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Supporting information

for

2-(4-Hydroxyphenyl)benzothiazole Dicarboxylate Ester TACN Chelators for ^{64}Cu PET imaging in Alzheimer's Disease

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I. Experimental procedures

General Methods. All reagents were purchased from commercial sources and used as received unless stated otherwise. 1,4,7-triazacyclononane (TACN) was synthesized according to reported procedures.[1] Solvents were purified prior to use by passing through a column of activated alumina using an MBRAUN SPS. All solutions and buffers were prepared using metal-free Millipore water that was treated with Chelex overnight and filtered through a 0.22 μm nylon filter. UV−visible spectra were recorded on a Varian Cary 50 Bio spectrophotometer and are reported as λ_{max}, nm (ε, M^{-1} cm^{-1}). EPR spectra were recorded on a Bruker 10" EMXPlus X-band Continuous Wave EPR spectrometer at 77 K. EPR spectra simulation and analysis were performed using Bruker WINEPR SimFonia program, version 1.25. ESI-MS experiments were performed by the Mass Spectrometry Lab at UIUC using a Waters Q-TOF Ultima ESI mass spectrometer with an electron spray ionization source.

Amyloid β Peptide Experiments. $A\beta_{42}$ powder was prepared by dissolving commercial Aβ peptide (AnaSpec) in ammonia hydroxide solution (1%, v/v). The solution was then aliquoted out and lyophilized overnight. The resulting aliquoted powder was stored at –80 °C. $A\beta_{42}$ monomers were generated by dissolving Aβ42 powder in 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP, 1 mM) and incubating for 1 h at room temperature. The solution was then evaporated overnight and dried by vacuum centrifuge for monomeric films. $A\beta_{42}$ fibrils were generated by dissolving monomeric $A\beta_{42}$ films in DMSO, diluting into PBS buffer (pH 7.4), and incubating for 24 h at 37 °C with continuous agitation (final DMSO concentration was < 2%).

Direct binding constant measurements. All fluorescence measurements were performed using a SpectraMax M2e plate reader (Molecular Devices). To PBS solutions (100 μL) of $A\beta_{42}$ species prepared as above (10 μM), various amount of **YW-13** stock solution was added to the solution. For **YW-13**-$A\beta_{42}$ solution, fluorescence intensities were measured with excitation wavelength at 330 nm and emission wavelength at 450 nm.

Histological Staining of 5xFAD Mouse Brain Sections. Eleven-month-old 5xFAD mice brain sections were blocked with bovine serum albumin (2% BSA in PBS, pH 7.4, 10 min) and covered with a PBS solution of compound for 30 min, then Congo Red (2 μM) for 30 min. The sections were treated with BSA again (4 min) to remove any compound non-specifically bound to the tissue.

Finally, the sections were washed with PBS (3 x 2 min), DI water (2 min), and mounted with non-fluorescent mounting media. For antibody staining, six-month old brain sections were incubated with AF594-conjugated anti-Aβ antibody (AF594-HJ3.4 antibody)[2, 3] solution (1µg/ml) at room temperature for 1 h instead of Congo Red. The brain sections were then washed with PBS (3 x 2 min) and mounted with mounting media. The stained brain sections were imaged using a Zeiss LSM 7010 confocal fluorescent microscope and Invitrogen EVOS FL Auto 2 Imaging System (Thermo Fisher, USA). Excitation and emission wavelengths of DAPI channel and Texas Red channel are λ_{ex} = 357/44 nm, λ_{em}= 447/60 nm and λ_{ex} = 585/29 nm, λ_{em}= 628/32 nm, respectively. Colocalization analysis and determination of the Pearson's correlation coefficient was performed with the imaging software Fiji (ImageJ 1.52p).

Acidity and Stability Constants Determination. UV–Vis pH titrations were employed for the determination of acidity constants of BFCs and stability constants with Cu(II). For acidity constants, solutions of ligands (20 µM, 0.1 M NaCl, pH 3) were titrated with small aliquots of 0.1 M NaOH at room temperature under a steady moist flow of N_2. At least 30 UV-Vis spectra were collected in the pH 3-11 range. DMSO stock solutions (10 mM) were diluted in MeOH-water mixture in which MeOH did not exceed 1% (v:v). Similarly, stability constants were determined by titrating solutions of ligands and 1 equiv of $Cu(ClO_4)_2{\cdot}6H_2O$ with small aliquots of 0.1 M NaOH at room temperature. At least 30 UV-Vis spectra were collected in the pH 3-11 range. The acidity and stability constants were calculated using the HypSpec computer program (Protonic Software, UK).[4] Speciation plots of the compounds and their metal complexes were calculated using the program HySS2009 (Protonic Software, UK).[5]

Job's plots for solution stoichiometry determination. To determine the ligand:Cu stoichiometry for the benzothiazole-diester series of ligands, a stock solution (500µM) of and $CuCl_2{\cdot}H_2O$ (500µM) were prepared in DMSO and spectra were measured using a Cary Bio UV-Vis instrument. Solutions containing different ratios of ligand and Cu ions were recorded from 0 to 100 mol % Cu(II) (total concentration = 25 µM). Appropriate amounts of the **YW-13** stock solution were diluted into 500 µL PBS (pH=7.4) buffer and allowed to equilibrate for 5 minutes before recording the UV-vis spectra.

Radiolabeling. ^{64}Cu was produced by a (p,n) reaction on enriched ^{64}Ni on a TR-19 biomedical cyclotron (Cyclotron Corporation, Berkeley, CA) at Mallinckrodt Institute of Radiology,

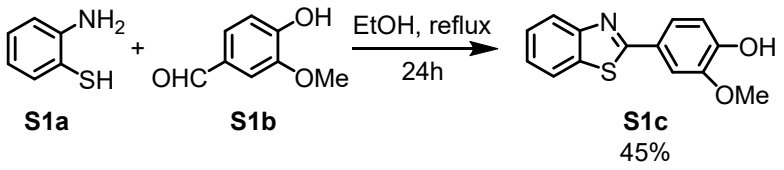
Washington University School of Medicine, and purified with an automated system using standard procedures.[6, 7] A stock solution of $^{64}CuCl_2$ was diluted with a 10-fold excess of 0.1 M ammonium acetate (NH_4OAc) buffer (pH 5.5). Typical labeling of compounds was achieved by adding 20 μL of compound (1 mM) solutions in DMSO into 7.4 MBq (200 μCi) of $^{64}CuCl_2$ in 100 μL of 0.1 M NH_4OAc (pH 5.5). The reactions were incubated on a thermomixer with 1000 rpm agitation at 45 ℃ for 1h. Radiolabeled compounds were analyzed by high-performance HPLC, with water (0.1% TFA) and acetonitrile (0.1% TFA) mobile phase with a gradient of 0–100% acetonitrile over 11 min with a flow rate of 1 mL/min. All ^{64}Cu-labeled complexes were obtained in high radiochemical yield (> 95%) and therefore used without further purification.

Lipophilicity Studies. Ten replicate Eppendorf tubes of 1:1 (v/v) n-octanol and PBS 1X were prepared (500 μL each). The ^{64}Cu-labeled complexes (0.37 MBq, 10 μCi) were added to each tube, vortexed and incubated in a thermomixer at 1000 rpm for 1 h. After 1 h, the solution was kept without shaking for 30 minutes to allow for the separation of the two layers. Aliquots (100 μL) from the aqueous and the n-octanol layers were removed and counted separately in an automated gamma counter. *Log D* values were obtained as the logarithms of the ratio of (activity detected in n-octanol)/(activity detected in aqueous layer). The overall average was recorded as the final *log D* value for each compound. High *log D* (1-2.5) is desired as it indicates the ability of radiolabeled products in crossing the BBB and reaching concentrations for imaging purposes in the brain.[8]

***Ex vivo* Autoradiography Studies.** Brain sections of eleven-month-old 5xFAD transgenic mice and aged-matched wild type (WT) mice were obtained as described previously and immersed into a cryo-protectant solution. These sections were sorted and carefully removed using phosphate buffer in saline (PBS) to a 12-well plate. Each brain section was washed with 100% PBS three times, and ~0.925 MBq (25 μCi) of ^{64}Cu-labeled BFC in 2.5 mL PBS was added to completely cover the brain section and incubate for 1 h at room temperature in a shielded bunker. For blocking studies, 2-(4-hydroxyphenol)benzothiazole (B_1, Figure S15) was added to evaluate the specific binding of radiolabeled compounds.[9] After the incubation, brain sections (WT, 5xFAD, 5xFAD with blocking) were washed once with 1:1 (v/v) ethanol:PBS and then twice with PBS for 10 minutes of each washing cycle. Brain sections were removed, mounted onto microscopic slides and briefly air-dried. The imaging slides were then mounted onto phosphor imaging screen plate (GE Healthcare Life Sciences), and exposed overnight at -20 °C. The plates were scanned using a

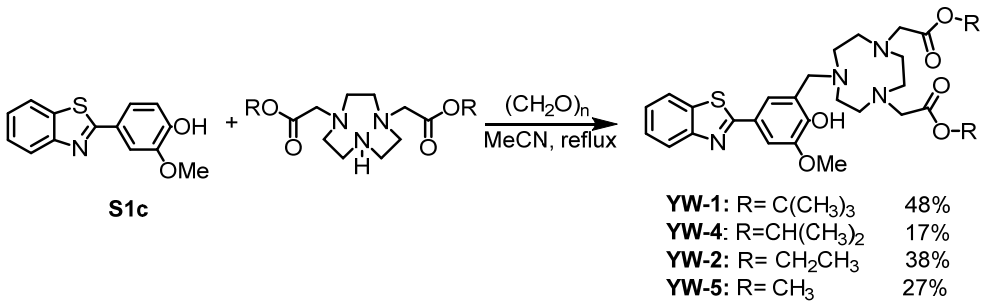
phosphor imager plate scanner (Storm 840) and the resulting images were processed using ImageJ (v1.48, public domain) software.

II. Synthesis and characterization of ligands

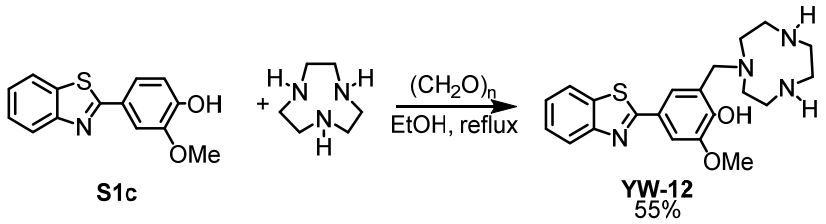


Scheme S1. Synthesis of **S1c.**

S1c. A solution of 2-aminothiophenol **S1a** (3.5 g, 28 mmol) and vanillin **S1b** (4.3 g, 28 mmol) in ethanol (30 mL) was refluxed for 24 h. The precipitated solid obtained upon dilution with water was crystallized from methanol to give yellow crystals of **S1c** in 45% yield (3.3 g), ^{1}H NMR (300 MHz, $(CD_3)_2CO$) δ 8.01 (dddd, *J* = 15.1, 8.1, 1.3, 0.7 Hz, 2H), 7.74 (d, *J* = 2.0 Hz, 1H), 7.60 (d, *J* = 2.1 Hz, 0H), 7.58 (d, *J* = 2.1 Hz, 0H), 7.55 – 7.47 (m, 1H), 7.41 (ddd, *J* = 8.0, 7.2, 1.2 Hz, 1H), 6.99 (d, *J* = 8.3 Hz, 1H), 3.99 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.90, 147.25, 134.94, 126.55, 125.16, 122.92, 122.26, 121.77, 115.01, 109.61, 56.46.

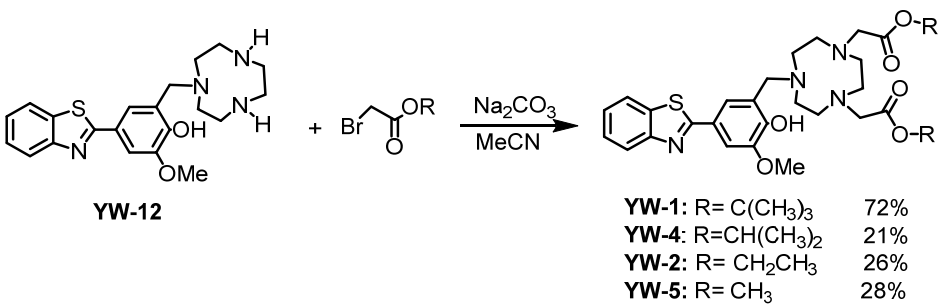


Scheme S2. Synthesis of **YW-1** to **YW-5.**



Scheme S3. Synthesis of **YW-12.**

YW-12. Paraformaldehyde (0.042 g, 1.4 mmol) was added to a solution of 1,4,7-triazacyclononane (0.10 g, 0.77 mmol) in EtOH (10 mL) and the resultant mixture was heated to reflux for 1 h. Then **S1c** (0.30 g, 1.17 mmol) in EtOH (10 mL) was added, the solution was refluxed for an additional 24 hours, and then cooled to room temperature. The solvent was removed to give an orange-yellow residue that was purified by Combi-Flash (reverse-phase) using MeCN/H_2O/TFA (35:65:0.1) to yield a yellow solution, which was then neutralized with $NaHCO_3$, extracted with dichloromethane and dried to give a yellow solid (170 mg, yield 55%).^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 (d, J = 9.9 Hz, 1H), 7.84 (d, J = 7.9 Hz, 1H), 7.53 (s, 1H), 7.41 (s, 2H), 7.31 (s, 1H), 3.96 (s, 3H), 3.83 (s, 2H), 2.87 (s, 13H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 154.67, 150.23, 134.78, 126.16, 125.59, 124.23, 123.93, 122.25, 121.53, 116.68, 109.53, 56.63, 55.94, 50.06, 46.03, 45.29.



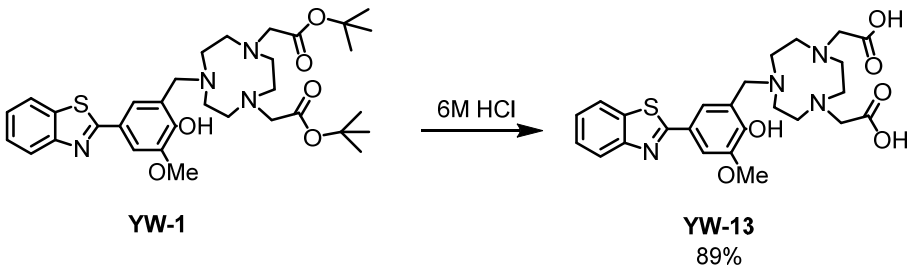
Scheme S4. Synthesis of **YW-1** to **YW-5** via reactions from **YW-12.**

YW-1. To a suspension of YW-12 (75 mg, 0.19 mmol) and sodium carbonate (44 mg, 0.42 mmol) in chloroform (5 ml), tert-butyl bromoacetate (81 mg, 0.42 mmol) in chloroform (10 ml) was added. The reaction mixture was stirred at room temperature for 1d. The solvent was removed to give an orange-yellow residue that was purified by Combi-Flash (reverse-phase) using MeCN/H_2O/TFA (74:26:0.1) to yield a yellow solution, which was then neutralized with $NaHCO_3$, extracted with dichloromethane and dried to give a yellow solid (85 mg, yield 72%).^{1}H NMR (300 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.1 Hz, 1H), 7.87 (d, *J* = 7.9 Hz, 1H), 7.60 (s, 1H), 7.50 – 7.42 (m, 2H), 7.39 – 7.31 (m, 1H), 4.01 (s, 5H), 3.39 (s, 4H), 3.06 (d, *J* = 33.3 Hz, 7H), 2.78 (s, 4H), 1.45 (s, 18H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.37, 151.61, 137.55, 128.92, 127.64, 125.43, 124.22, 113.97, 84.45, 59.05, 54.54, 30.77. HR-ESI-MS: Calcd for $[M+H]^+$, 627.3172; Found, 627.3203.

YW-4. To a suspension of YW-12 (80 mg, 0.2 mmol) and sodium carbonate (47 mg, 0.44 mmol) in acetonitrile (5 ml), isopropyl bromoacetate (80 mg, 0.44 mmol) in acetonitrile (10 ml) was added. The reaction mixture was stirred at room temperature for 12 h. The solvent was removed to give an orange-yellow residue that was purified by Combi-Flash (reverse-phase) using MeCN/H_2O/TFA (60:40:0.1) to yield a yellow solution, which was then neutralized with $NaHCO_3$, extracted with dichloromethane and dried to give a yellow solid (25 mg, yield 21%).^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.2 Hz, 1H), 7.86 (d, *J* = 8.4 Hz, 1H), 7.56 (s, 1H), 7.51 – 7.42 (m, 1H), 7.34 (d, *J* = 17.4 Hz, 2H), 5.03 (p, *J* = 6.3 Hz, 2H), 3.99 (d, *J* = 7.5 Hz, 5H), 3.45 (s, 4H), 2.98 (s, 8H), 2.78 (s, 4H), 1.28 – 1.20 (m, 12H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.81, 154.37, 126.44, 124.99, 122.91, 121.75, 110.42, 68.30, 58.35, 56.50, 53.16, 29.95, 22.20, 1.27. HR-ESI-MS: Calcd for $[M+H]^+$, 599.2859; Found, 599.2916.

YW-2. To a suspension of YW-12 (28 mg, 0.07 mmol) and sodium carbonate (16 mg, 0.15 mmol) in acetonitrile (5 ml), ethyl bromoacetate (25 mg, 0.15 mmol) in acetonitrile (3 ml) was added. The reaction mixture was stirred at room temperature for 20 h. The solvent was removed to give an orange-yellow residue that was purified by Combi-Flash (reverse-phase) using MeCN/H_2O/TFA (45:55:0.1) to yield a yellow solution, which was then neutralized with $NaHCO_3$, extracted with dichloromethane and dried to give a yellow solid (10 mg, yield 26%).^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.2 Hz, 1H), 7.86 (d, *J* = 9.1 Hz, 1H), 7.65 (d, *J* = 12.1 Hz, 2H), 7.52 – 7.41 (m, 1H), 7.39 – 7.29 (m, 1H), 4.38 (s, 2H), 4.16 (s, 4H), 4.01 (s, 3H), 3.84 (s, 4H), 3.47 (s, 6H), 3.26 (d, *J* = 38.8 Hz, 12H), 1.27 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.49, 126.40, 124.87, 122.84, 121.70, 120.82, 116.62, 110.07, 60.49, 58.54, 56.43, 53.70, 14.57, 2.13. HR-ESI-MS: Calcd for $[M+H]^+$, 571.2546; Found, 571.2587.

YW-5. To a suspension of YW-12 (46 mg, 0.12 mmol) and sodium carbonate (27 mg, 0.25 mmol) in DCM (5 ml), methyl bromoacetate (39 mg, 0.25 mmol) in DCM (10 ml) was added. The reaction mixture was stirred at room temperature for 6h. The solvent was removed to give a yellow residue that was purified by silica gel chromatography using DCM/MeOH (30:1) (18 mg, yield 28%).^{1}H NMR (300 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.1 Hz, 1H), 7.85 (d, *J* = 11.8 Hz, 1H), 7.54 (s, 1H), 7.44 (t, *J* = 7.7 Hz, 1H), 7.32 (d, *J* = 14.5 Hz, 2H), 3.98 (s, 3H), 3.94 (s, 2H), 3.67 (s, 6H), 3.47 (s, 12H), 2.77 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.94, 154.27, 126.40, 124.89, 122.85, 121.70, 109.97, 58.26, 56.45, 53.53, 51.52, 29.95. HR-ESI-MS: Calcd for $[M+H]^+$, 543.2233; Found, 543.2281.



Scheme S5. Synthesis of **YW-13.**

YW-13. The ester YW-1 (15 mg, 0.024 mmol) was dissolved in 5 mL of 6 M HCl and stirred at room temperature for 4h. The solvent was removed to get a yellow residue, which was dissolved in diethyl ether and filtered. Removal of the solvent gave a light yellow powder, which was dried under vacuum to obtain the product (11 mg, yield 89%). ^{1}H NMR (300 MHz, C_2D_6OS) δ 8.11 (d, J = 8.6 Hz, 1H), 8.01 (d, J = 8.3 Hz, 1H), 7.74 (d, J = 1.8 Hz, 1H), 7.52 (t, J = 8.3 Hz, 1H), 7.43 (t, J = 7.0 Hz, 1H), 4.49 (s, 2H), 3.99 (s, 7H), 3.23 (s, 12H). ^{13}C NMR (126 MHz, CD_3OD) δ 171.83, 149.31, 126.52, 125.06, 121.09, 109.80, 54.48, 53.07, 26.85. HR-ESI-MS: Calcd for $[M+H]^+$, 515.1920; Found, 515.1955.

III. Brain section staining results for BFCs.

Ligand	Conc. (μM)	Ligand to Congo Red ratio
YW-1	50	25:1
Cu-YW-1	50	25:1
YW-2	50	25:1
Cu-YW-2	50	25:1
YW-13	50	25:1
Cu-YW-13	50	25:1

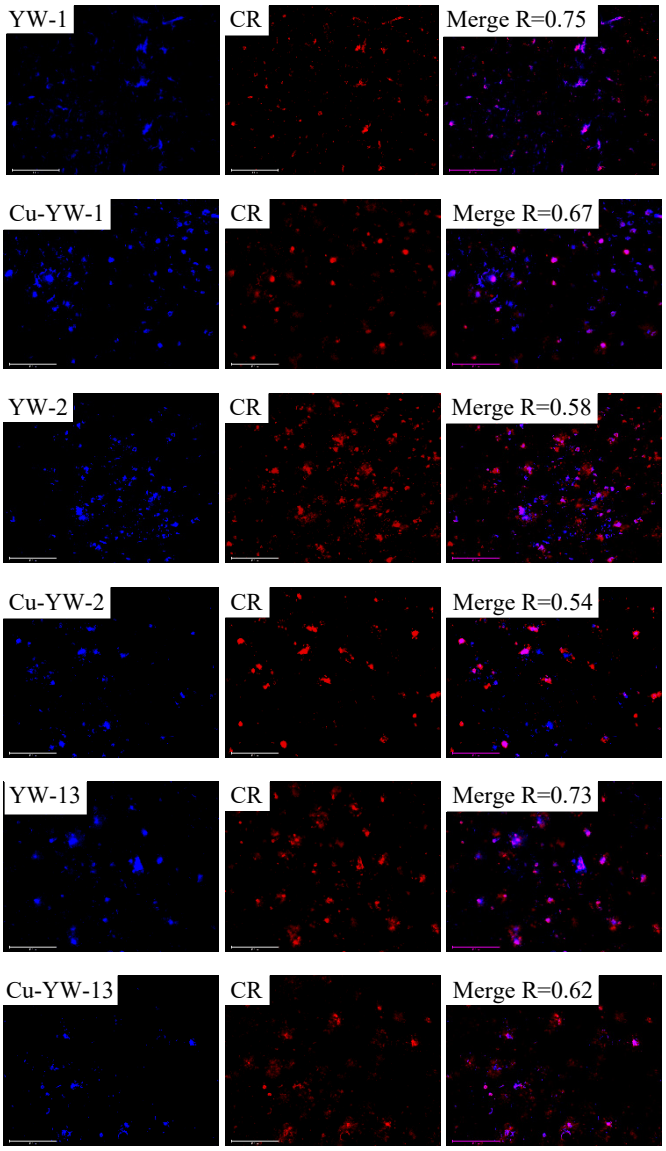


Figure S1. Fluorescence microscopy images of 5xFAD brain sections incubated with **YW-1, YW-2, YW-13** and their Cu(II) complexes (left panels), Congo Red (middle panels), and merged images (right panels). Magnification: 20x. Scale bar: 125 μm.

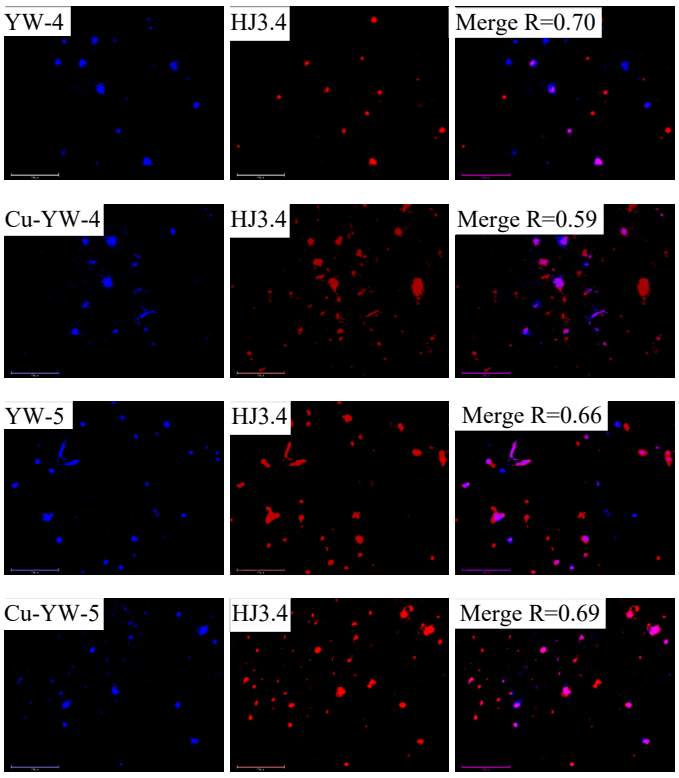


Figure S2. Fluorescence microscopy images of 5xFAD brain sections incubated with **YW-4, YW-5,** and their Cu(II) complexes (left panels), AF594-HJ3.4 (middle panels), and merged images (right panels). Magnification: 20x. Scale bar: 125 μm.

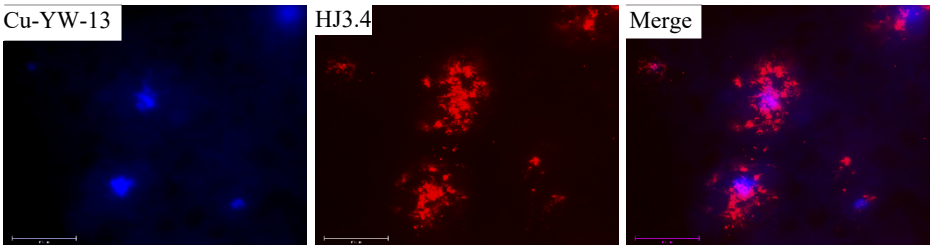


Figure S3. Fluorescence microscopy images of 5xFAD mouse brain sections incubated with **Cu-YW-13** (left panel), AF594-HJ3.4 (middle panel), and merged images (right panel). Magnification: 40x. Scale bar: 50 μm.

IV. pH-Spectrophotometric Titrations for BFCs

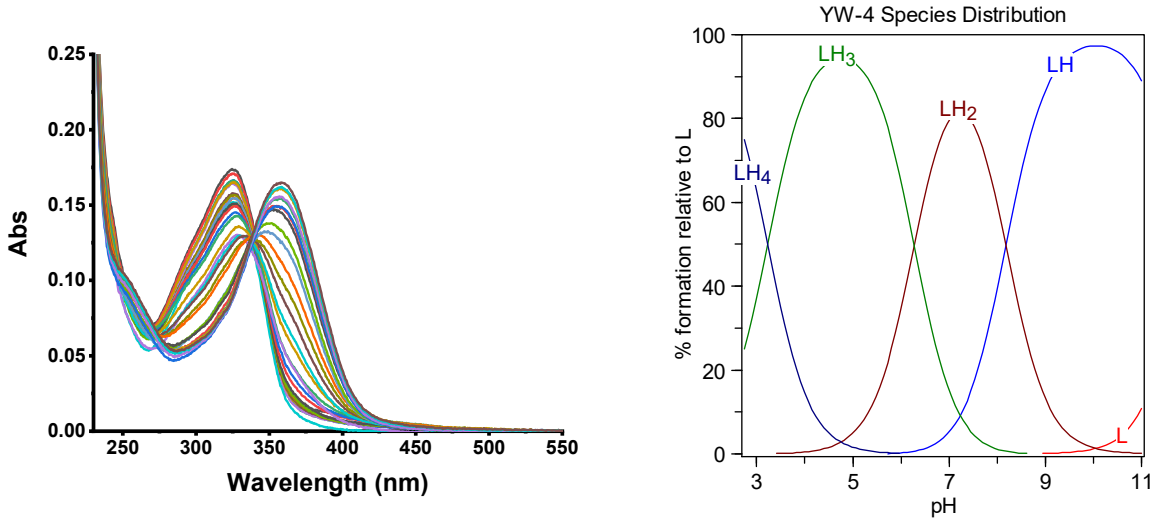


Figure S4. Variable pH (pH 3−11) UV-Vis spectra of **YW-4** (20 μM, 25 °C, I = 0.1 M NaCl) and the species distribution plot.

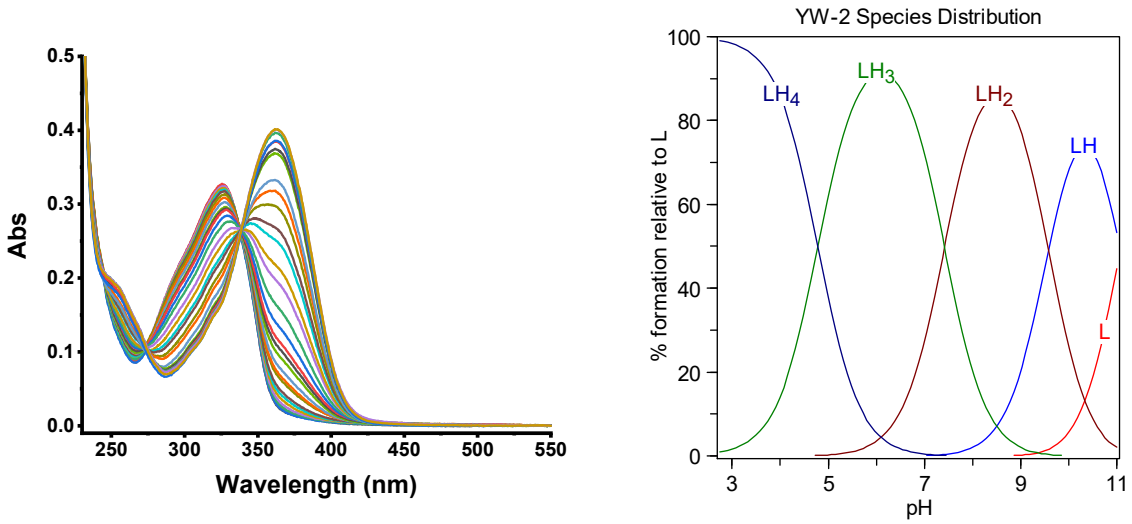


Figure S5. Variable pH (pH 3−11) UV-Vis spectra of **YW-2** (20 μM, 25 °C, I = 0.1 M NaCl) and the species distribution plot.

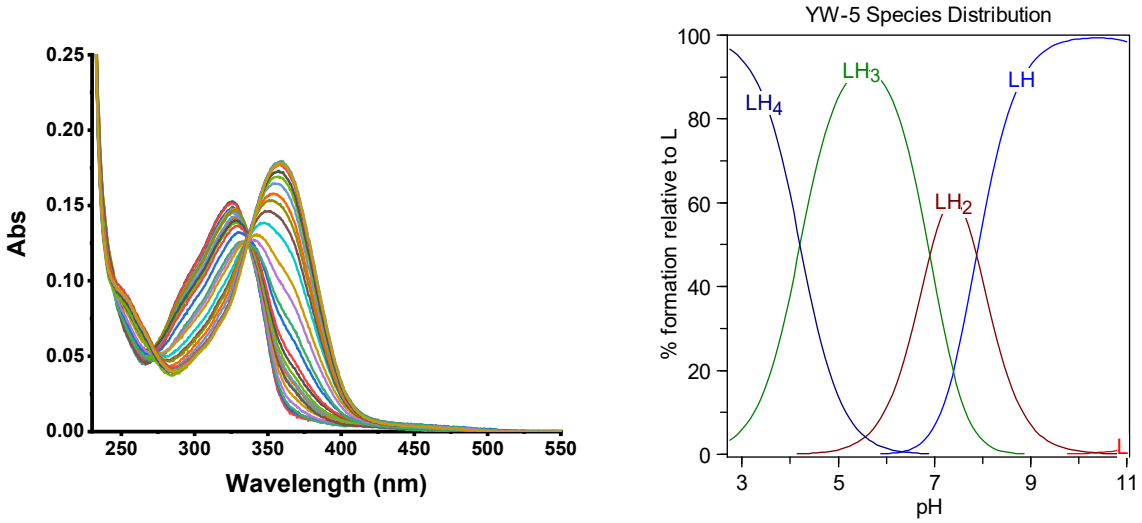


Figure S6. Variable pH (pH 3−11) UV-Vis spectra of **YW-5** (20 μM, 25 °C, I = 0.1 M NaCl) and the species distribution plot.

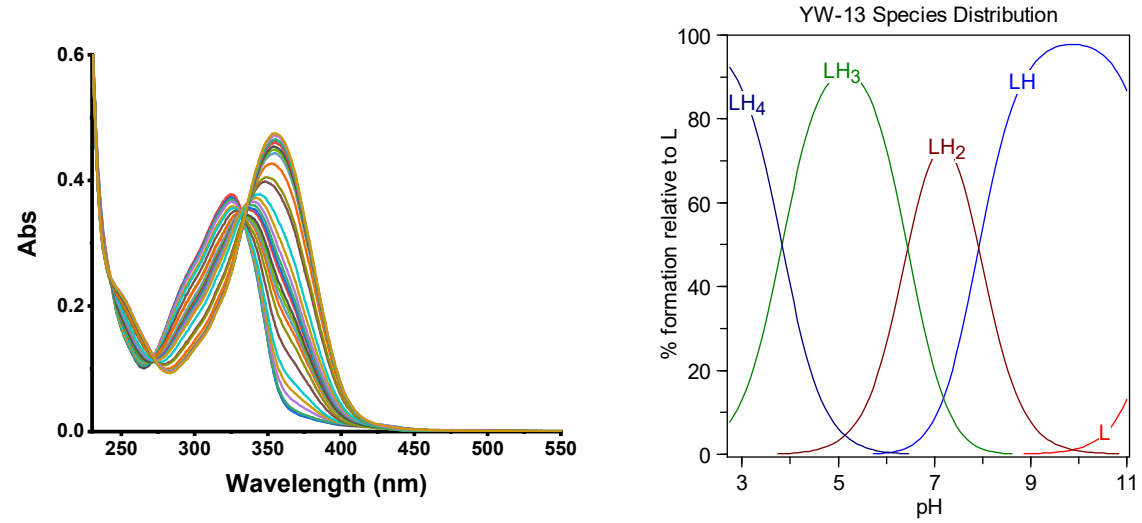


Figure S7. Variable pH (pH 1.7−11) UV-Vis spectra of **YW-13** (20 μM, 25 °C, I = 0.1 M NaCl) and the species distribution plot.

V. pH-Spectrophotometric Titrations for Cu(II) Complexes

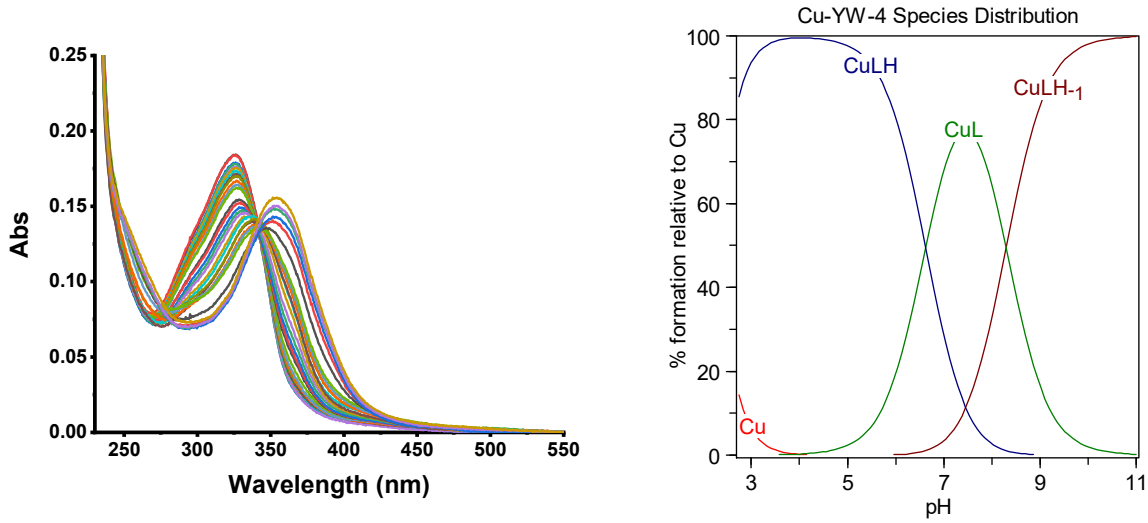


Figure S8. Variable pH (pH 3-11) UV-Vis spectra of **YW-4** and Cu^{2+} system ([**L**] = [Cu^{2+}] = 20μM, 25 °C, I = 0.1 M NaCl) and the species distribution plot.

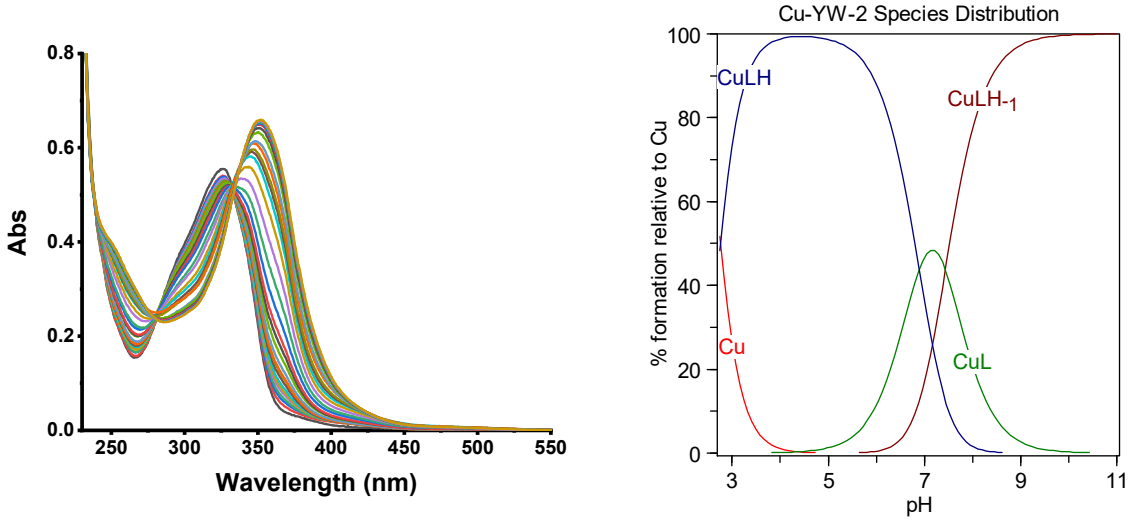


Figure S9. Variable pH (pH 3-11) UV-Vis spectra of **YW-2** and Cu^{2+} system ([**L**] = [Cu^{2+}] = 20μM, 25 °C, I = 0.1 M NaCl) and the species distribution plot.

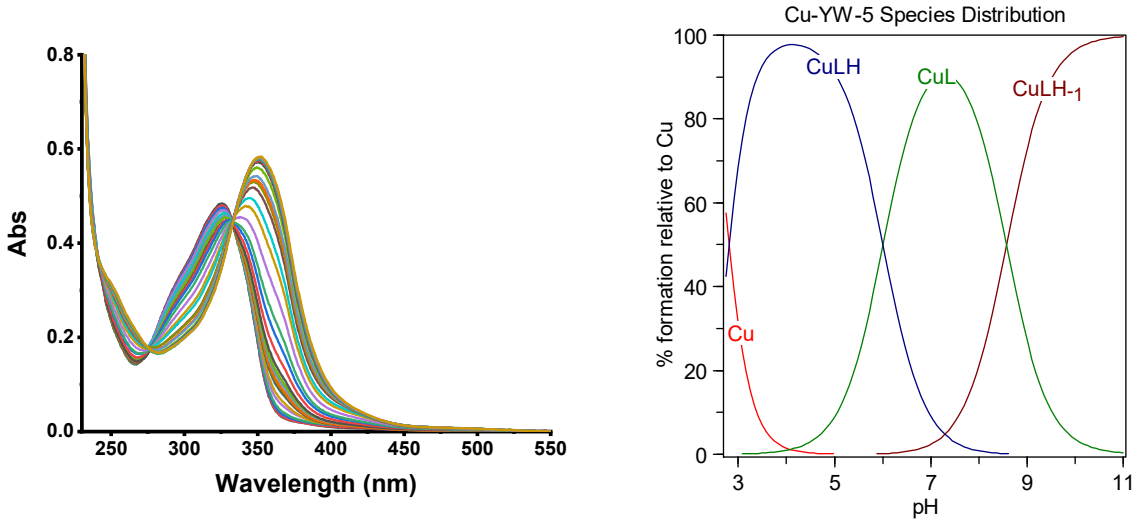


Figure S10. Variable pH (pH 3-11) UV-Vis spectra of **YW-5** and Cu^{2+} system ([**L**] = [Cu^{2+}] = 20μM, 25 °C, I = 0.1 M NaCl) and the species distribution plot.

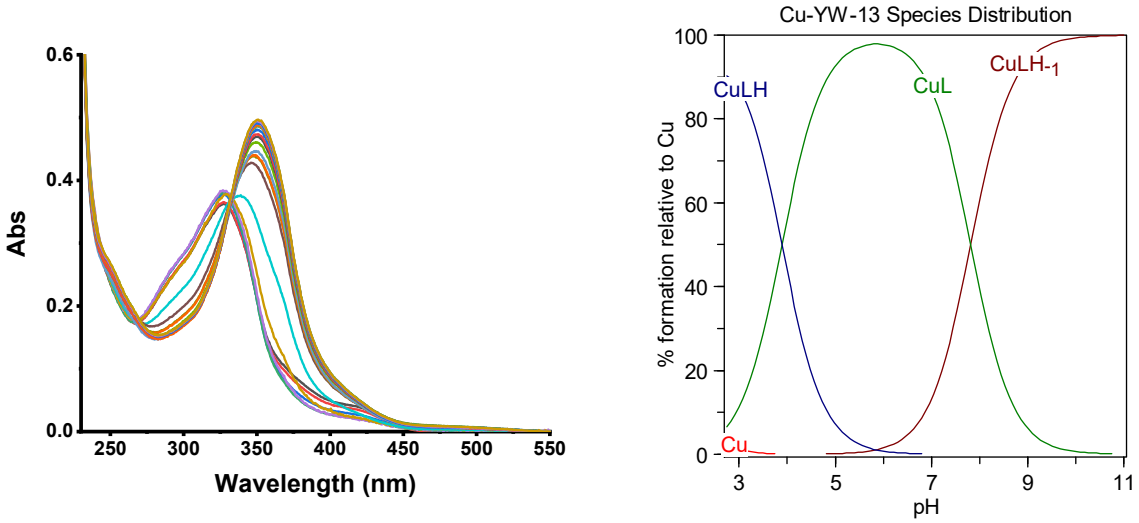


Figure S11. Variable pH (pH 1.7-11) UV-Vis spectra of **YW-13** and Cu^{2+} system ([**L**] = [Cu^{2+}] = 20μM, 25 °C, I = 0.1 M NaCl) and the species distribution plot.

VI. Job's plot of solution stoichiometry determination for YW-13

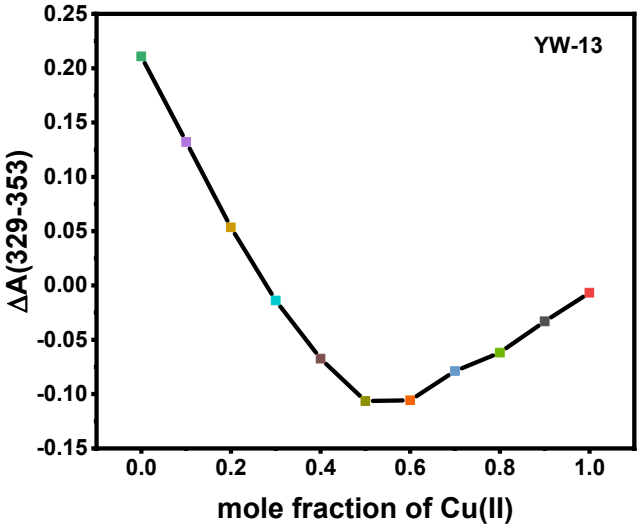


Figure S12. Job's plot for **YW-13** and Cu^{2+} in PBS.

VII. HPLC traces from ^{64}Cu radiolabeling of BFCs.

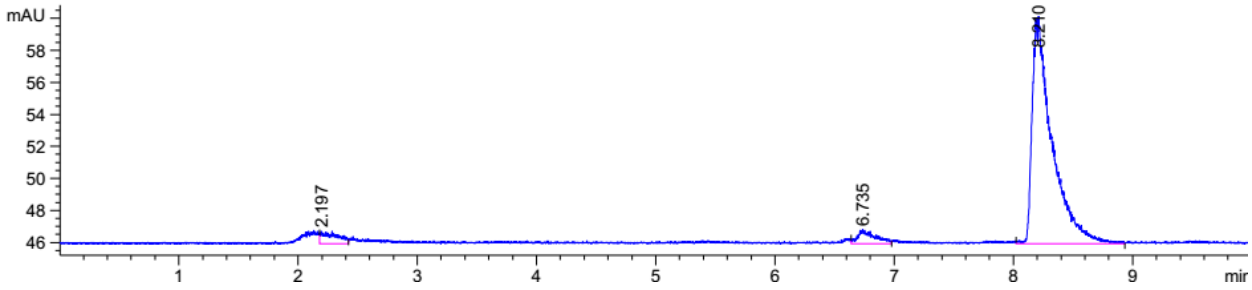


Figure S13. Radio-HPLC profile of **^{64}Cu-YW-1** complex, retention time is 8.3 minutes.

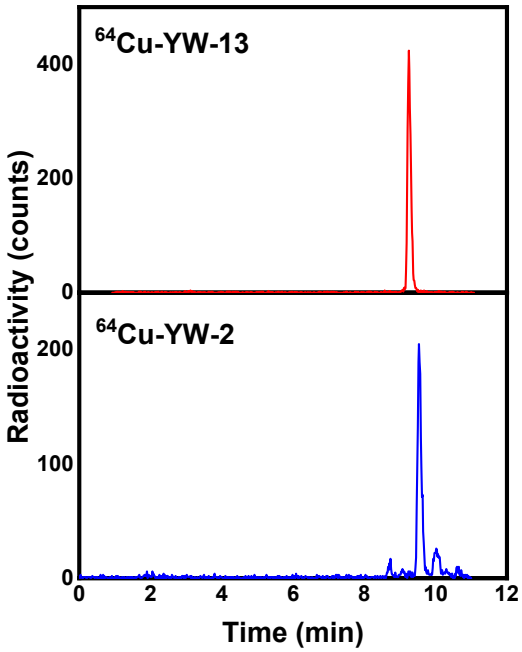
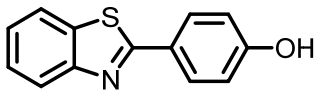


Figure S14. Radio-HPLC profiles of **^{64}Cu-YW-13** and **^{64}Cu-YW-2** complexes, retention times are 9.0 and 9.5 minutes, respectively.

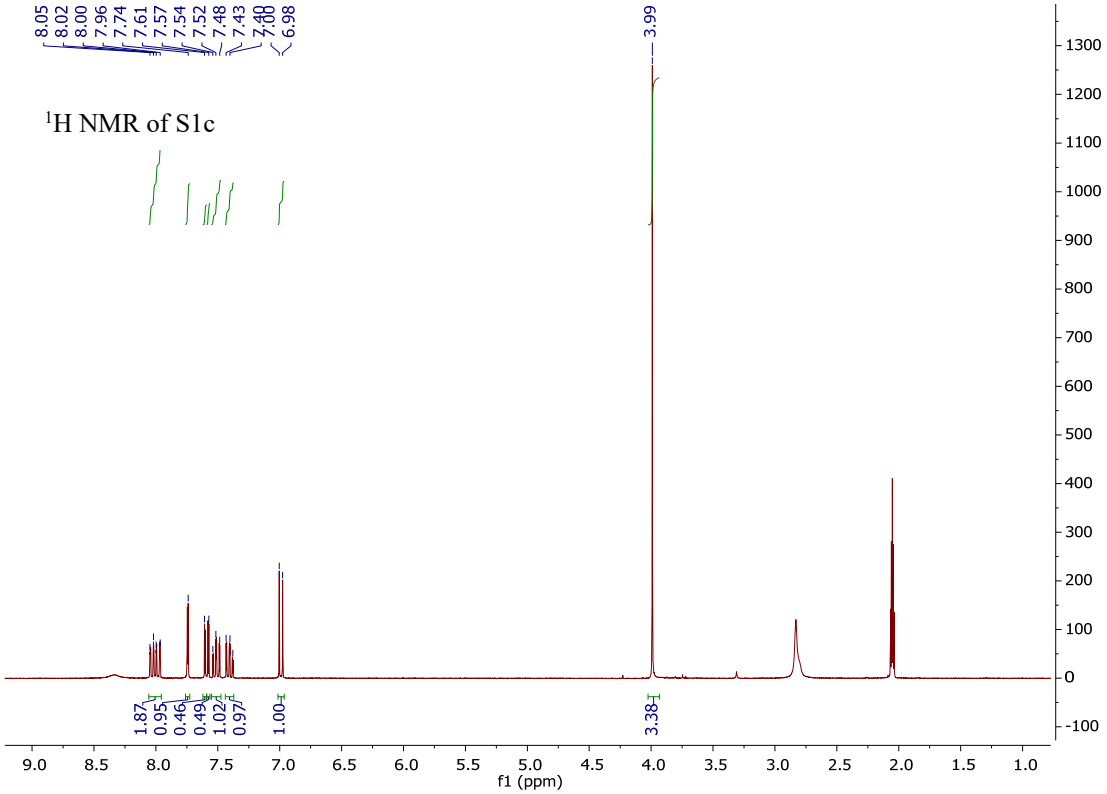
VIII. Structure of blocking agent B_1



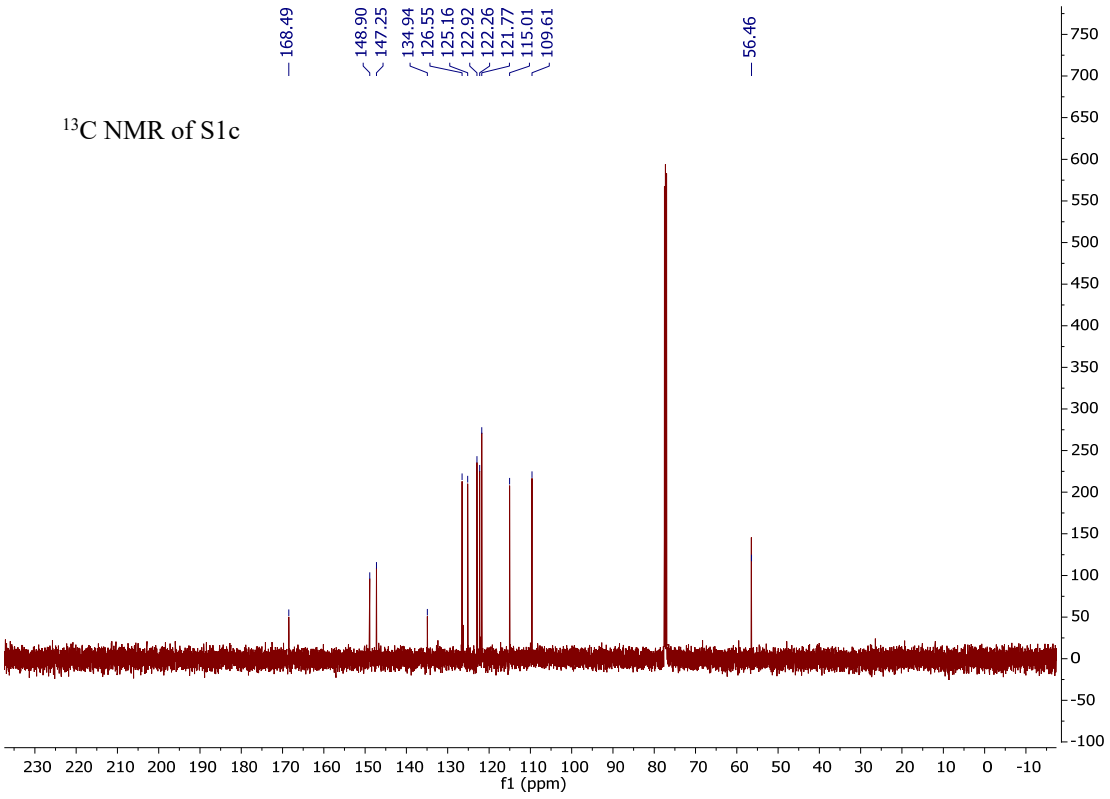
2-(4-hydroxyphenol)benzothiazole (B_1)

Figure S15. Structure of the non-radioactive compound used as blocking agent in the autoradiography studies.

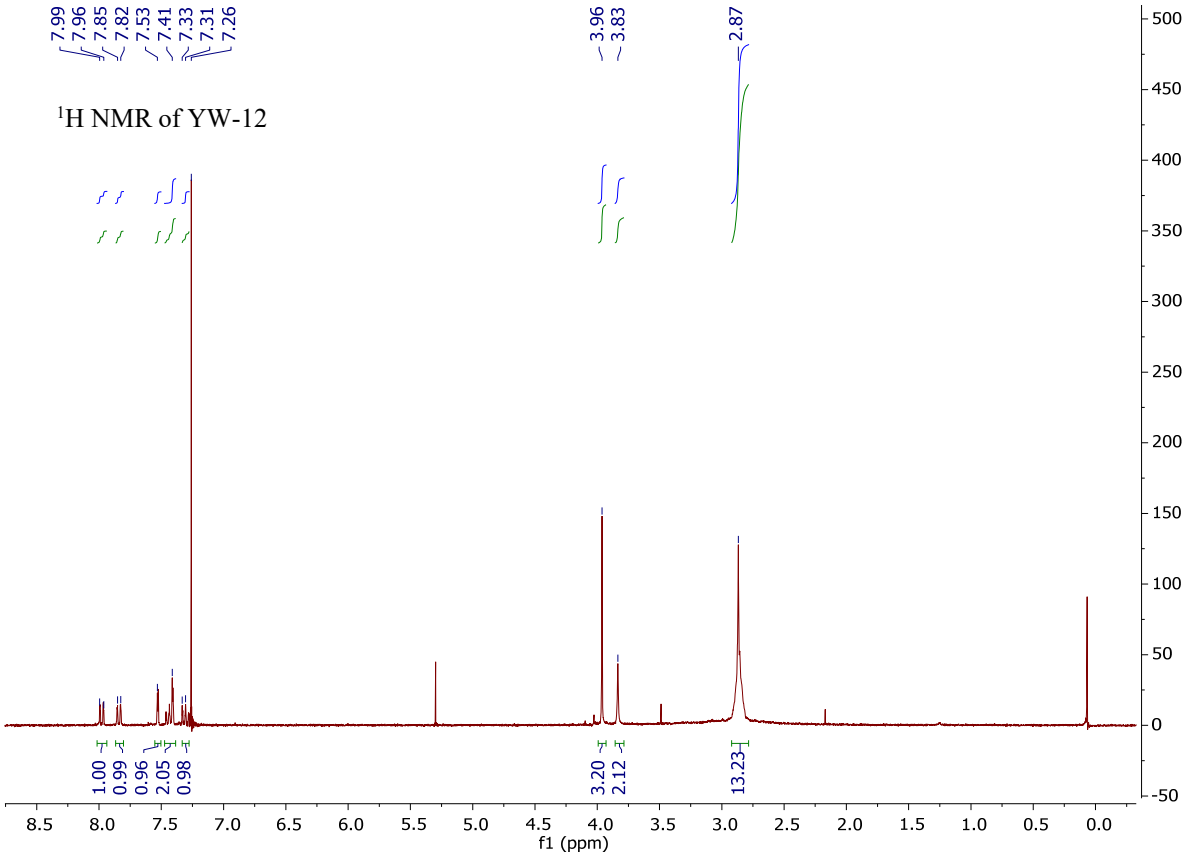
IX. NMR spectra and HR-MS profiles of BFCs



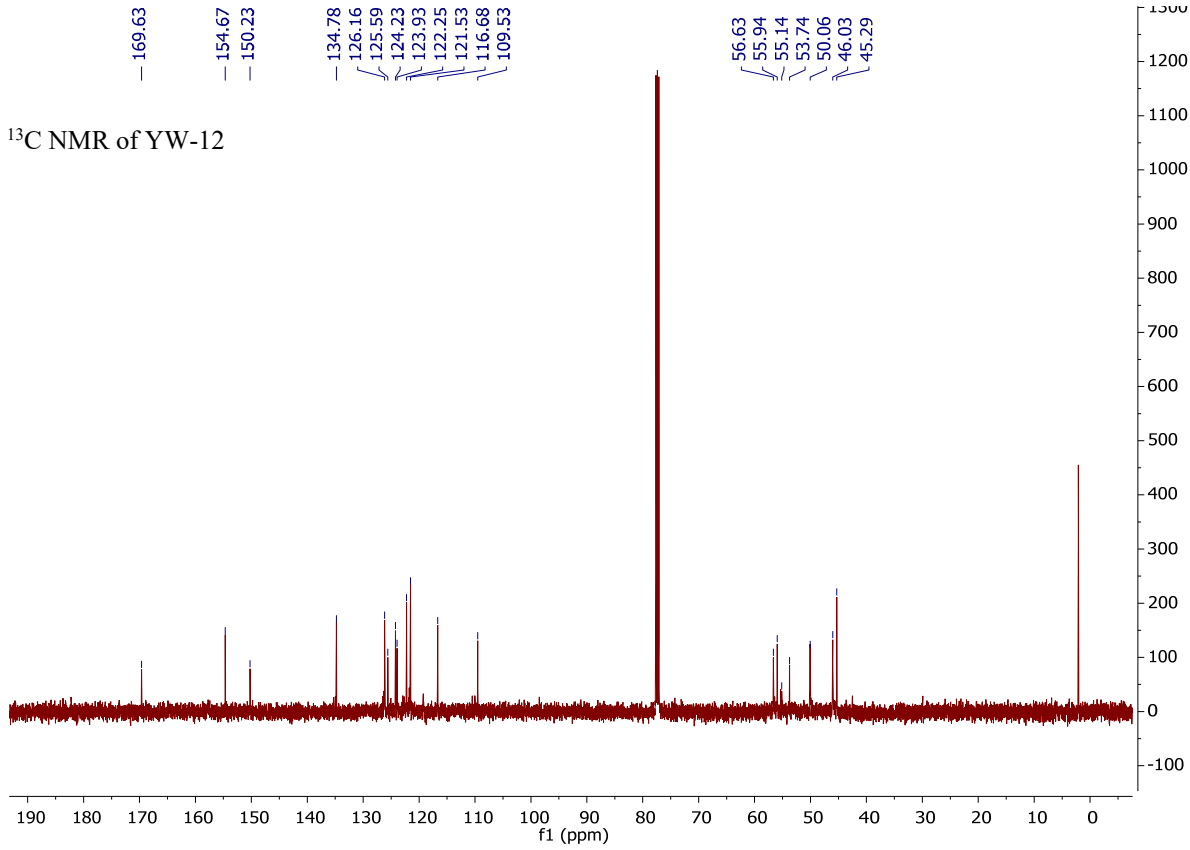
^{1}H NMR of S1c



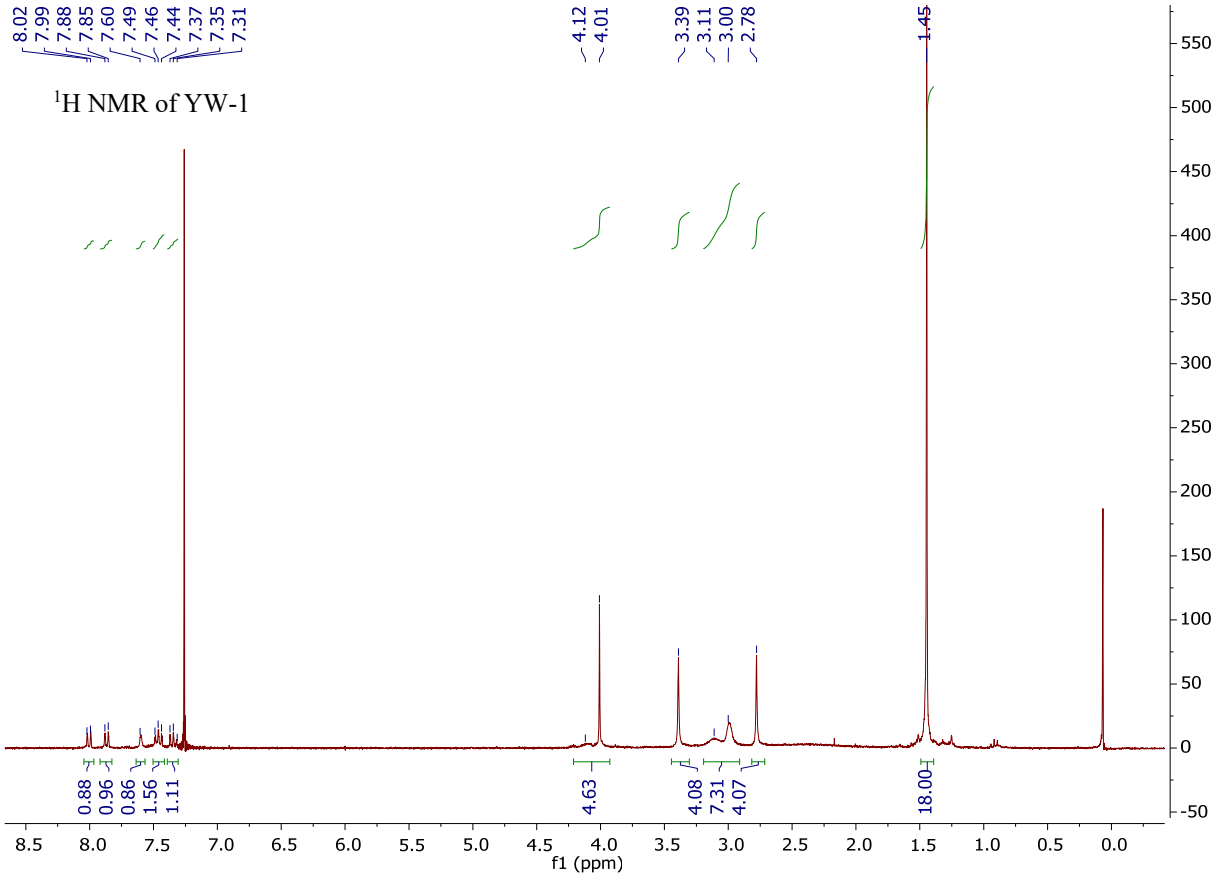
^{13}C NMR of S1c



^{1}H NMR of YW-12

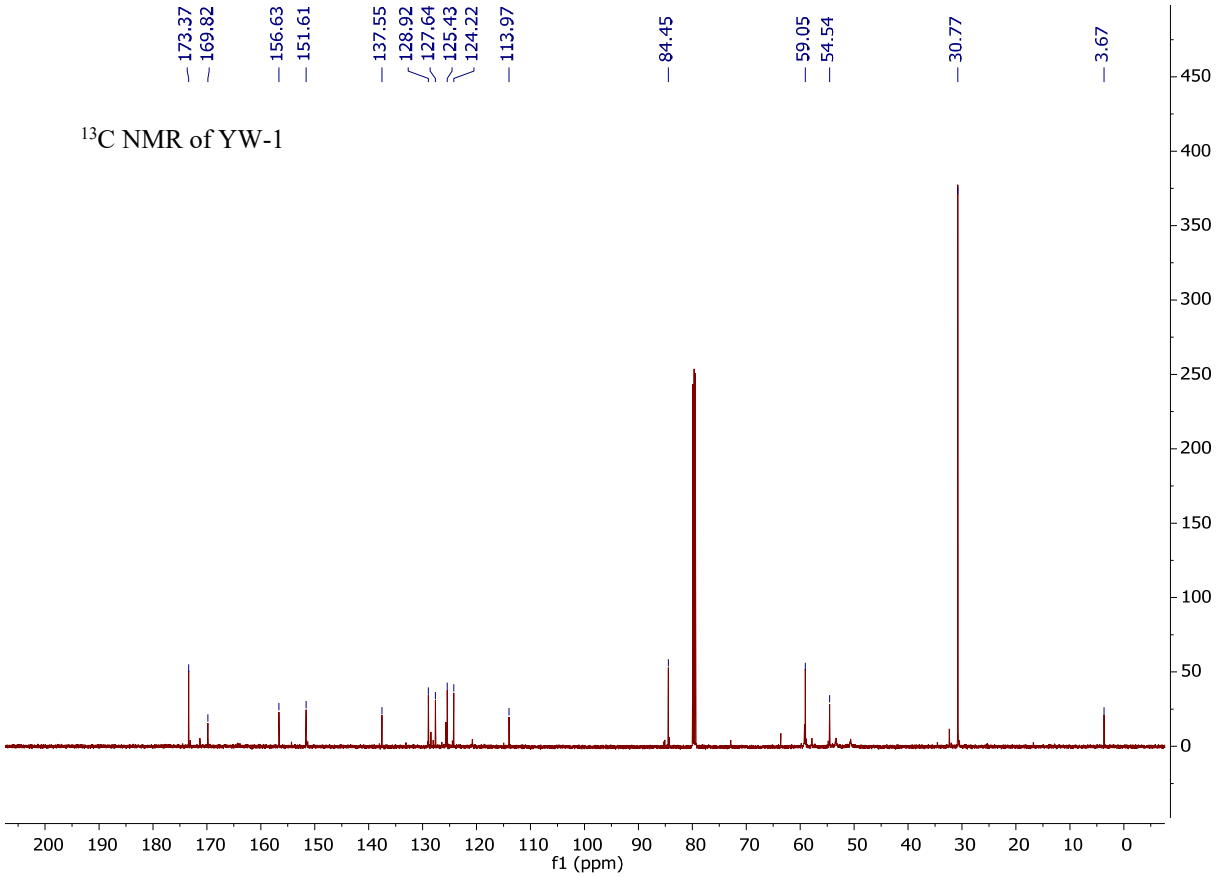


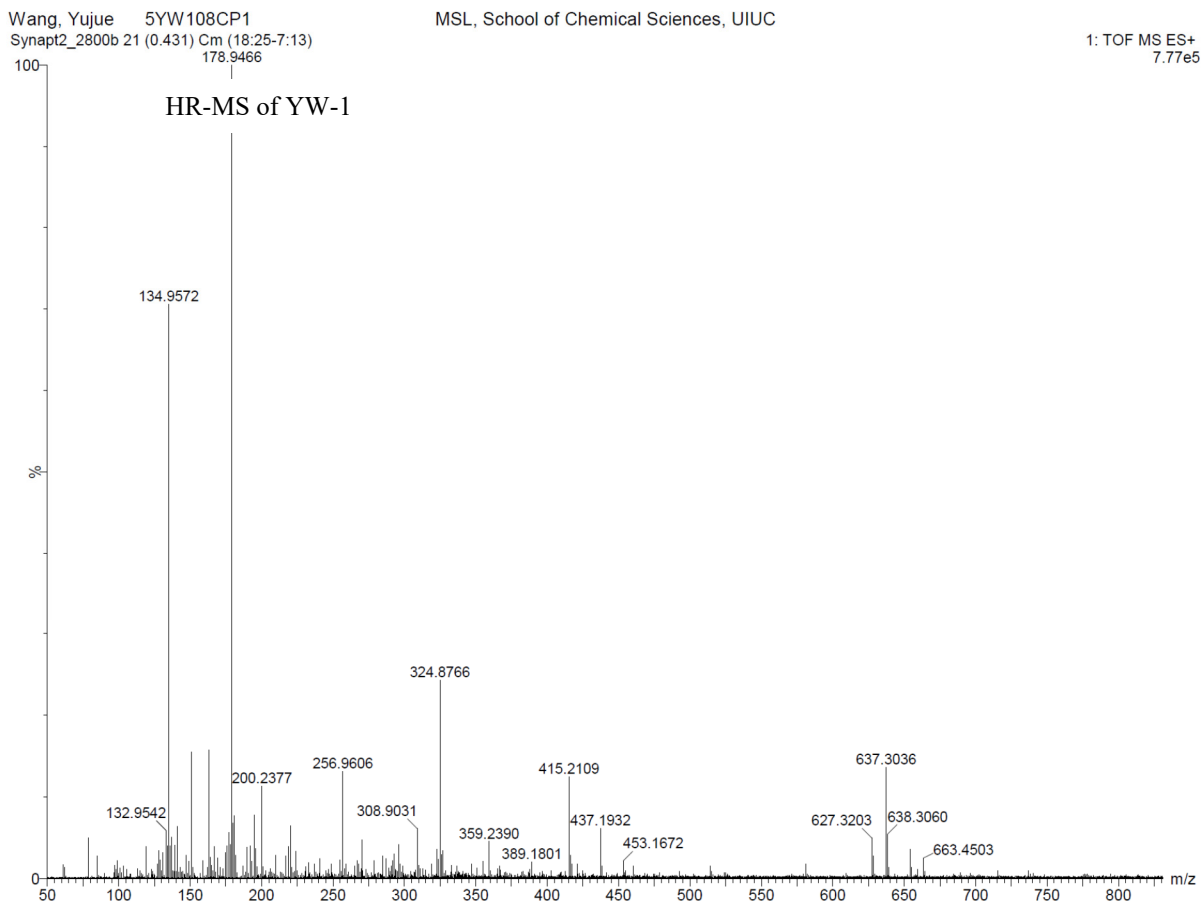
^{13}C NMR of YW-12

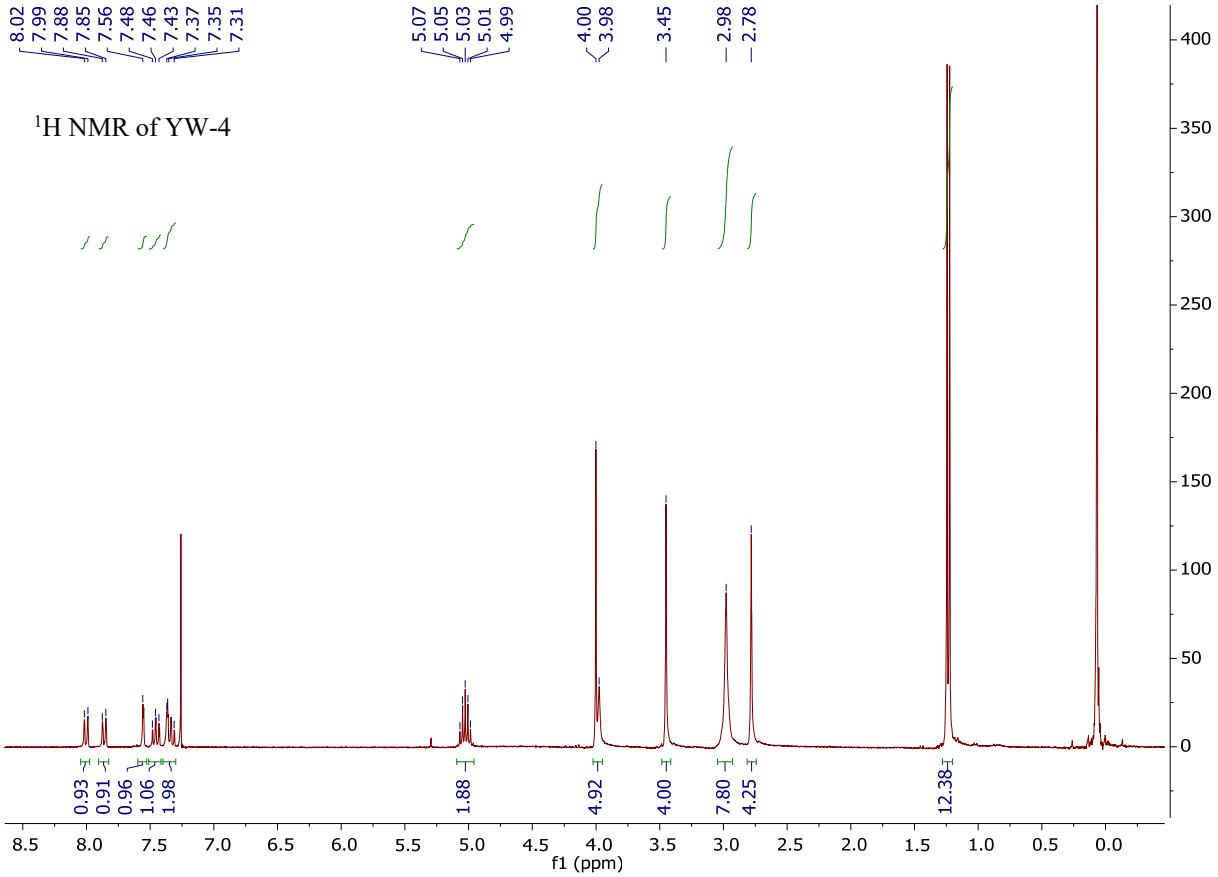

8.02
7.99
7.88
7.85
7.60
7.49
7.46
7.44
7.37
7.35
7.31
4.12
4.01
3.39
3.11
3.00
2.78
1.45
0.88
0.96
0.86
1.56
1.11
4.63
4.08
7.31
4.07
18.00
f1 (ppm)

^{1}H NMR of YW-1

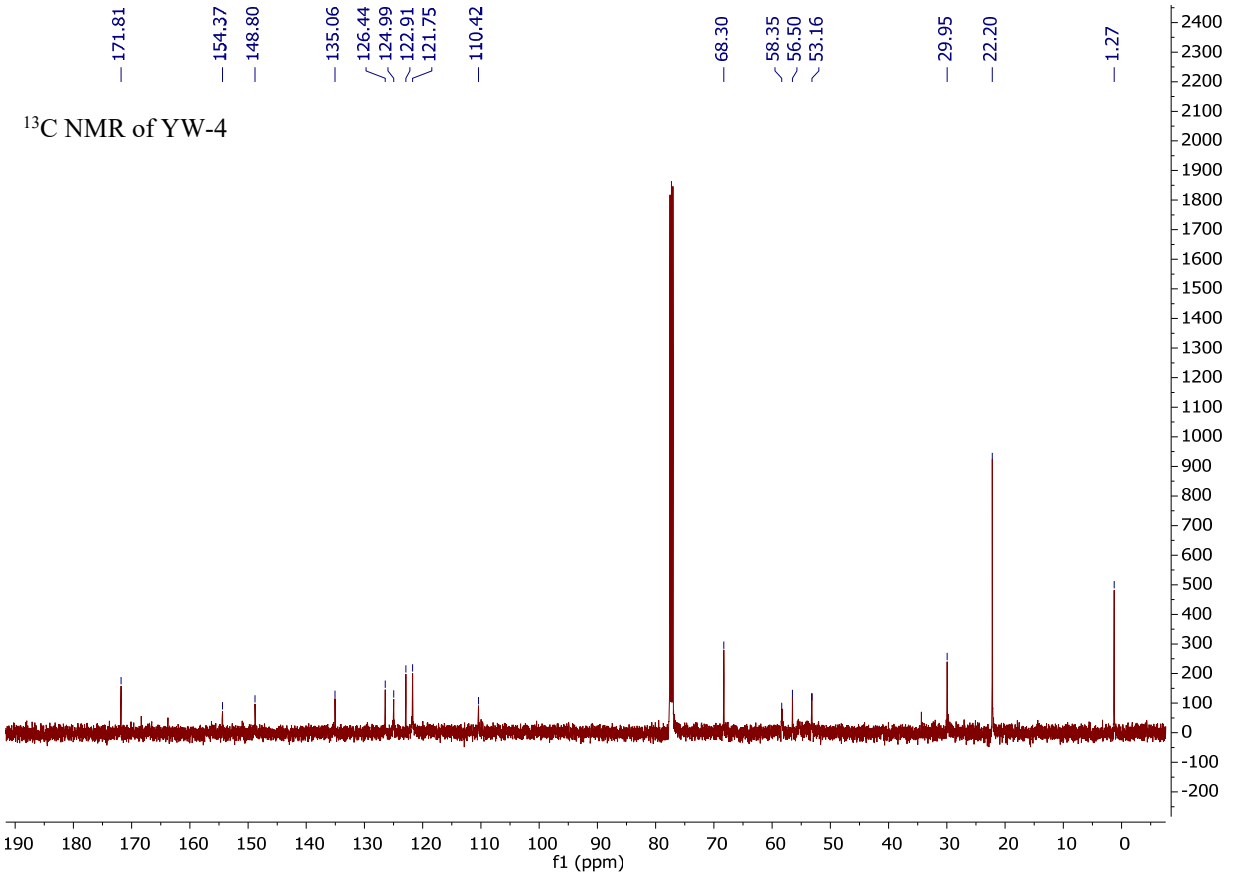
^{13}C NMR of YW-1



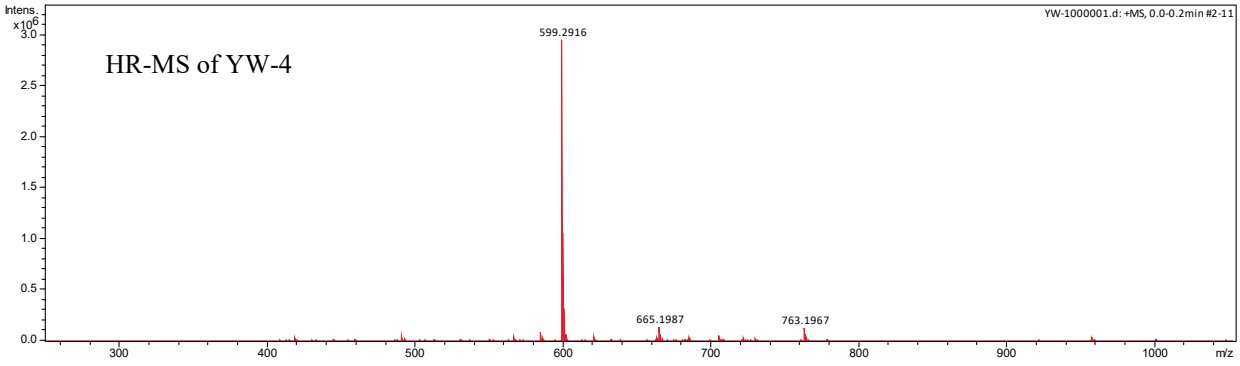

Wang, Yujue 5YW108CP1
MSL, School of Chemical Sciences, UIUC
Synapt2_2800b 21 (0.431) Cm (18:25-7:13)
1: TOF MS ES+
7.77e5
HR-MS of YW-1
178.9466
134.9572
324.8766
200.2377
256.9606
415.2109
637.3036
132.9542
308.9031
437.1932
627.3203
638.3060
359.2390
453.1672
389.1801
663.4503
%
m/z


8.02
7.99
7.88
7.85
7.56
7.48
7.46
7.43
7.37
7.35
7.31
5.07
5.05
5.03
5.01
4.99
4.00
3.98
3.45
2.98
2.78
0.93
0.91
0.96
1.06
1.98
1.88
4.92
4.00
7.80
4.25
12.38
f1 (ppm)

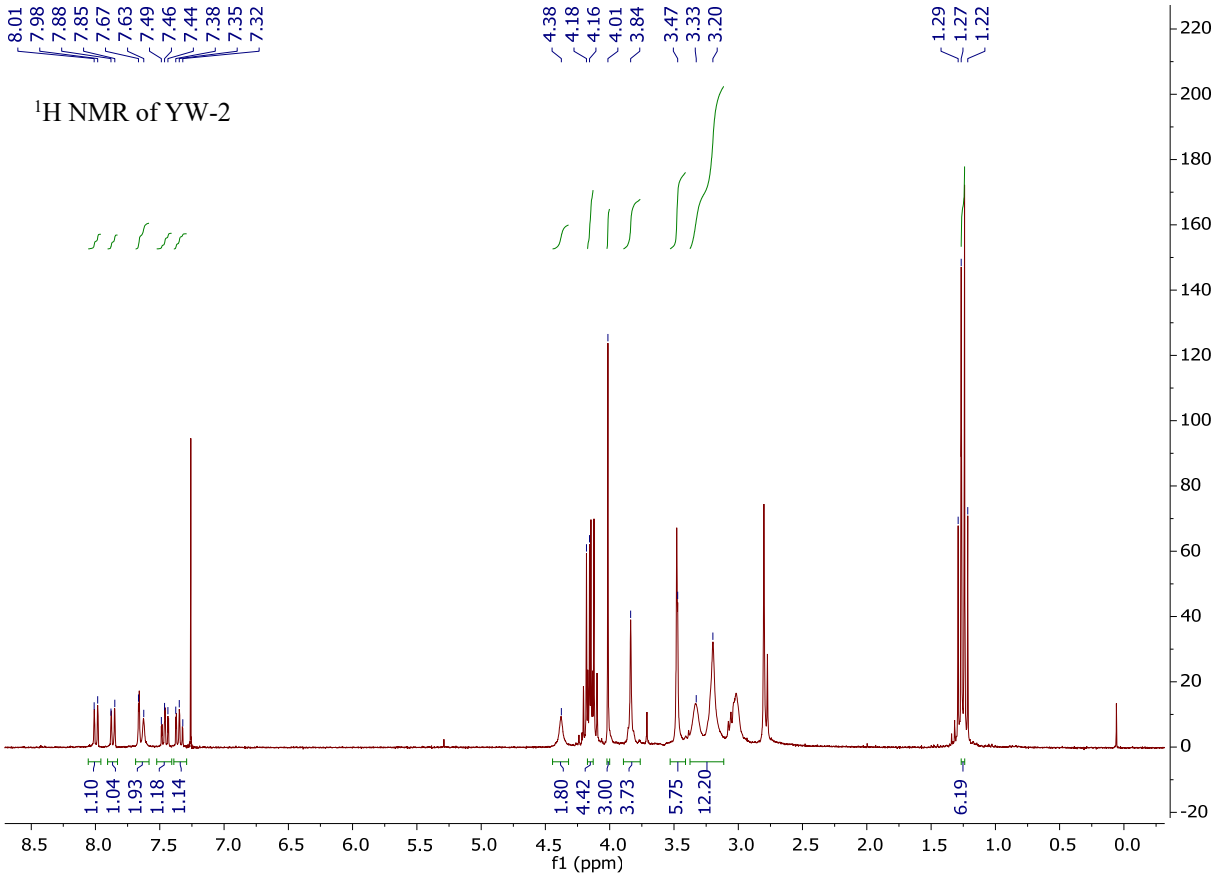
^{1}H NMR of YW-4



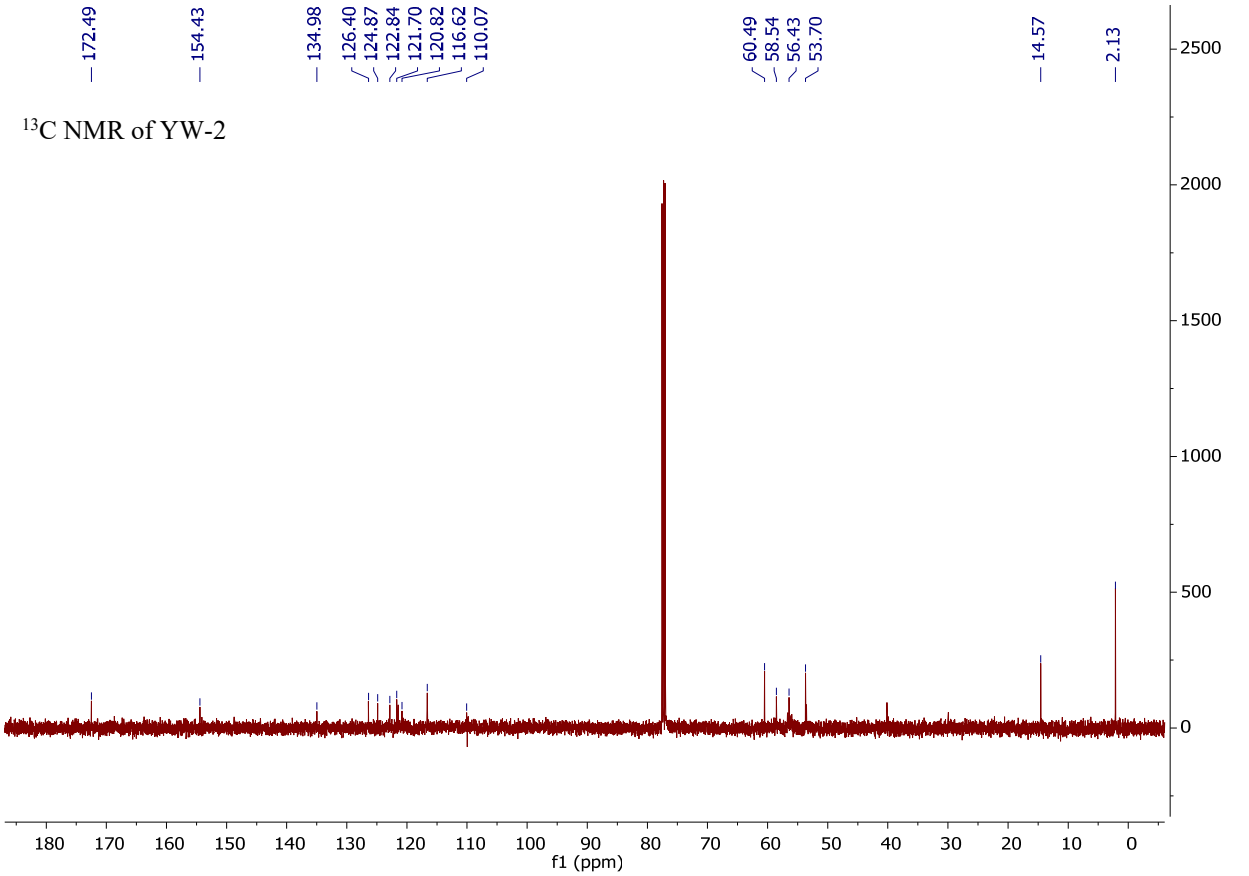
^{13}C NMR of YW-4



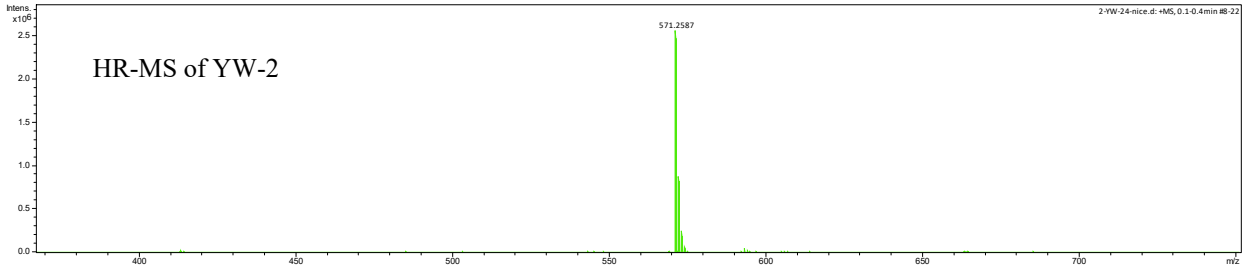
HR-MS of YW-4


8.01
7.98
7.88
7.85
7.67
7.63
7.49
7.46
7.44
7.38
7.35
7.32
4.38
4.18
4.16
4.01
3.84
3.47
3.33
3.20
1.29
1.27
1.22
1.10
1.04
1.93
1.18
1.14
1.80
4.42
3.00
3.73
5.75
12.20
6.19
f1 (ppm)

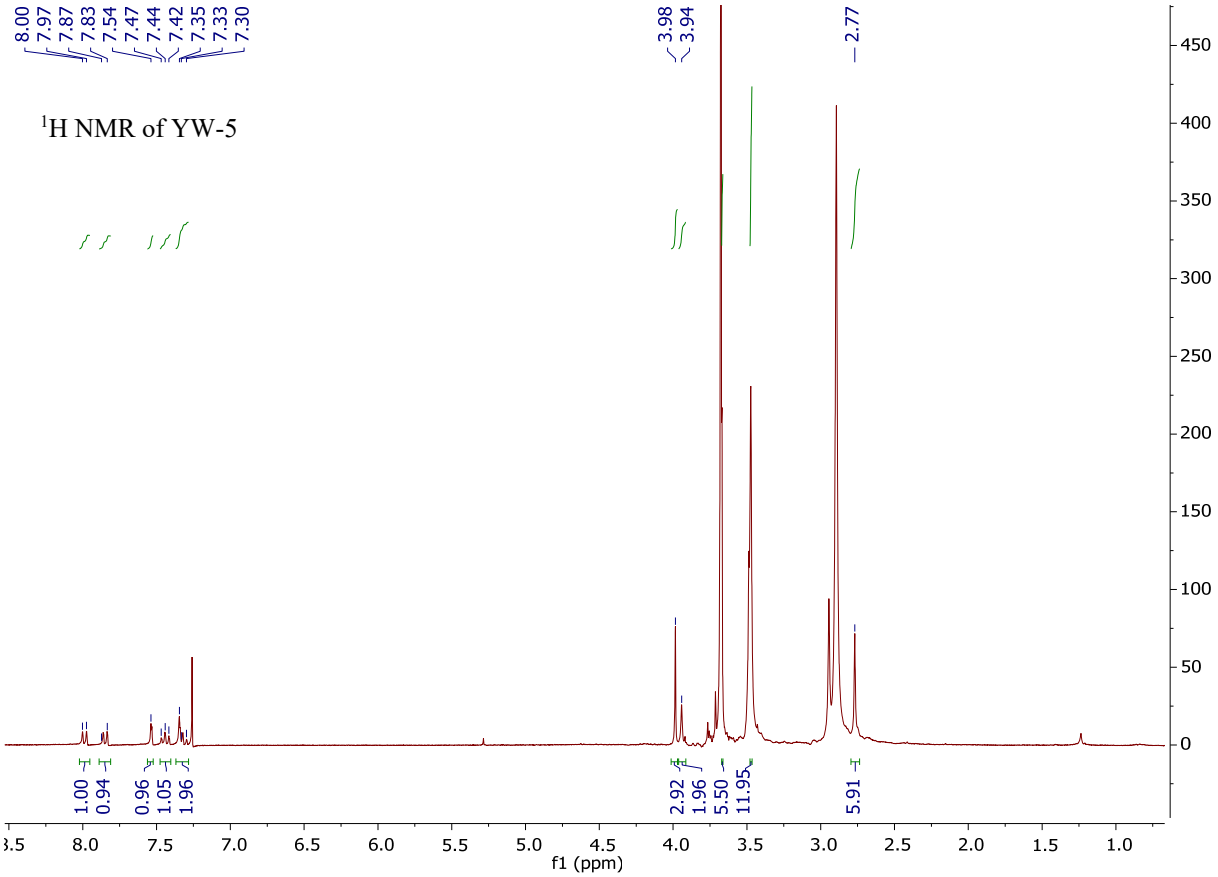
^{1}H NMR of YW-2



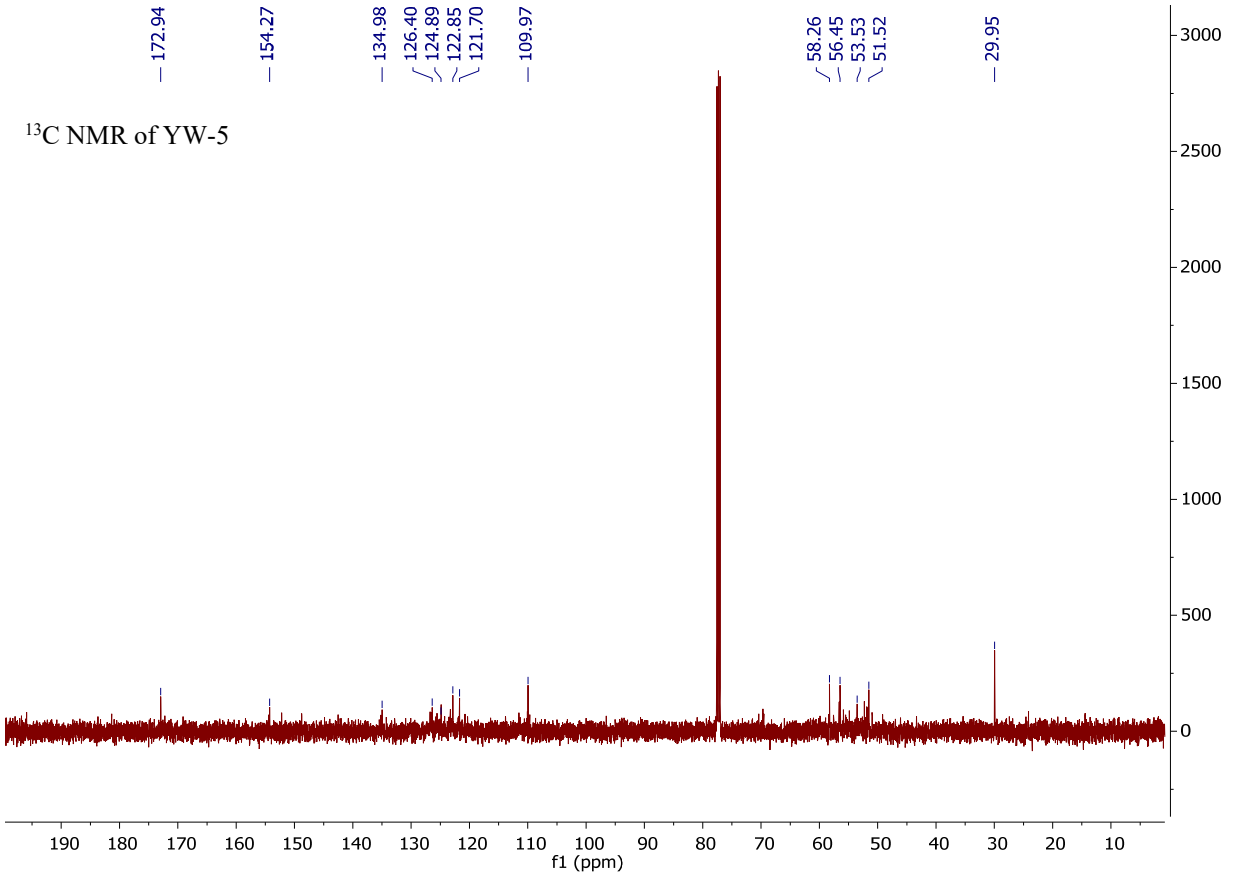
^{13}C NMR of YW-2

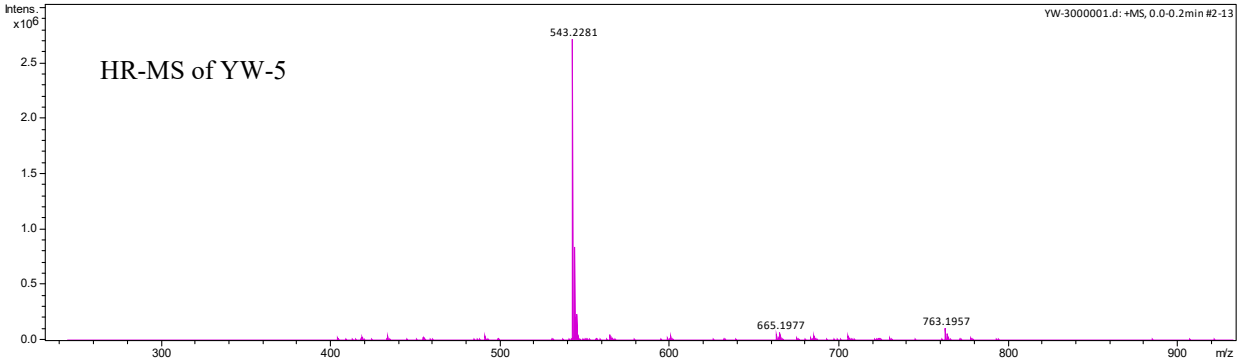


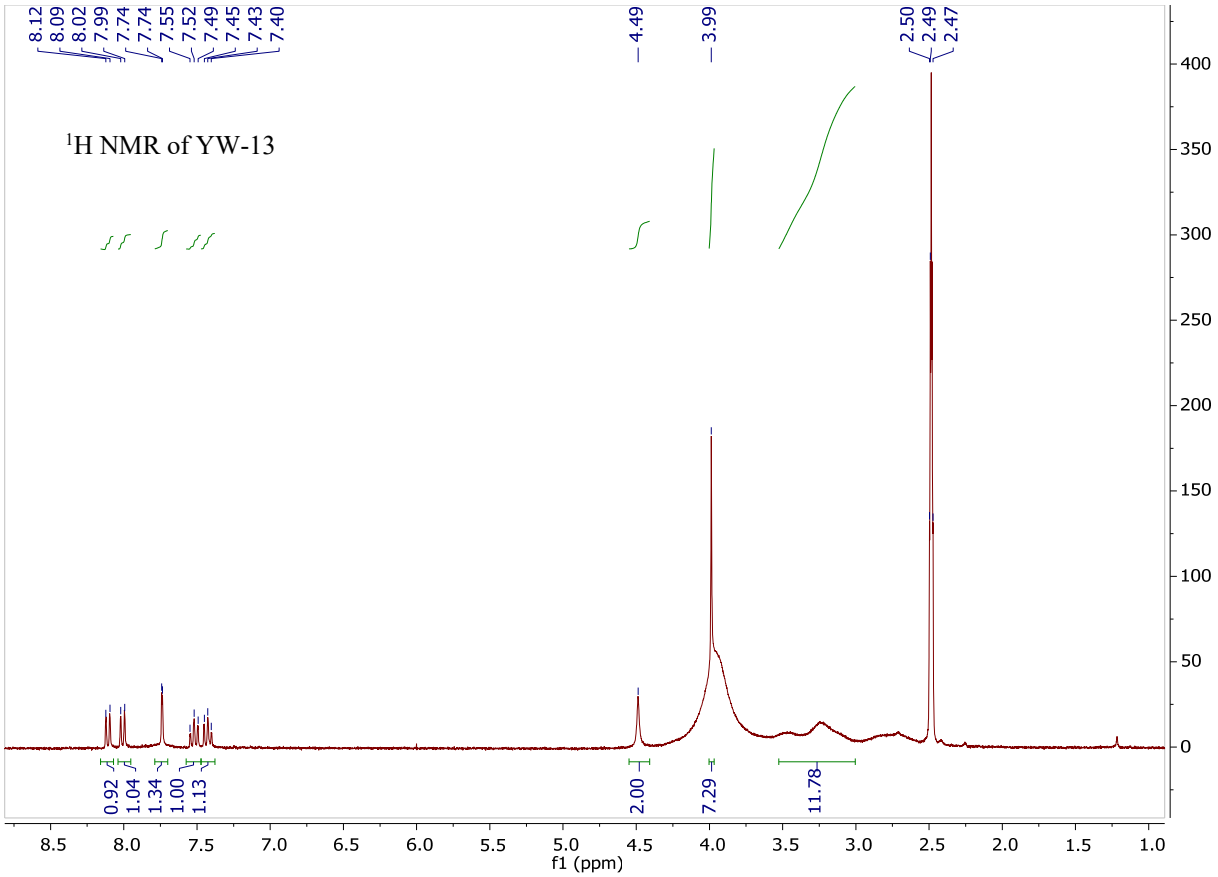
HR-MS of YW-2


8.00
7.97
7.87
7.83
7.54
7.47
7.44
7.42
7.35
7.33
7.30
3.98
3.94
2.77
1.00
0.94
0.96
1.05
1.96
2.92
1.96
5.50
11.95
5.91
f1 (ppm)

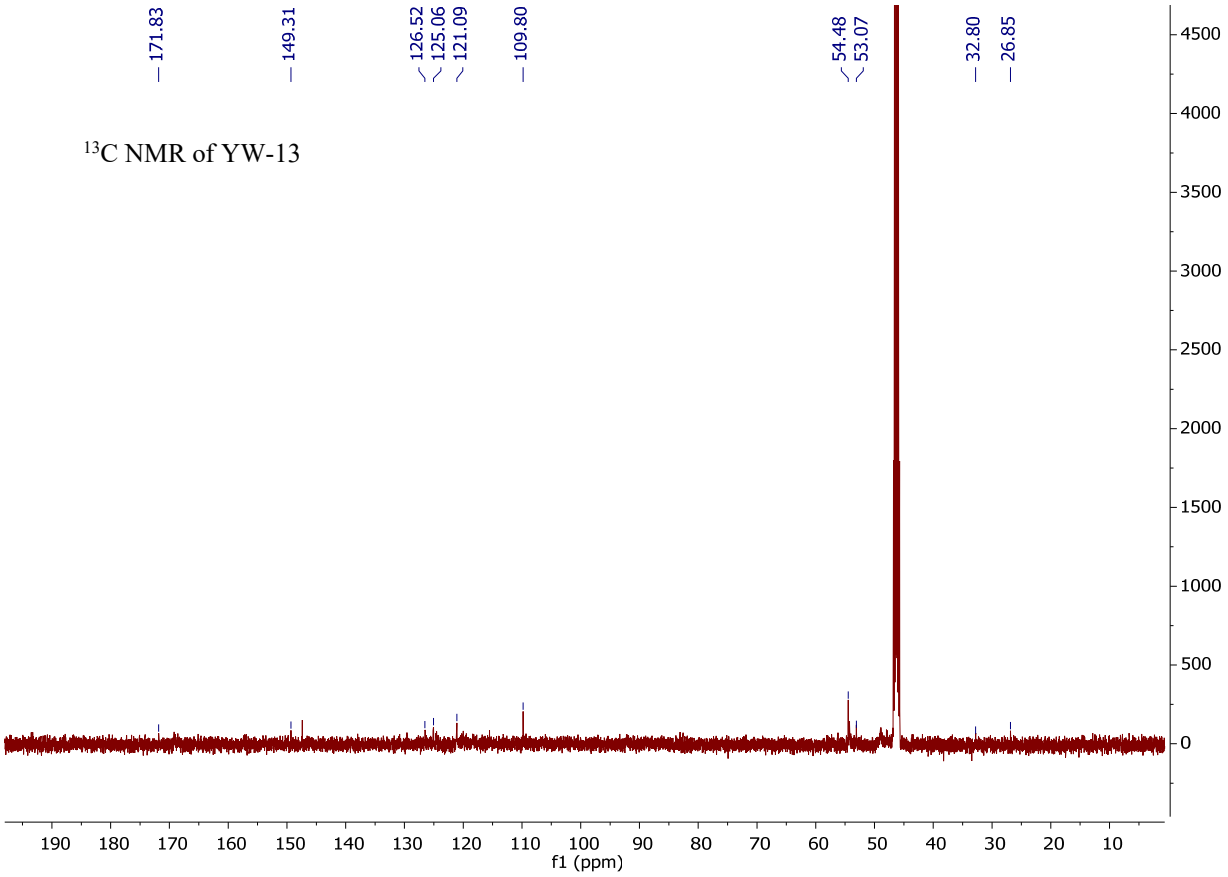
^{1}H NMR of YW-5

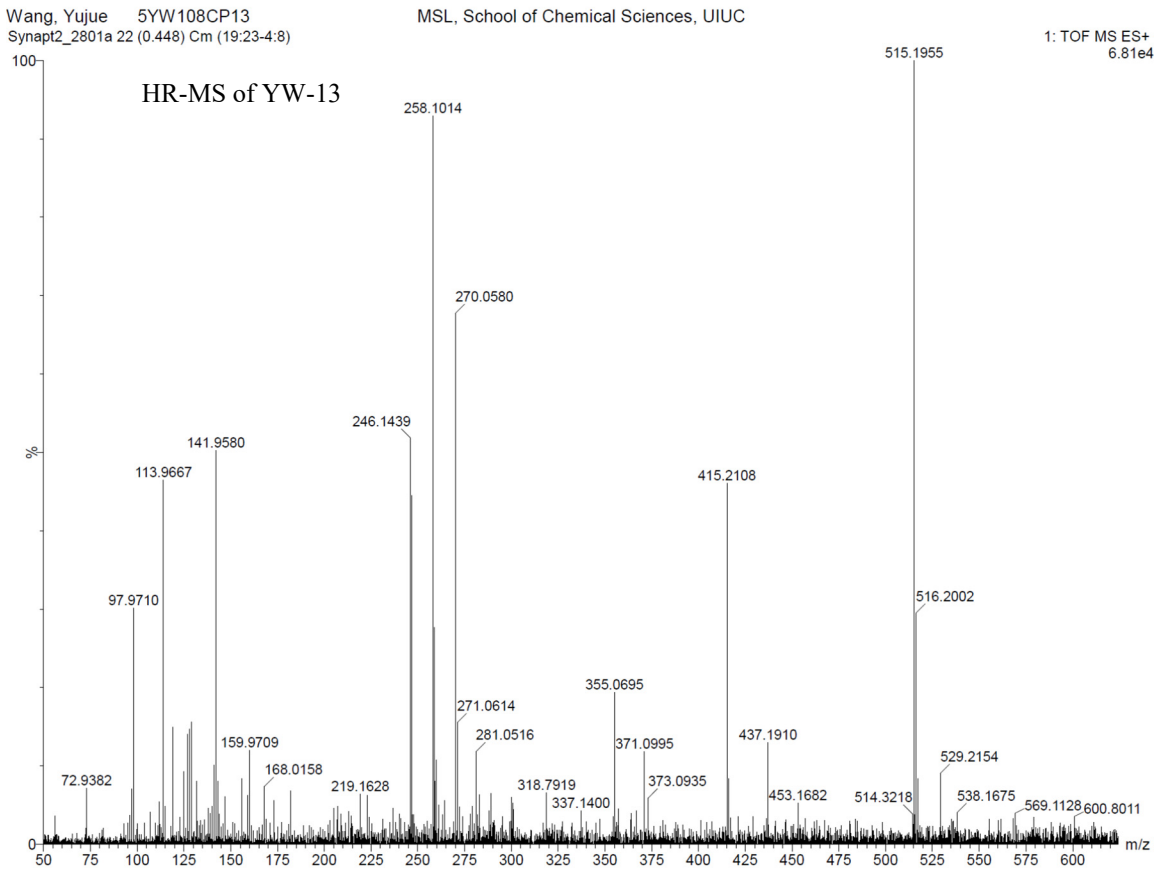

^{13}C NMR of YW-5
172.94
154.27
134.98
126.40
124.89
122.85
121.70
109.97
58.26
56.45
53.53
51.52
29.95
3000
2500
2000
1500
1000
500
0
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


HR-MS of YW-5
Intens.
x10^6
2.5
2.0
1.5
1.0
0.5
0.0
YW-3000001.d: +MS, 0.0-0.2min #2-13
543.2281
665.1977
763.1957
300 400 500 600 700 800 900 m/z


8.12
8.09
8.02
7.99
7.74
7.74
7.55
7.52
7.49
7.45
7.43
7.40
4.49
3.99
2.50
2.49
2.47
1H NMR of YW-13
0.92
1.04
1.34
1.00
1.13
2.00
7.29
11.78
f1 (ppm)

^{13}C NMR of YW-13




Wang, Yujue 5YW108CP13
MSL, School of Chemical Sciences, UIUC
Synapt2_2801a 22 (0.448) Cm (19:23-4:8)
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6.81e4
HR-MS of YW-13
515.1955
258.1014
270.0580
246.1439
141.9580
113.9667
415.2108
97.9710
516.2002
355.0695
271.0614
281.0516
159.9709
371.0995
437.1910
529.2154
168.0158
72.9382
219.1628
318.7919
373.0935
337.1400
453.1682
514.3218
538.1675
569.1128
600.8011
%
m/z

X. References

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