, *J* = 15.6 Hz, -CH=CH-), 7.36 (d, 1H, *J* = 15.1 Hz, -CH=CH-), 1.76 (p, 4H, $-OC_{16}H_{33}$), 1.43 (q, 4H, $-OC_{16}H_{33}$), 1.24-1.26 (m, 10H, $-OC_{16}H_{33}$), 0.90 (t, 4H, $-OC_{16}H_{33}$). ^{13}C NMR: 159.3, 157.1, 144.6, 141.7, 131.7, 130.6, 129.5, 128.5, 121.9, 110.4, 77.6, 76.9, 68.7, 31.6, 14.6.

Preparation of p-*tert*-butyl calix[4]arene (4)

p-tert-butyl calix[4]arene (**4**) was synthesized by reported method in the literature[4], white precipitates, yield 87%. Elemental analysis: $C_{44}H_{56}O_4$: Calcu: C, 80.44; H, 8.70; O, 9.80 %, Found: C, 80.14; H, 8.62; O, 9.72 %. ^{1}H NMR: (400 MHz, $CDCl_3$): 1.18 (s, 36H, t-butyl), 3.61 (d, J = 12.0Hz, 4H, Ar-CH_2-Ar), 4.16 (d, J = 12.0Hz, 4H, Ar-CH_2-Ar), 7.08 (s, 8H, Ar-H), 9.78 (s, 4H, Ar-OH); ^{13}C NMR: 149.1, 126.2, 126.1, 34.2, 31.4, 32.6.

Preparation of p-*tert*-butyl calix[4]arene di-propionic acid (5)

p-*tert*-butyl calix[4]arene bi-propionic acid (**5**) is prepared by the condensation reaction of compound **(4)** with bromo propionic acid in acetonitrile with presence of anhydrous K_2CO_3 as a base.[5] White precipitates, yield 76%. ^{1}H NMR ($CDCl_3$, 400 MHz): 1.31 (s, 36H), 2.53 (t, 2H), 3.64 (s, 2H), 4.17 (s, 2H, J = 6.0Hz), 6.84 (s, 4H), 7.64 (s, 4H, J = 8.0Hz), 8.42 (s, 2H), 4.61 (s, 2H, -OH), 10.5 (s, 2H, -COOH); ^{13}C NMR: 160.6, 156.6, 144.7, 136.4, 122.5, 77.4, 77.4, 67.1, 35.2.

Preparation of p-*tert*-butyl calix[4]arene di-butyl di-propionic acid (6)

p-*tert*-butyl calix[4]arene di-butyl di-propionic acid (**6**) is prepared by the condensation reaction of compound **(5)** with butyl bromide in acetonitrile with presence of anhydrous K_2CO_3 as a base.[5] Light yellow precipitates, yield 69 %. ^{1}H NMR ($CDCl_3$, 400 MHz): 1.34 (s, 36H), 2.53 (t, 2H), 3.67 (s, 2H), 4.17 (s, 2H, J = 6.0Hz), 6.84 (s, 4H), 7.64 (s, 4H, J = 8.0Hz), 8.42 (s, 2H), 4.61 (s, 2H, -OH), 10.5 (s, 2H, -COOH), 4.08 (t, 4H), 1.71 (p, 4H), 1.41 (q, 4H), 0.88 (t, 4H); ^{13}C NMR: 171.1, 166.7, 160.6, 156.6, 141.7, 136.4, 125.6, 122.5, 77.4, 77.4, 67.1, 35.2.

Preparation of p-*tert*-butyl calix[4]arene chalcone amine di-butyloxy derivatives (7a)

The compound has been prepared by the refluxing the reaction of compound (**6**) (0.0015 mol.) and compound (**3a-3d**) (0.0030 mol.), EDC.HCl (0.0030 mol.) in DCM (30 ml) with presence of catalytic amount of DMAP for 24 hr. The reaction mixture was filter and evaporates to dryness to get final solid material. Evaporation of the solvent by using rota evaporator and purification of the residue by using column chromatography followed by methanol-chloroform system (1:4).[6]

(7a): Yield 71 %, FT-IR (KBr) in cm^{-1}: 2990, 2810, 1740, 1610, 1522, 1440, 1320, 1230, 1130, 1120, 986, 886. ^{1}H NMR ($CDCl_3$, 400 MHz): 0.90 (t, *J* = 6.3 Hz, 18H, $-OC_4H_9$), 1.31 (s, 36H, t-butyl group), 1.47 (sext, 12H, $-OC_4H_9$), 1.71 (p, 12H, $-OC_4H_9$), 4.08 (t, 12H, $-OC_4H_9$), 4.27 (s, 4H, $-CH_2-$), 3.74 (s, 4H, $-CH_2-$), 6.87 (d, *J* = 6.7 Hz, 4H, Ar), 7.54 (s, 2H, Ar), 7.47 (d, *J* = 6.3 Hz, 8H, Ar), 6.91 (d, 8H, Ar), 6.74 (d, *J* = 8.7 Hz, 8H, Ar), 7.02 (s, 2H, Ar), 6.64 (s, 2H, Ar), 6.64 (d, 1H, *J* = 15.6 Hz, -CH=CH-), 7.61 (d, 1H, 15.1 Hz, -CH=CH-). ^{13}C NMR: 170.1, 169.2, 160.4, 159.4, 147.8, 144.7, 141.7, 128.6, 129.4, 135.8, 131.6, 129.7, 127.6, 122.2, 117.7, 105.1, 71.3, 69.1, 68.7, 35.2, 34.8, 31.8, 19.6, 14.2. MALDI Tof MS for compound **7a** (M+1) Calculated: 1788.0546 Found 1789.2321.

Preparation of p-*tert*-butyl calix[4]arene chalcone amine di-hexyloxy derivatives (7b)

(7b): Yield 64 %, FT-IR (KBr) in cm^{-1}: 2990, 2883, 1730, 1660, 1520, 1424, 1320, 1120, 981, 886. ^{1}H NMR ($CDCl_3$, 400 MHz): 0.90 (t, *J* = 6.3 Hz, 18H, $-OC_4H_9$), 1.26 (m, 12H), 1.31 (s, 36H, t-butyl group), 1.47 (sext, 12H, $-OC_4H_9$), 1.71 (p, 12H, $-OC_4H_9$), 4.08 (t, 12H, $-OC_4H_9$), 4.27 (s, 4H, $-CH_2-$), 3.74 (s, 4H, $-CH_2-$), 6.87 (d, *J* = 6.7 Hz, 4H, Ar), 7.54 (s, 2H, Ar), 7.47 (d, *J* = 6.3 Hz, 8H, Ar), 6.91 (d, 8H, Ar), 6.74 (d, *J* = 8.7 Hz, 8H, Ar), 7.02 (s, 2H, Ar), 6.64 (s, 2H, Ar), 6.64 (d, 1H, *J* = 15.6 Hz, -CH=CH-), 7.61 (d, 1H, 15.1 Hz, -CH=CH-). ^{13}C NMR: 170.1,

169.2, 160.4, 159.4, 147.8, 144.7, 141.7, 128.6, 129.4, 135.8, 131.6, 129.7, 127.6, 122.2, 117.7, 105.1, 71.3, 69.1, 68.7, 35.2, 34.8, 31.8, 19.6, 14.2..MALDI Tof MS for compound **7b** (M+1) Calculated: 1900.1706 Found 1901.5421.

Preparation of p-*tert*-butyl calix[4]arene chalcone amine di-decyloxy derivatives (7c)

(7c): Yield 73 %, FT-IR (KBr) in cm^{-1}: 2990, 2880, 1730, 1630, 1520, 1440, 1320, 1140, 1120, 981, 886. ^{1}H NMR ($CDCl_3$, 400 MHz): 0.90 (t, *J* = 6.3 Hz, 18H, $-OC_4H_9$), 1.26 (m, 16H), 1.31 (s, 36H, t-butyl group), 1.47 (sext, 12H, $-OC_4H_9$), 1.71 (p, 12H, $-OC_4H_9$), 4.08 (t, 12H, $-OC_4H_9$), 4.27 (s, 4H, $-CH_2-$), 3.74 (s, 4H, $-CH_2-$), 6.87 (d, *J* = 6.7 Hz, 4H, Ar), 7.54 (s, 2H, Ar), 7.47 (d, *J* = 6.3 Hz, 8H, Ar), 6.91 (d, 8H, Ar), 6.74 (d, *J* = 8.7 Hz, 8H, Ar), 7.02 (s, 2H, Ar), 6.64 (s, 2H, Ar), 6.64 (d, 1H, *J* = 15.6 Hz, -CH=CH-), 7.61 (d, 1H, 15.1 Hz, -CH=CH-). ^{13}C NMR: 170.1, 169.2, 160.4, 159.4, 147.8, 144.7, 141.7, 128.6, 129.4, 135.8, 131.6, 129.7, 127.6, 122.2, 117.7, 105.1, 71.3, 69.1, 68.7, 35.2, 34.8, 31.8, 19.6, 14.2. MALDI Tof MS for compound **7c** (M+1) Calculated: 2124.4360 Found 2125.0164.

Preparation of p-*tert*-butyl calix[4]arene chalcone amine di-hexadecyloxy derivatives (7d)

(7d): Yield 69 %, FT-IR (KBr) in cm^{-1}: 2890, 2880, 1730, 1620, 1520, 1441, 1320, 1240, 1140, 1120, 981, 780. ^{1}H NMR ($CDCl_3$, 400 MHz): 0.90 (t, *J* = 6.3 Hz, 18H, $-OC_4H_9$), 1.26 (m, 26H), 1.31 (s, 36H, t-butyl group), 1.47 (sext, 12H, $-OC_4H_9$), 1.71 (p, 12H, $-OC_4H_9$), 4.08 (t, 12H, $-OC_4H_9$), 4.27 (s, 4H, $-CH_2-$), 3.74 (s, 4H, $-CH_2-$), 6.87 (d, *J* = 6.7 Hz, 4H, Ar), 7.54 (s, 2H, Ar), 7.47 (d, *J* = 6.3 Hz, 8H, Ar), 6.91 (d, 8H, Ar), 6.74 (d, *J* = 8.7 Hz, 8H, Ar), 7.02 (s, 2H, Ar), 6.64 (s, 2H, Ar), 6.64 (d, 1H, *J* = 15.6 Hz, -CH=CH-), 7.61 (d, 1H, 15.1 Hz, -CH=CH-). ^{13}C NMR: 170.1, 169.2, 160.4, 159.4, 147.8, 144.7, 141.7, 128.6, 129.4, 135.8, 131.6, 129.7, 127.6, 122.2, 117.7, 105.1, 71.3, 69.1, 68.7, 35.2, 34.8, 31.8, 19.6, 14.2. MALDI Tof MS for compound **7d** (M+1) Calculated: 2460.8016 Found 2461.8323.

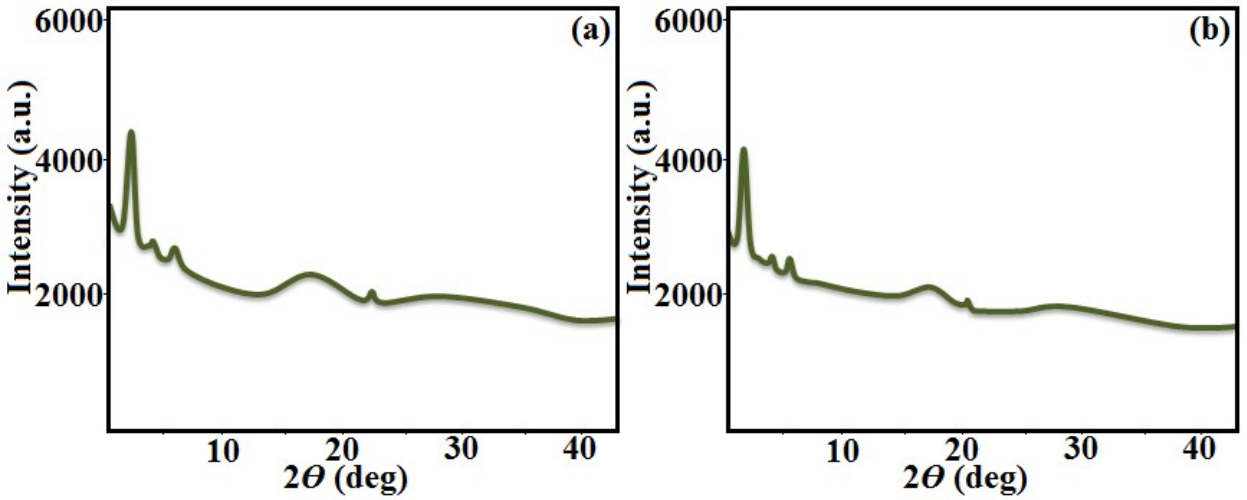

Figure S1. XRD profiles depicting the intensity against the 2Θ obtained for the Col_h phase of compound **7c** at 87.0 ˚C (a); Col_h phase of compound **7d** at 1.0 ˚C (b) on cooling from isotropic temperature; the insert shows the image pattern obtained.

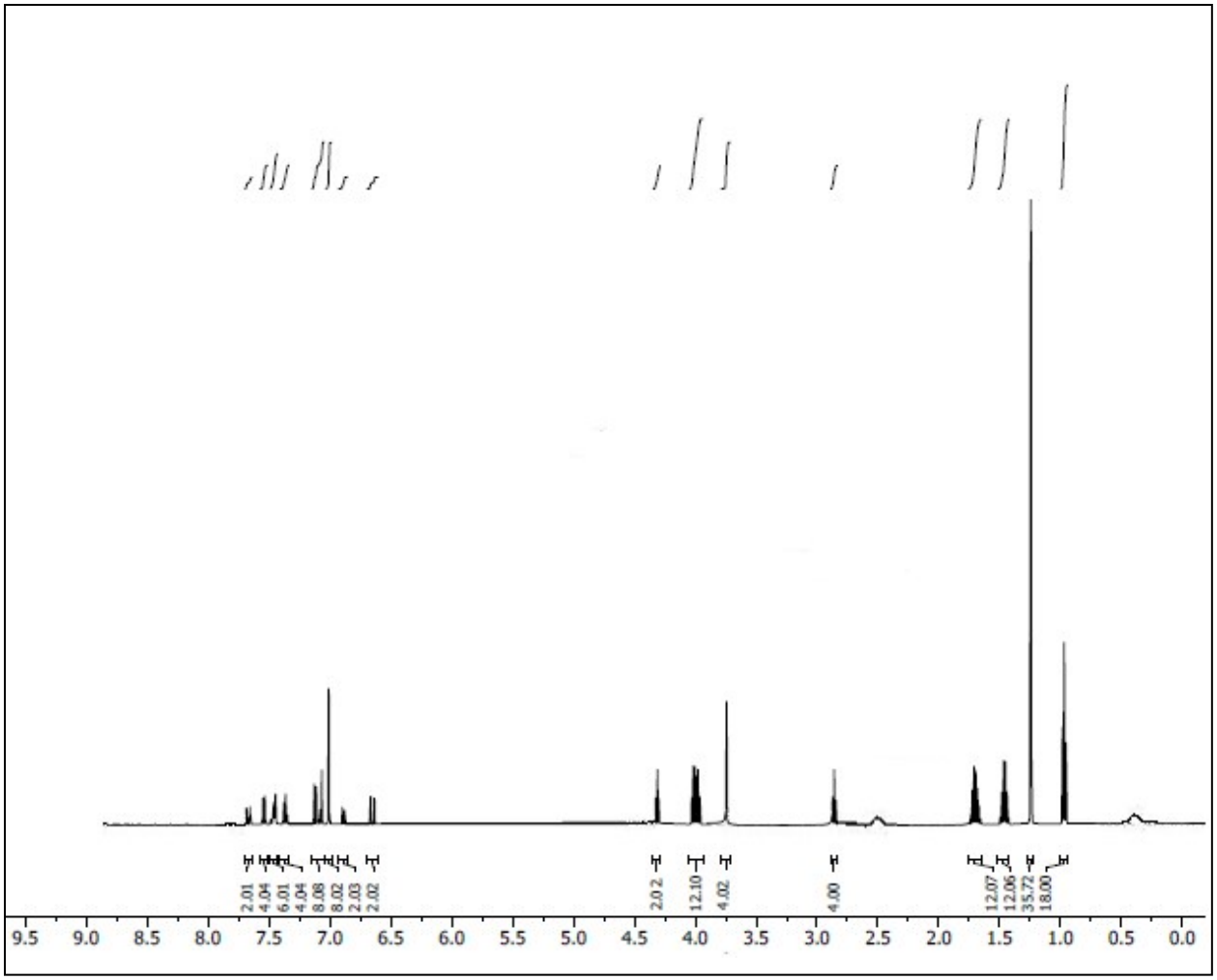

Figure S_2. ^{1}H NMR of compound **7a**

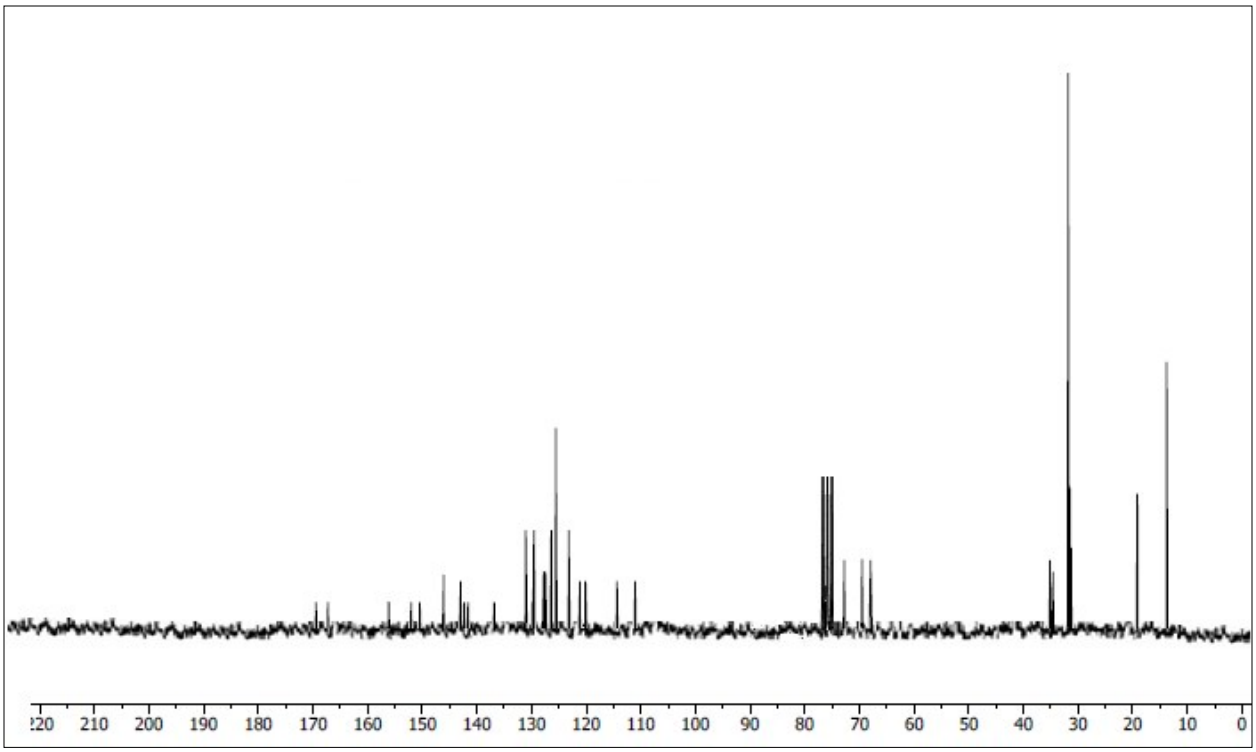

Figure S_3. ^{13}C NMR of compound **7a**

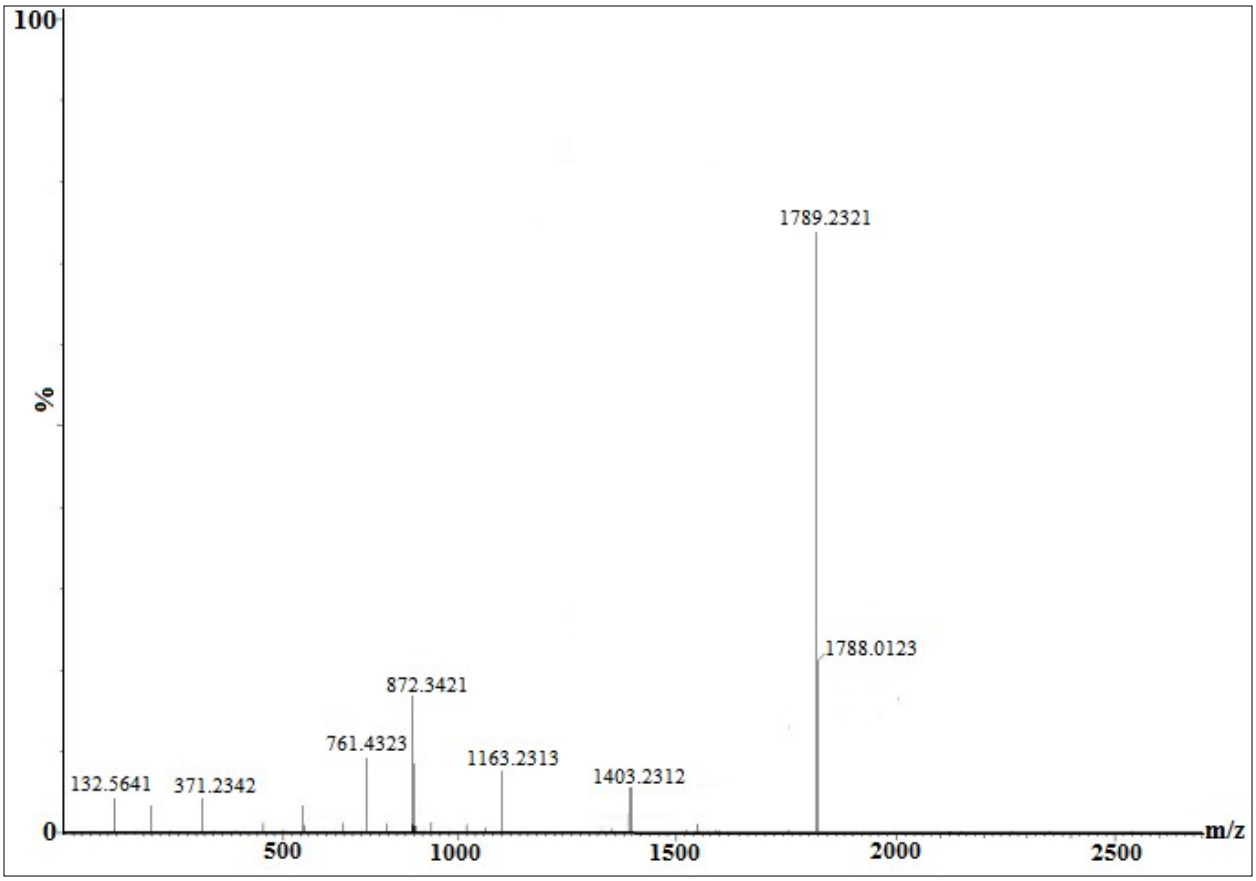

Figure S4. HRMS of compound **7a**

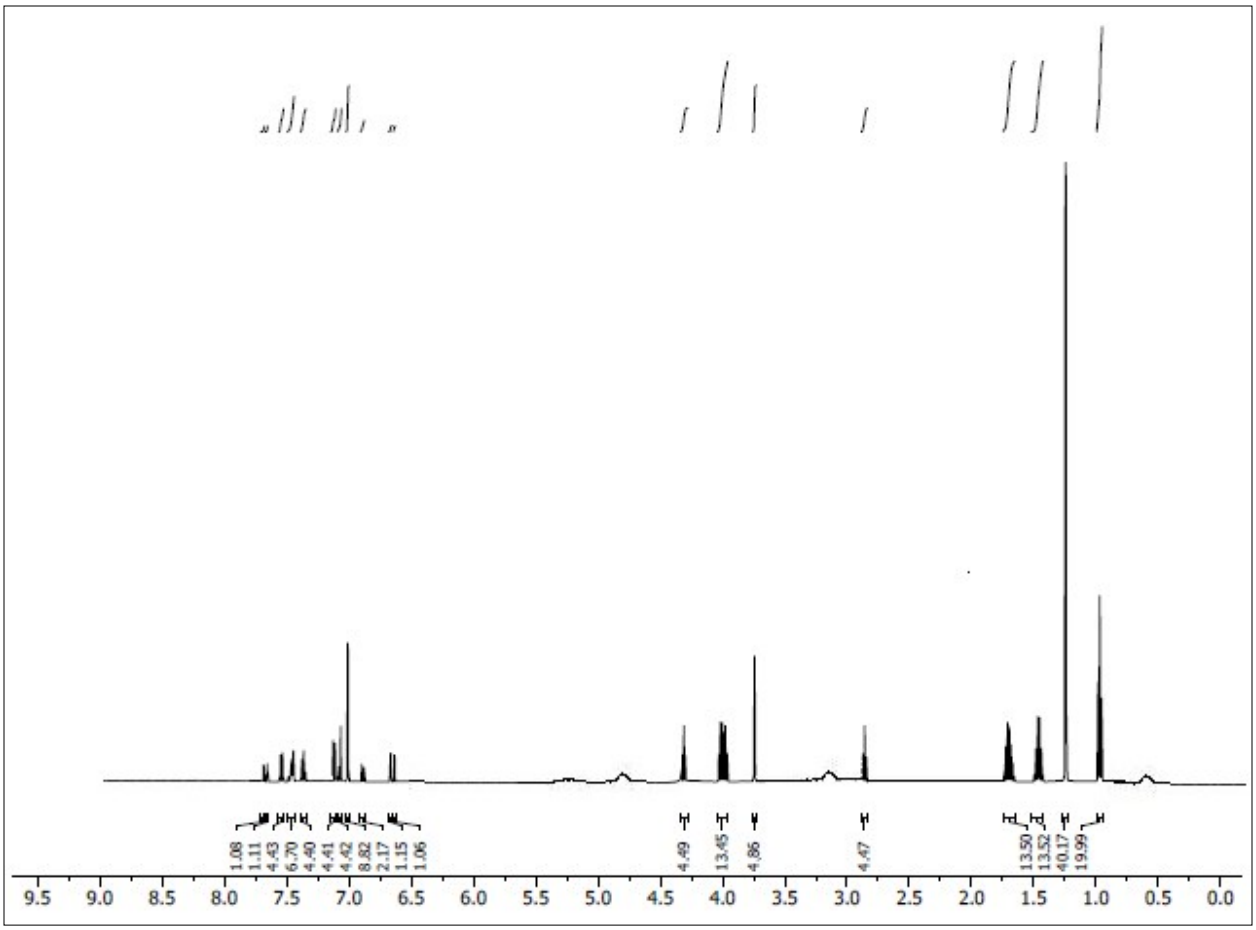

Figure S_5. ^{1}H NMR of compound **7b**

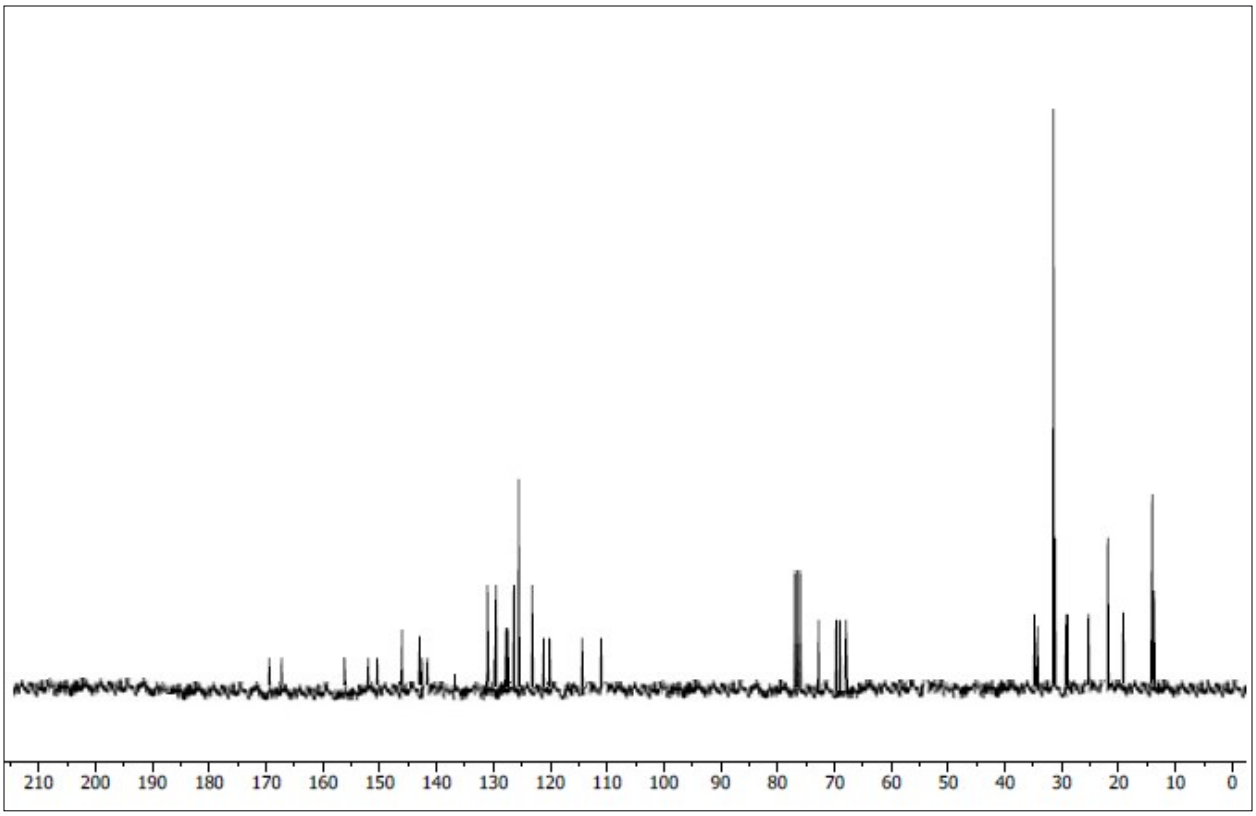

Figure S_6. ^{13}C NMR of compound **7b**

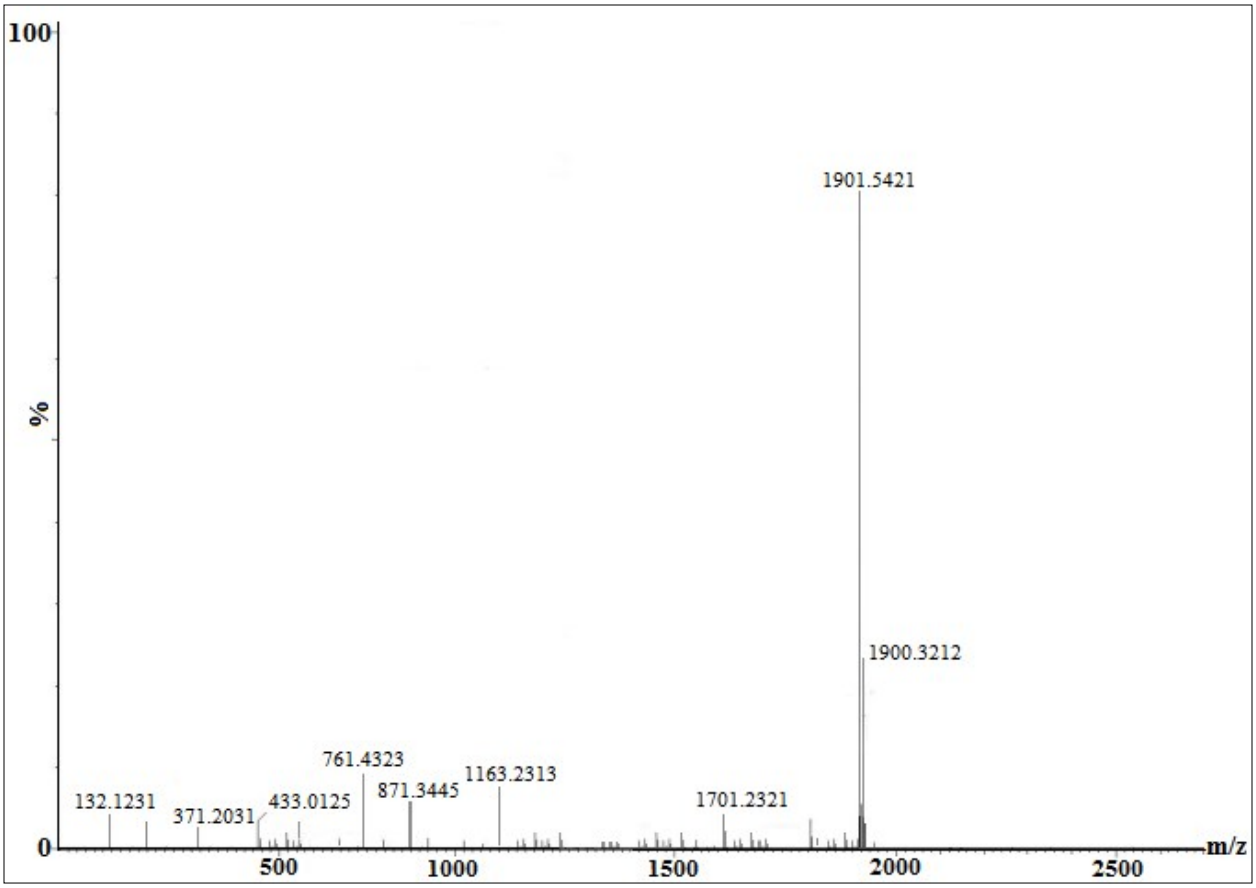

Figure S$_7$. HRMS of compound **7b**

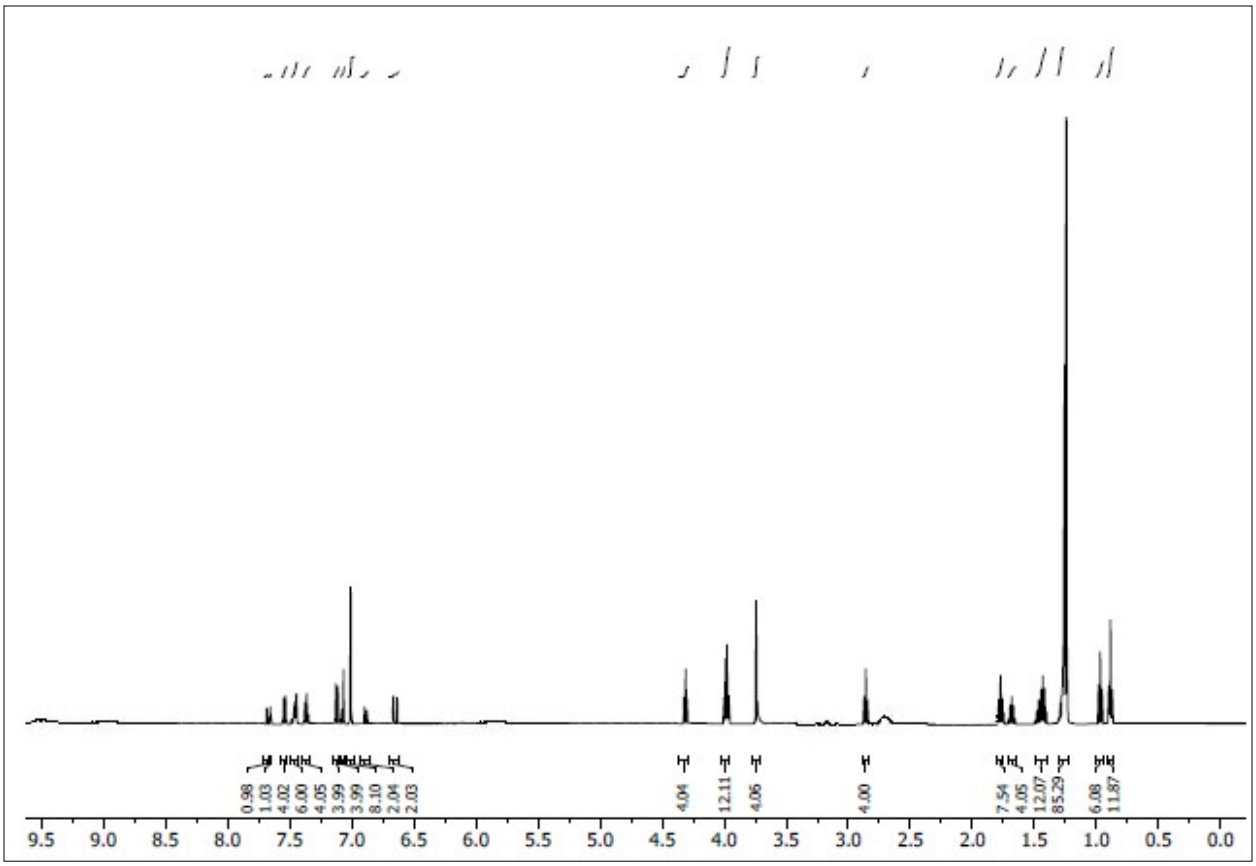

Figure S_8. ^{1}H NMR of compound **7c**

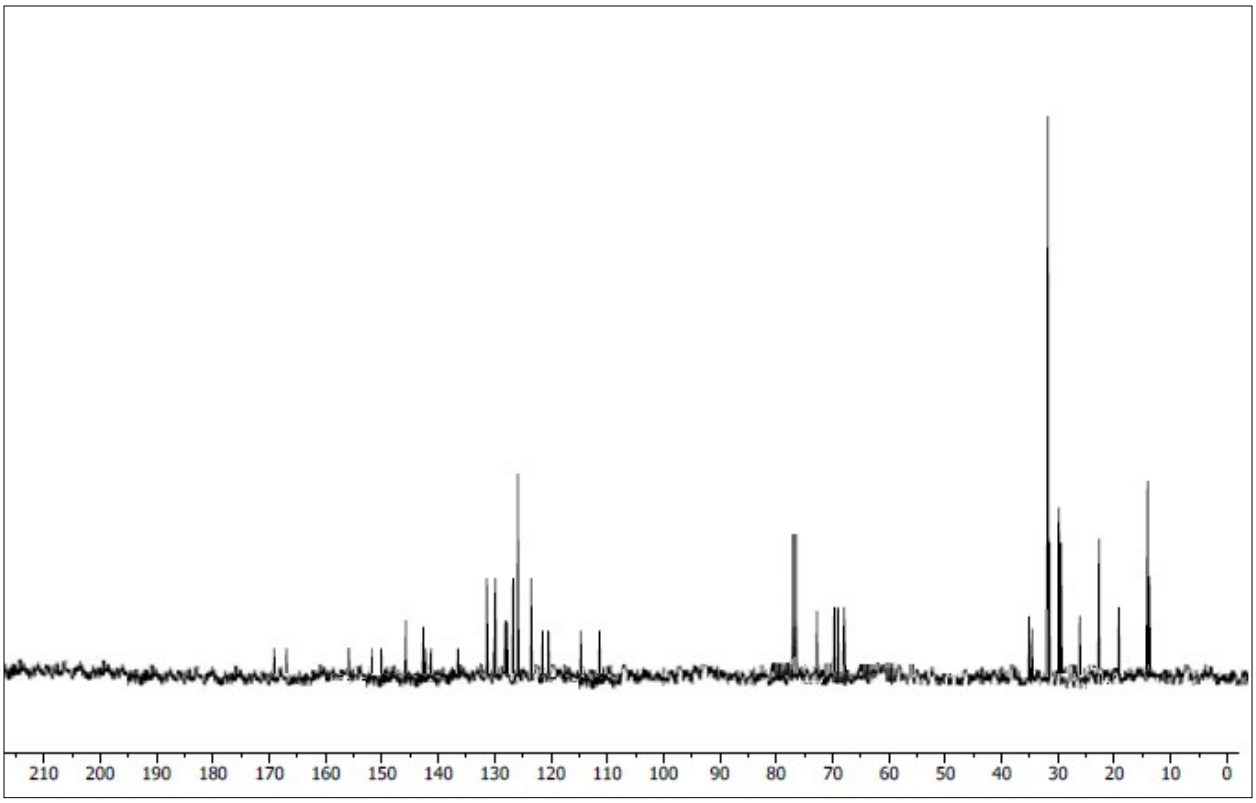

Figure S9. ^{13}C NMR of compound **7c**

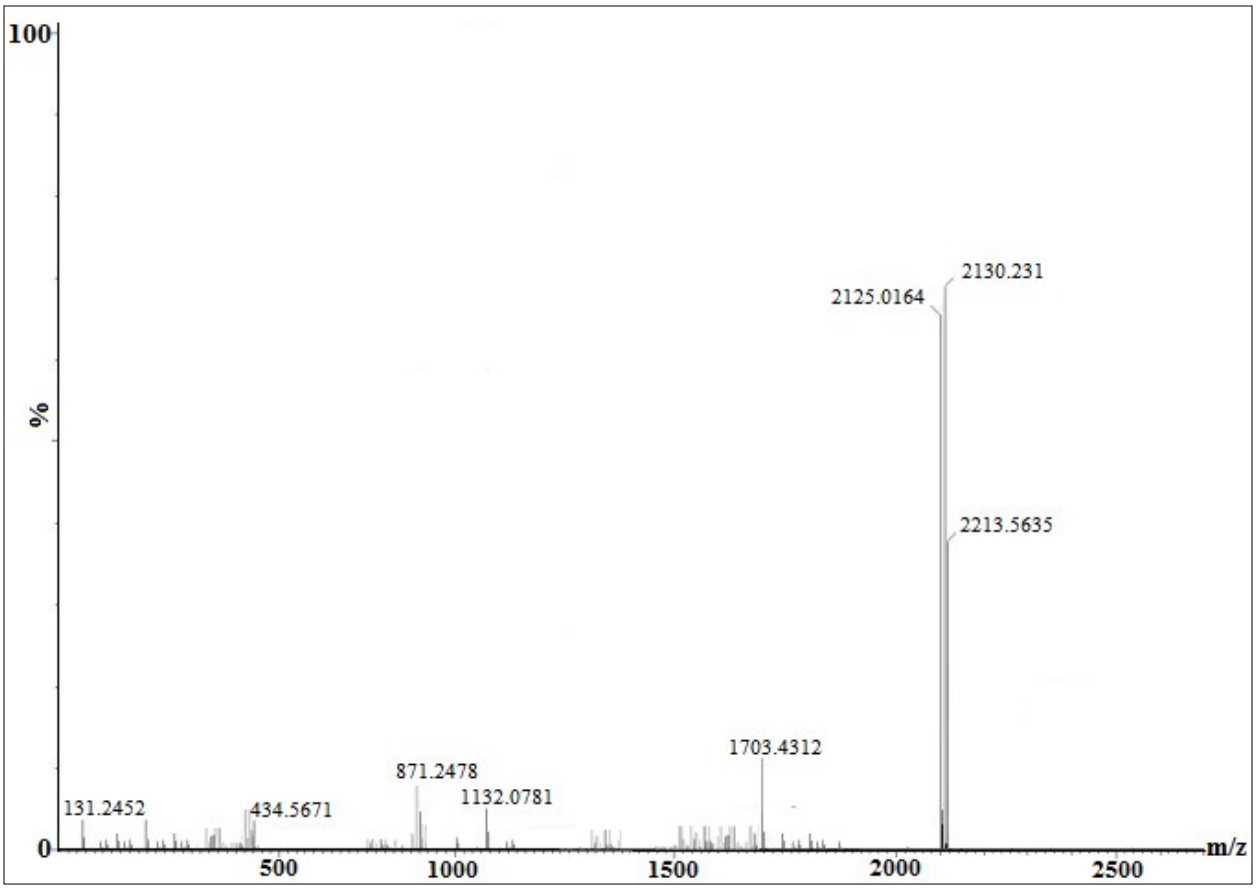

Figure S_{10}. HRMS of compound **7c**

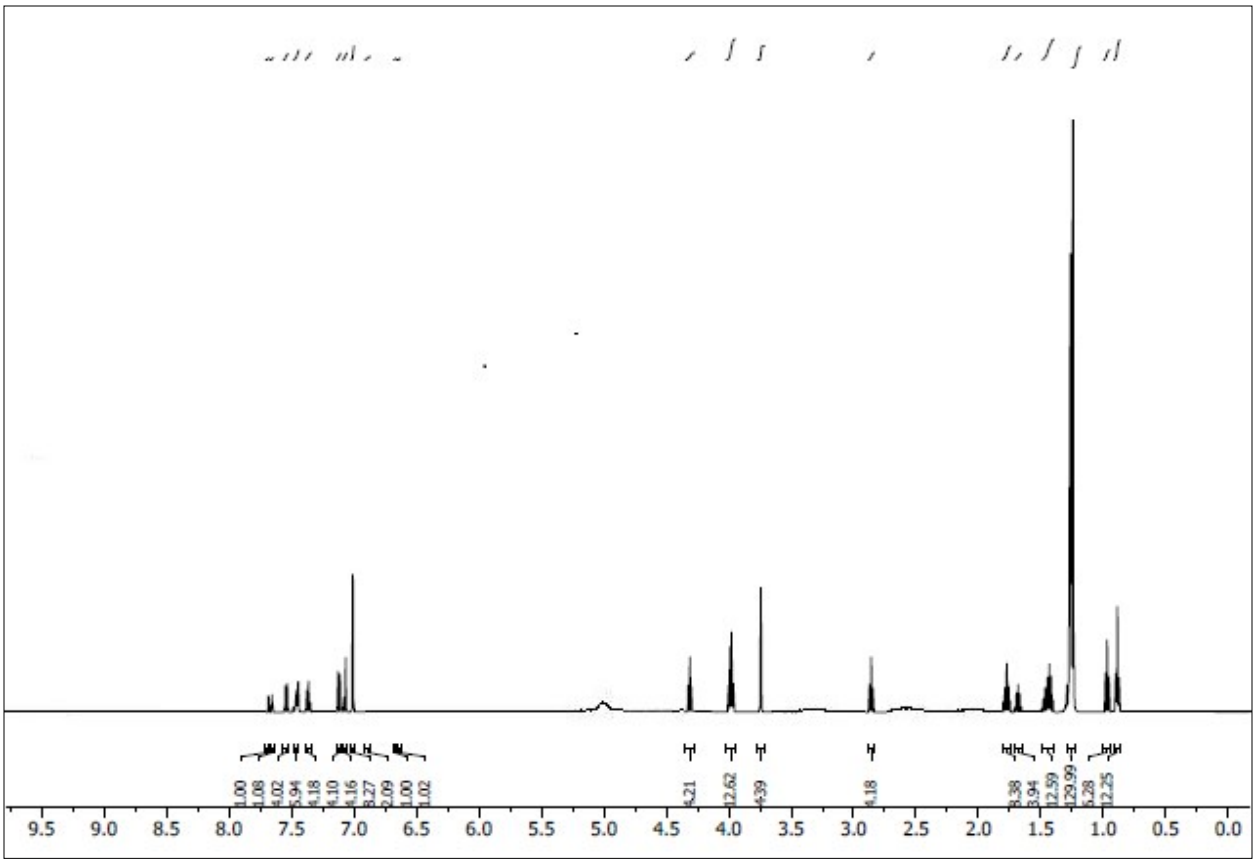

Figure S_{11}. ^{1}H NMR of compound **7d**

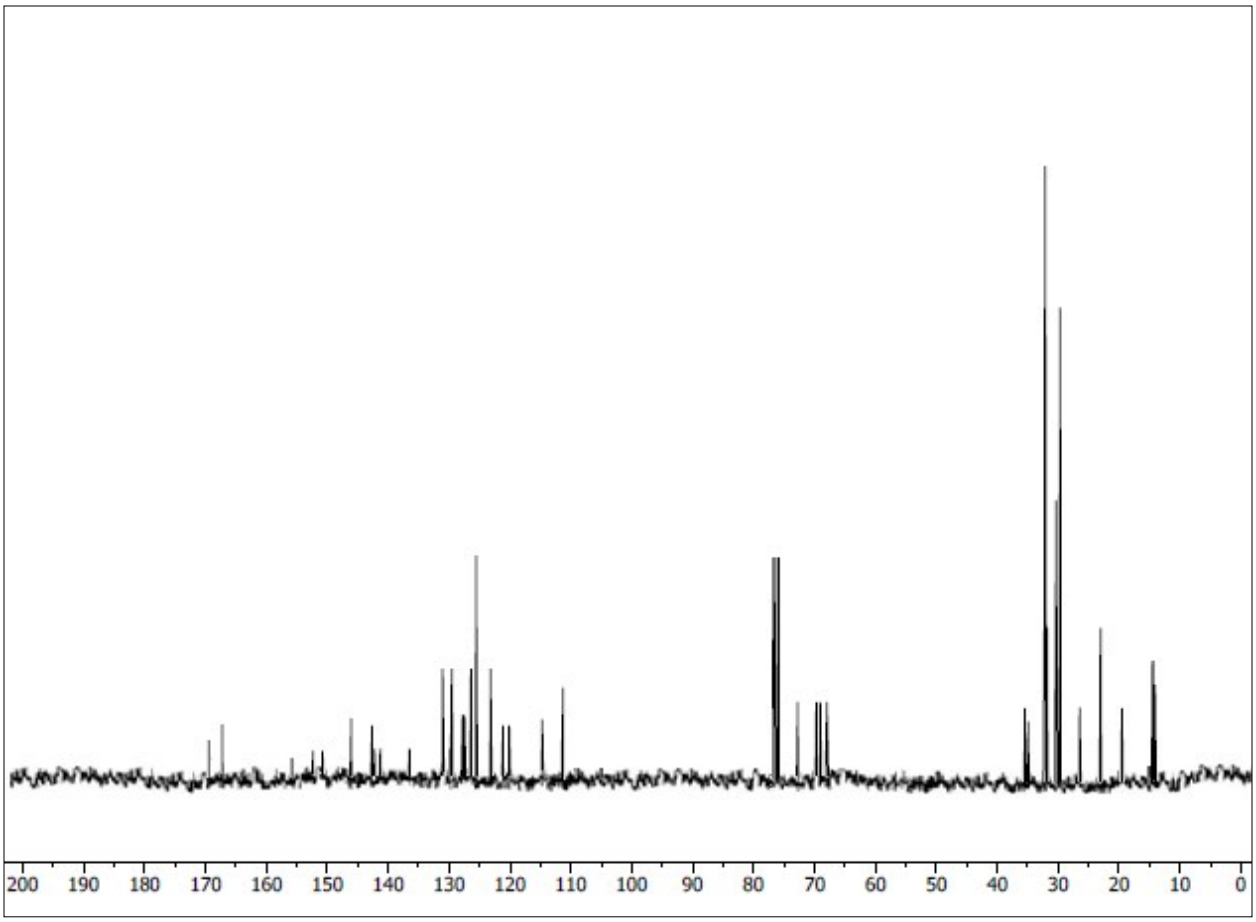

Figure S_{12}. ^{13}C NMR of compound **7d**

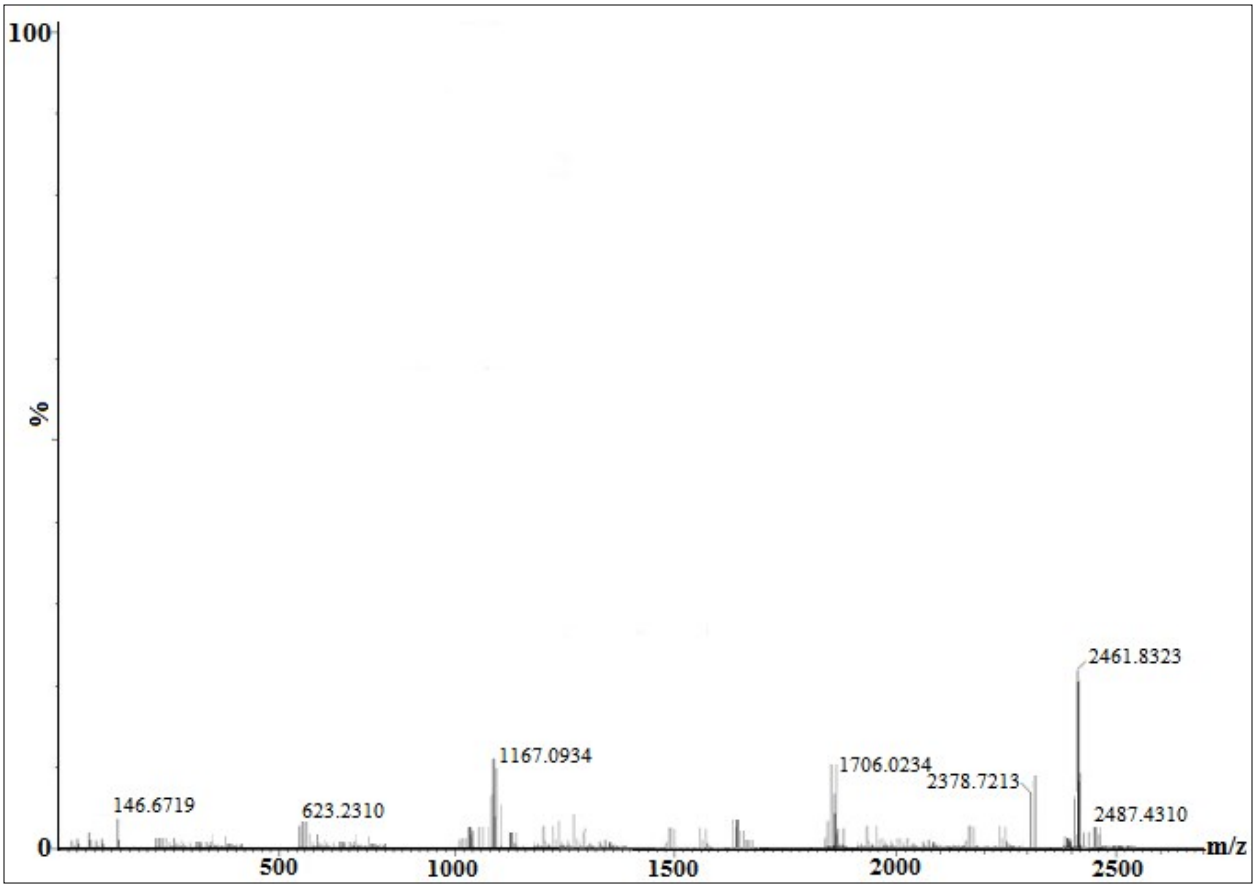

Figure S_{13}. HRMS of compound **7d**

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