Supporting Information

for

Copper-Free Cycloaddition of Azide and Alkyne in Crystalline State Facilitated by Arene-Perfluoroarene Interaction

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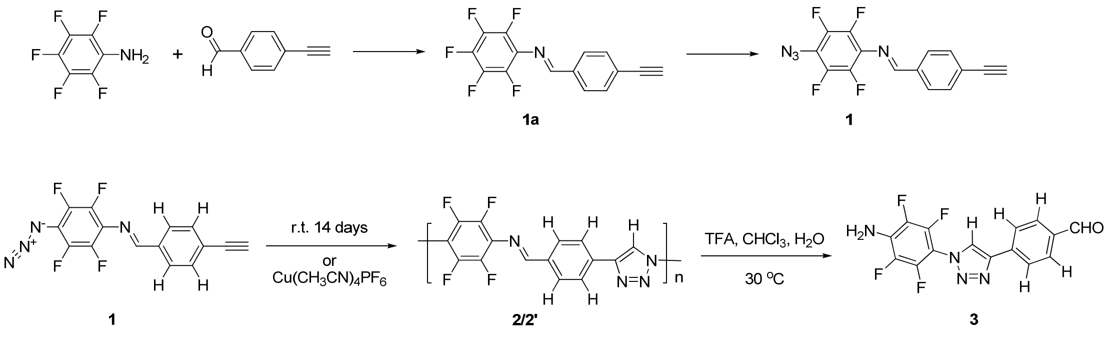
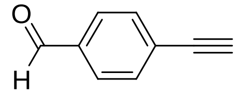
1. Experimental Section

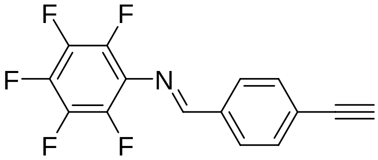
General Methods: Chemicals were purchased commercially and used without further purification. All air and moisture sensitive reactions were performed under a nitrogen atmosphere using standard Schlenk techniques. Tetrahydrofuran (THF) was freshly distilled from sodium under N_2 prior to use. Acetonitrile (CH_3CN) was bubbled with N_2 for 30 minutes before use. *N,N*-Dimethylformamide (DMF) was dried over molecular sieves. ^1H, ^{13}C, and ^{19}F NMR spectra were recorded on Varian Mercury-300 (300 MHz) spectrometer using acetone-d_6 as solvents. Chemical shifts are reported in parts per million (ppm) and coupling constants are reported in hertz (Hz). ^1H NMR chemical shifts were referenced versus TMS (0 ppm), ^{13}C NMR chemical shifts were referenced versus acetone-d_6 (206.68 ppm) and ^{19}F NMR chemical shifts were referenced versus a CF_3CO_2H external standard (0 ppm). Mass spectra were recorded on a VG ZAB-HS mass spectrometer. Elemental analysis was performed using an Elementar VARIO EL elemental analyzer. Melting points were measured on a METTLER TOLEDO Instrument DSC822 calorimeter. Infrared and Raman spectra were recorded in solid phase. Infrared spectra were recorded on a Bio-Rad FTS-65A FT-IR spectrometer. Raman spectra were recorded on a Micro-Raman spectroscopy using a Renishaw System-1000 spectrometer with a 532 nm Yd-YAG laser, CCD detector, and 5x, 20x, 50x, and 80x objective lenses. X-ray diffraction data were collected on a PHILIPS X' Pert Pro diffractometer with an X'celerator detector in the reflection mode at 30 °C, using monochromatized Cu Kα radiation. Single crystal X-ray diffraction data were collected with a NONIUS KappaCCD diffractometer for imine **1**, with graphite monochromator and Mo Kα radiation [λ ($Mo_{K\alpha}$) = 0.71073 Å]. Structures were solved by direct methods with SHELXS-97 and refined against F^2 with SHELXS-97.

Scheme S1. Synthesis of compounds **1**-**3**.

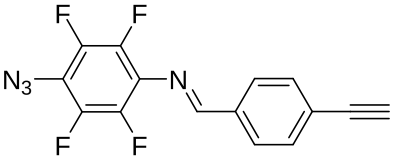




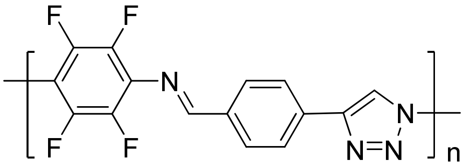
4-Ethynylbenzaldehyde was synthesized according to reference 1.[1]



(*E*)-*N*-(4-Ethynylbenzylidene)-2,3,4,5,6-pentafluorobenzenamine (1a). A solution of 1.30 g (10.0 mmol) of 4-ethynylbenzaldehyde, 1.83 g (10.0 mmol) of 2,3,4,5,6-pentafluoroaniline and 19 mg (0.10 mmol) of *p*-$MePhSO_3H{\cdot}H_2O$ in 50 mL of toluene was refluxed for 8 h. After cooling, the solvent was removed under reduced pressure, and the residue was purified by flash column chromatography over silica gel (petroleum ether/CH_2Cl_2, 3:1 with 1% Et_3N) to afford **1a** as a yellow solid (1.77g, 6.0 mmol, 60%). ^{1}H NMR (300MHz, acetone-d_6, ppm): δ 8.84 (s, 1H), 8.05 (m, 2H), 7.69 (m, 2H), 3.95 (s, 1H). ^{13}C NMR (75 MHz, acetone-d_6, ppm): δ 164.2, 135.5 ($J_{C\text{-}F}$ = 250 Hz), 133.5 ($J_{C\text{-}F}$ = 250 Hz), 130.9, 127.9, 124.7, 122.5, 122.2, 78.1, 76.9. ^{19}F NMR (282 MHz, acetone-d_6, ppm): δ -76.5, -85.6, -87.0. LR-MS (EI, *m/z*): 295 (M^+). Anal. Calcd. for $C_{15}H_6F_5N$: C, 61.03; H, 2.05; N, 4.74. Found: C, 60.89; H, 1.74; N, 4.70. Mp: 112.2 °C (onset).

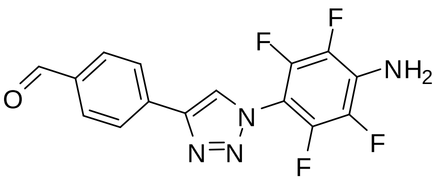


(*E*)-4-Azido-*N*-(4-ethynylbenzylidene)-2,3,5,6-tetrafluorobenzenamine (1). A solution of 1.48 g (5.0 mmol) of **1a** and 0.98 g (15 mmol) of NaN_3 in 75 mL of DMF was heated at 60 °C for 2 h. After cooling, 100 mL of brine was added and the mixture was extracted with CH_2Cl_2. The combined organic phase was dried with Na_2SO_4. After removal of solvent under reduced pressure, the residue was purified by flash column chromatography over silica gel (petroleum ether/CH_2Cl_2, 3:1 with 1% Et_3N) to afford **1** as a yellow solid (1.02 g, 3.2 mmol, 64%). ^{1}H NMR (300 MHz, acetone-d_6, ppm): δ 8.84 (s, 1H), 8.05 (m, 2H), 7.69 (m, 2H), 3.94 (s, 1H). ^{13}C NMR (75 MHz, acetone-d_6, ppm): δ 163.6, 136.6 ($J_{C\text{-}F}$ = 250 Hz), 135.5 ($J_{C\text{-}F}$ = 250 Hz), 131.0, 127.8, 124.6, 122.4, 122.0, 111.4, 78.0, 76.9. ^{19}F NMR (282 MHz, acetone-d_6, ppm): δ -76.3, -76.6. LR-MS (EI, *m/z*): 318 (M^+). Anal. Calcd. for $C_{15}H_6F_4N_4$: C, 56.61; H, 1.90; N, 17.61. Found: C, 56.24; H, 2.18; N, 17.42. Compound **1** decomposes at temperature above 80 °C before reaching its melting point.



Polymer 2. A solid of 500 mg of **1** was kept on shelf in the absence of light for 14 days and the residue was washed by using acetone to afford an insoluble yellow solid (500 mg, 99%).

Polymer 2'. A Schlenk-adapted tube was charged with 318 mg (1.0 mmol) of **1**, 372 mg (1.0 mmol) of $[Cu(CH_3CN)_4][PF_6]$ and 50 mL of THF under nitrogen atmosphere. After stirred for 36 h at room temperature, the mixture was filtrated and the residue was washed with acetone to afford an insoluble yellow solid (95 mg, 30%).



4-(1-(4-Amino-2,3,5,6-tetrafluorophenyl)-1*H*-1,2,3-triazol-4-yl)benzaldehyde (3). **Hydrolysis of polymer 2**: A suspension of 100 mg of polymer **2**, 2 mL of CF_3COOH, 2 mL of water and 100 mL of $CHCl_3$ was stirred under 30 °C for three days. The mixture was filtrated and yellow powder was recovered. A solution of Na_2CO_3 was added to the reaction solution until pH > 7. The resulting mixture was extracted with CH_2Cl_2 and the organic phase was dried with Na_2SO_4. After removal of solvent under reduced pressure, the residue was purified by flash column chromatography over silica gel (CH_2Cl_2, with 1% Et_3N) to afford **3** as a white solid (40 mg, 0.12 mmol, 40%). No triazole **3**'s 1,5-isomer was found.

Hydrolysis of polymer 2': A suspension of 100 mg of polymer **2'**, 2 mL of CF_3COOH, 2 mL of water and 100 mL of $CHCl_3$ was stirred at 30 °C for 1 day until polymer **2'** was totally dissolved. A solution of Na_2CO_3 was added to the reaction solution until pH > 7. The resulting mixture was extracted with CH_2Cl_2 and the organic phase was dried with Na_2SO_4. After removal of solvent under reduced pressure, the residue was purified by flash column chromatography over silica gel (CH_2Cl_2, with 1% Et_3N) to afford **3** as a white solid (91 mg, 0.27 mmol, 90%). Trace amount of 4-ethynylbenzaldehyde in the hydrolysis product coming from the terminal group of polymer **2**' was found. The different hydrolytic reactivity of polymer **2** and polymer **2'** is likely a result of different molecular weight and crystallinity of **2** and **2'**.

^{1}H NMR (300 MHz, acetone-d_6, ppm): δ 10.10 (s, 1H), 8.90 (s, 1H), 8.23 (m, 2H), 8.06 (m, 2H), 6.06 (s, 2H). ^{13}C NMR (75 MHz, acetone-d_6, ppm): δ 186.9, 141.6, 138.2 ($J_{C\text{-}F}$ = 250 Hz), 131.9, 131.4 ($J_{C\text{-}F}$ = 250 Hz), 131.3, 126.0, 125.6, 121.5, 120.7, 98.5. ^{19}F NMR (282 MHz, acetone-d_6, ppm): δ -74.3, -85.1. LR-MS (EI, *m/z*): 308 (M^+-28). Anal. Calcd. for $C_{15}H_8F_4N_4O$: C, 53.58; H, 2.40; N, 16.66. Found: C, 53.40; H, 2.56; N, 16.77. Mp: 220.2 °C (onset).

2. ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra

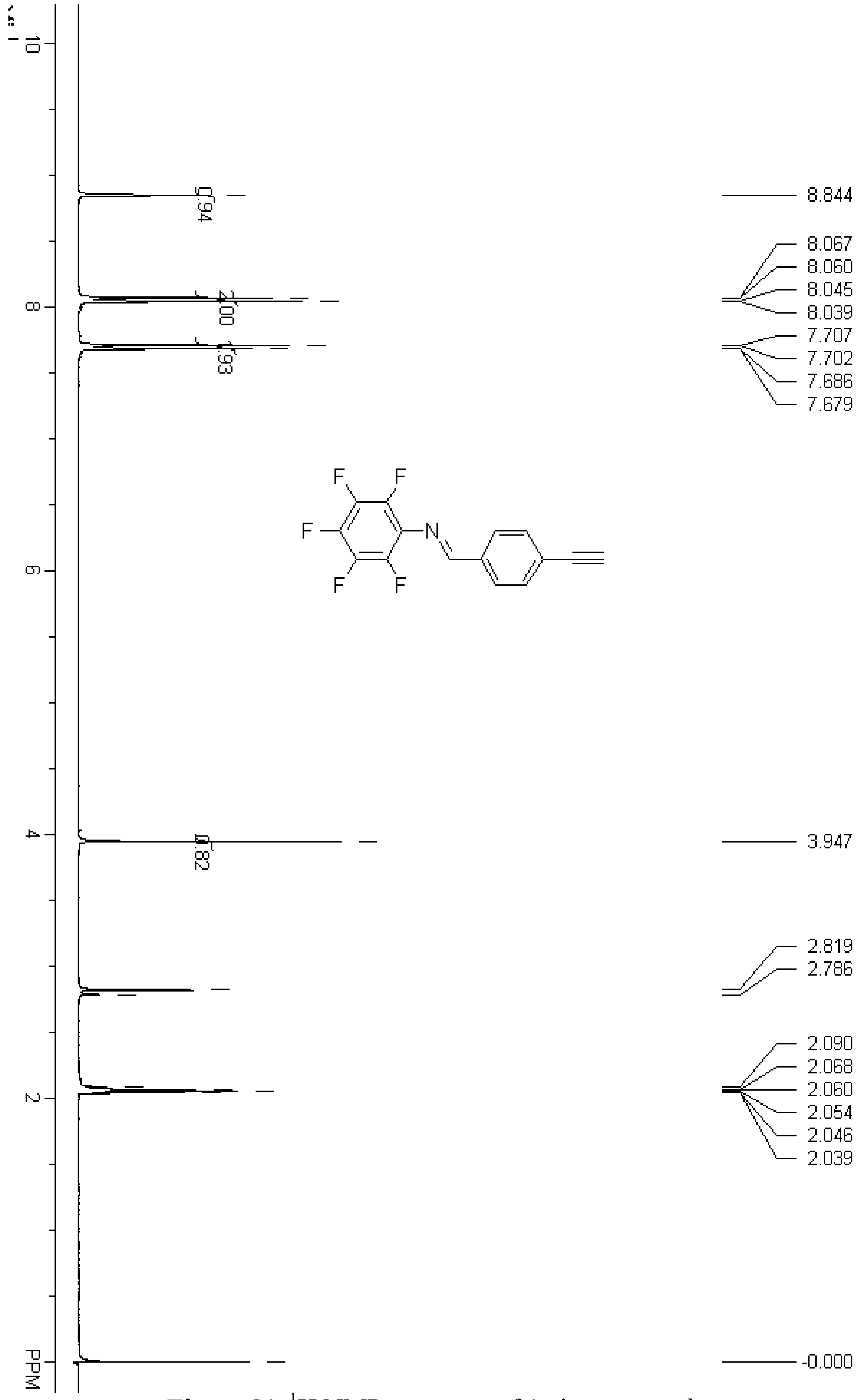


Figure S1. ^{1}H NMR spectrum of **1a** in acetone-d_6

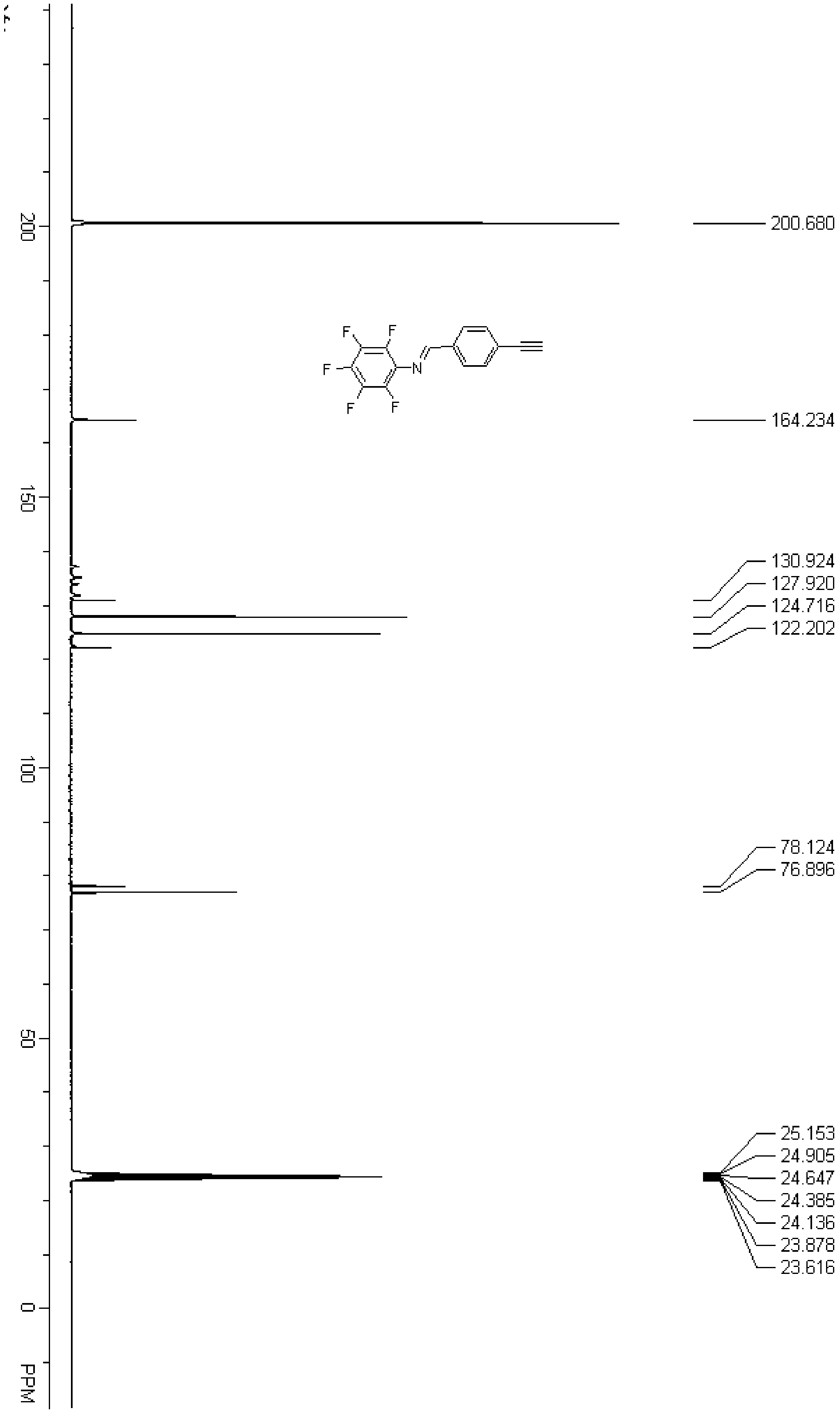


Figure S2. ^{13}C NMR spectrum of **1a** in acetone-d_6

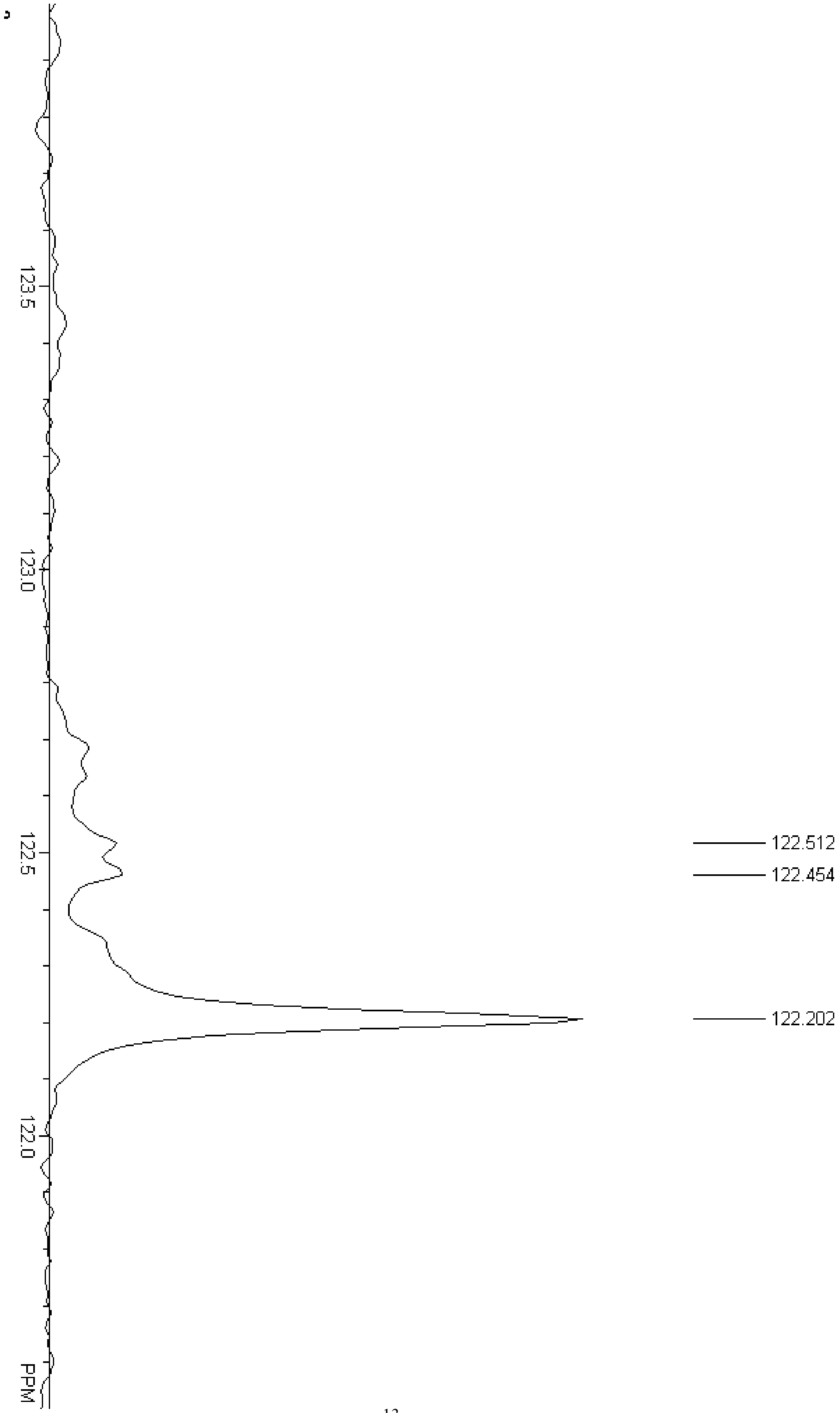


Figure S3. Aromatic region of ^{13}C NMR spectrum of **1a** in acetone-d_6

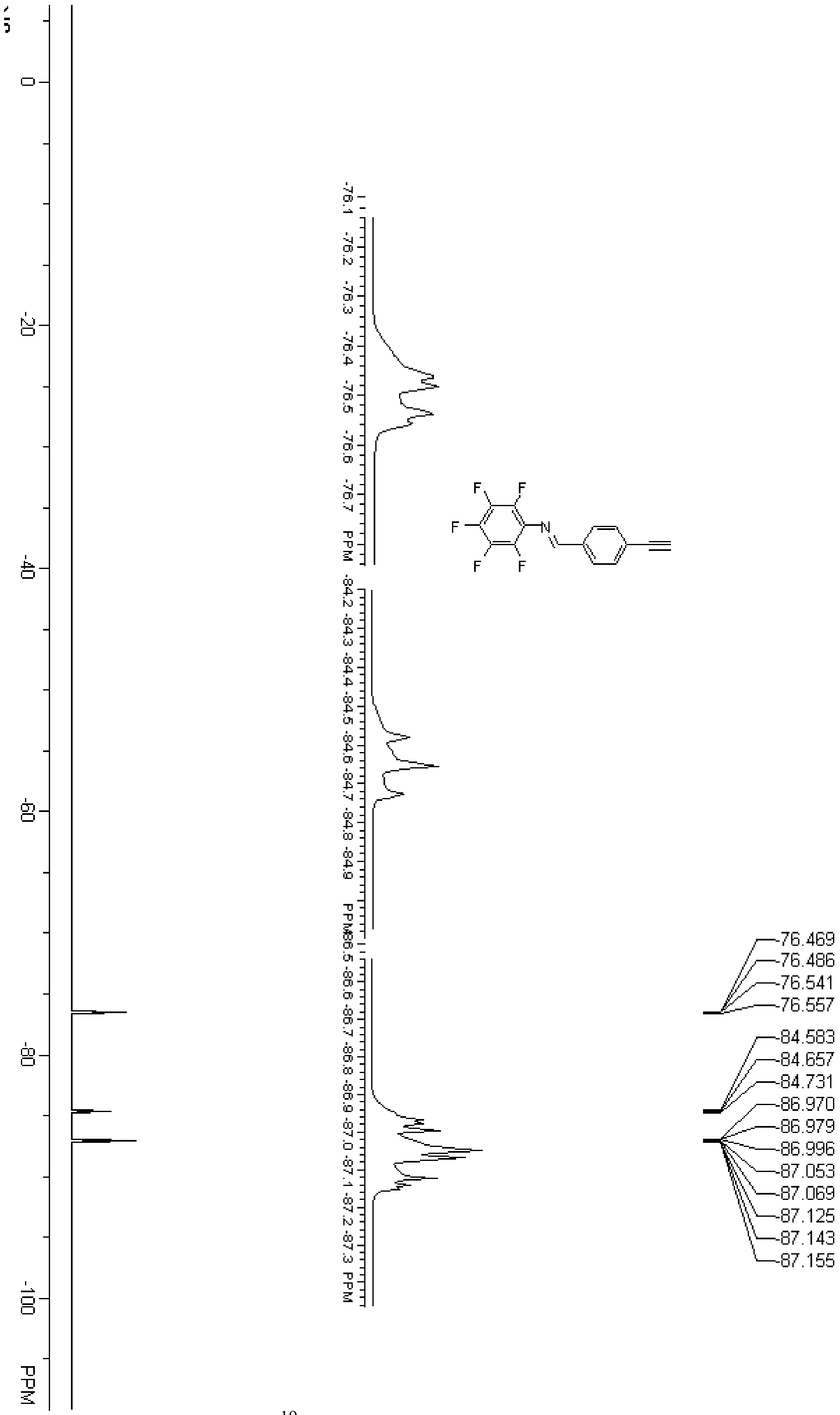


Figure S4. ^{19}F NMR spectrum of **1a** in acetone-d_6

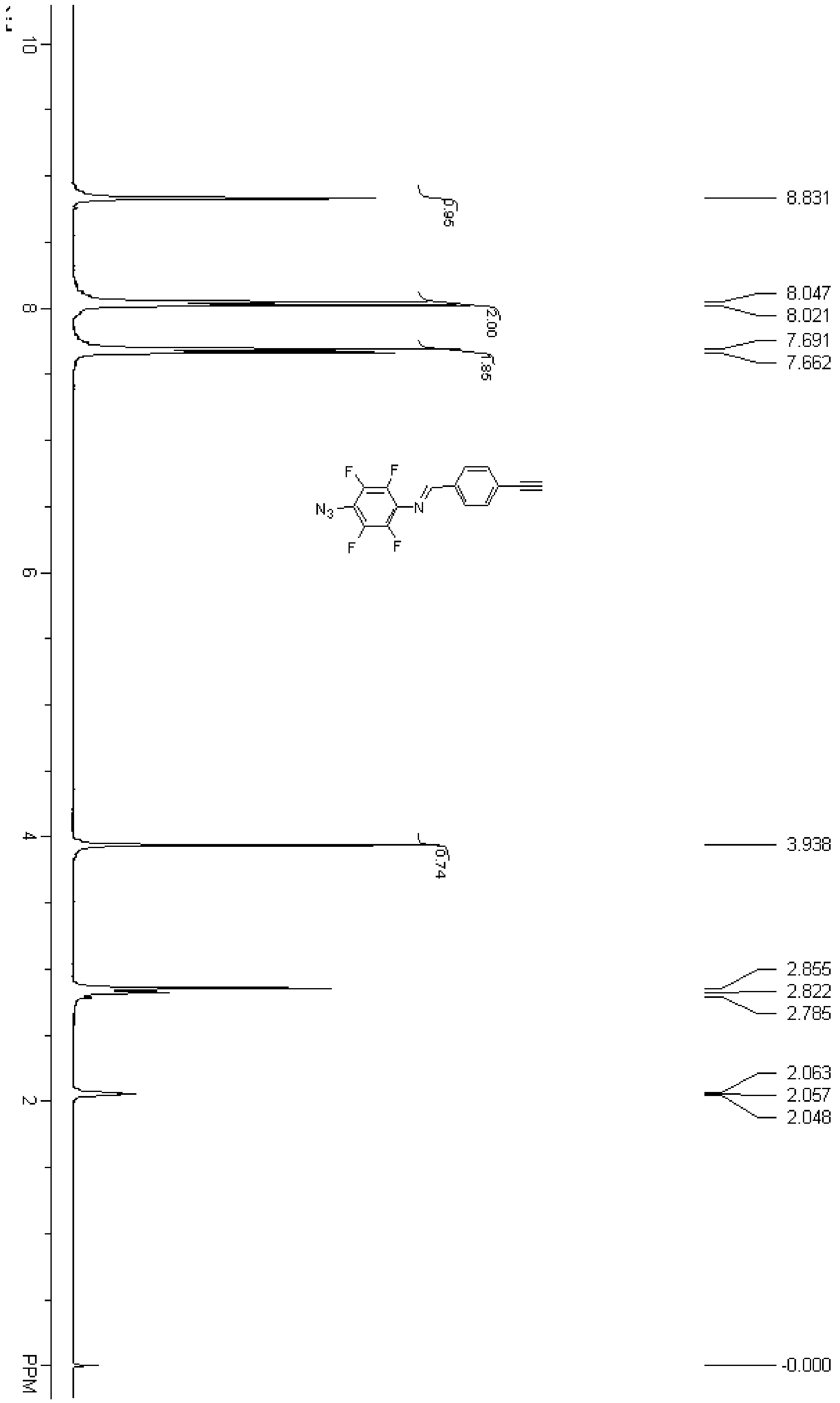


Figure S5. ^{1}H NMR spectrum of **1** in acetone-d_6

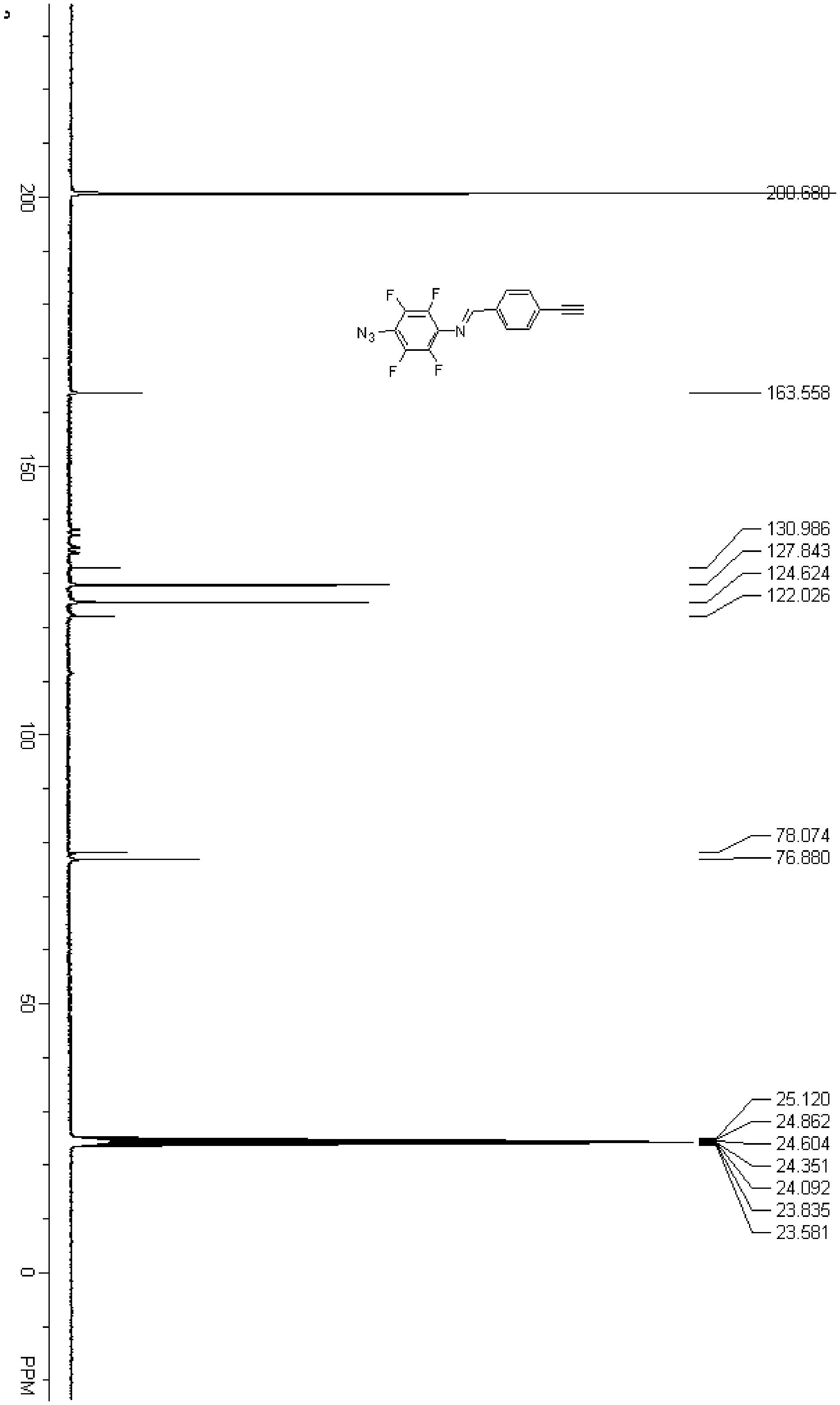


Figure S6. ^{13}C NMR spectrum of **1** in acetone-d_6

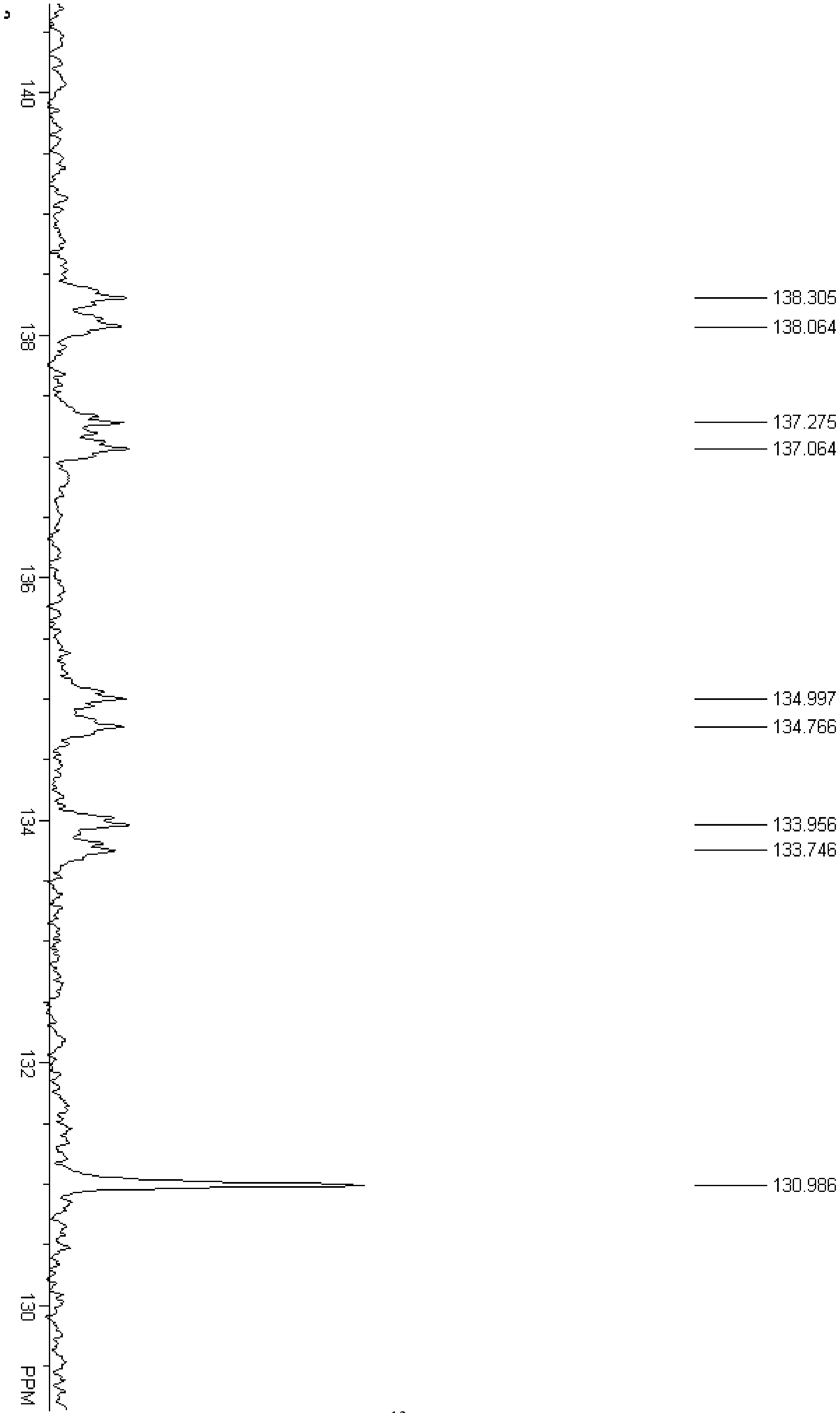


Figure S7. Aromatic region of ^{13}C NMR spectrum of **1** in acetone-d_6

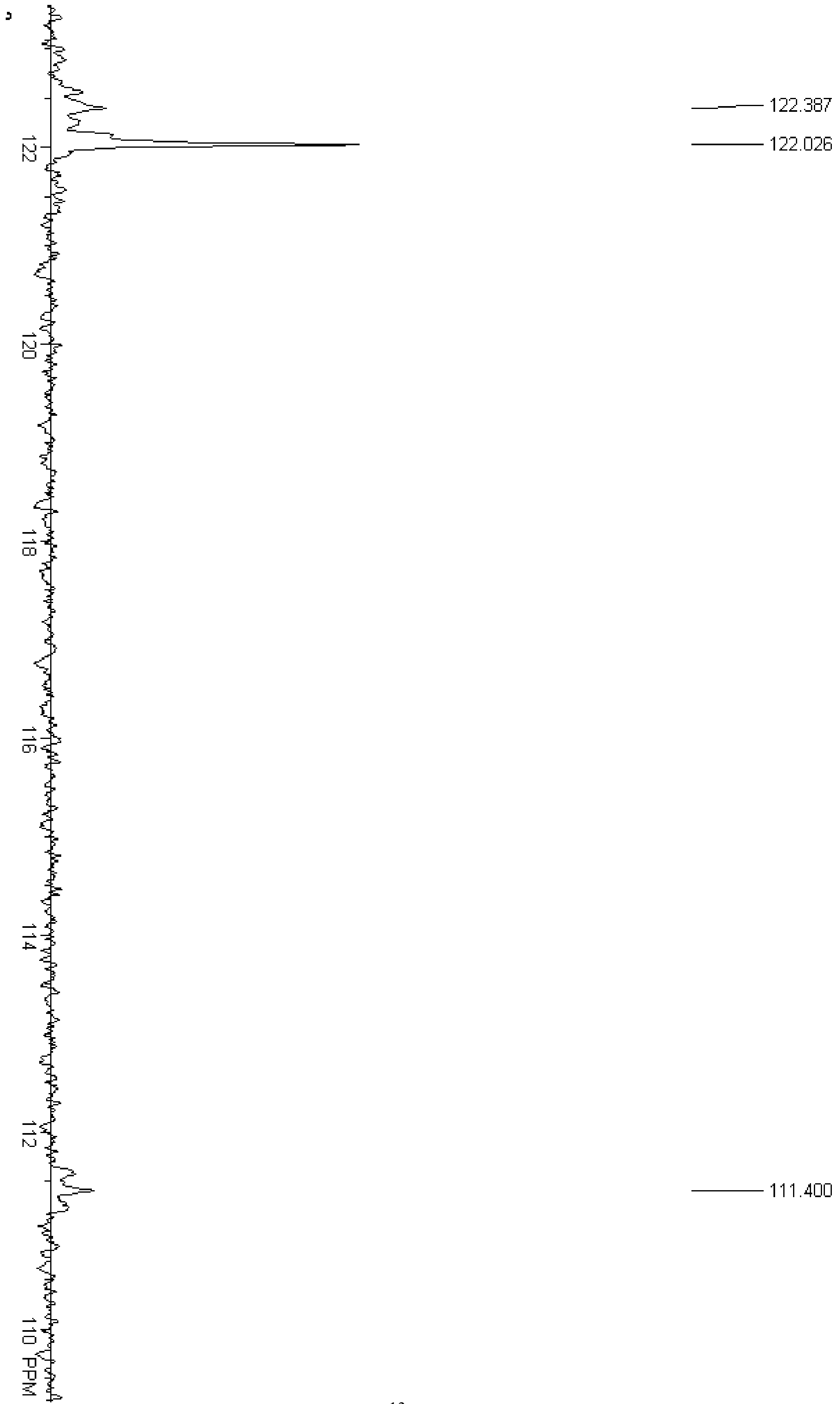


Figure S8. Aromatic region of ^{13}C NMR spectrum of **1** in acetone-d_6

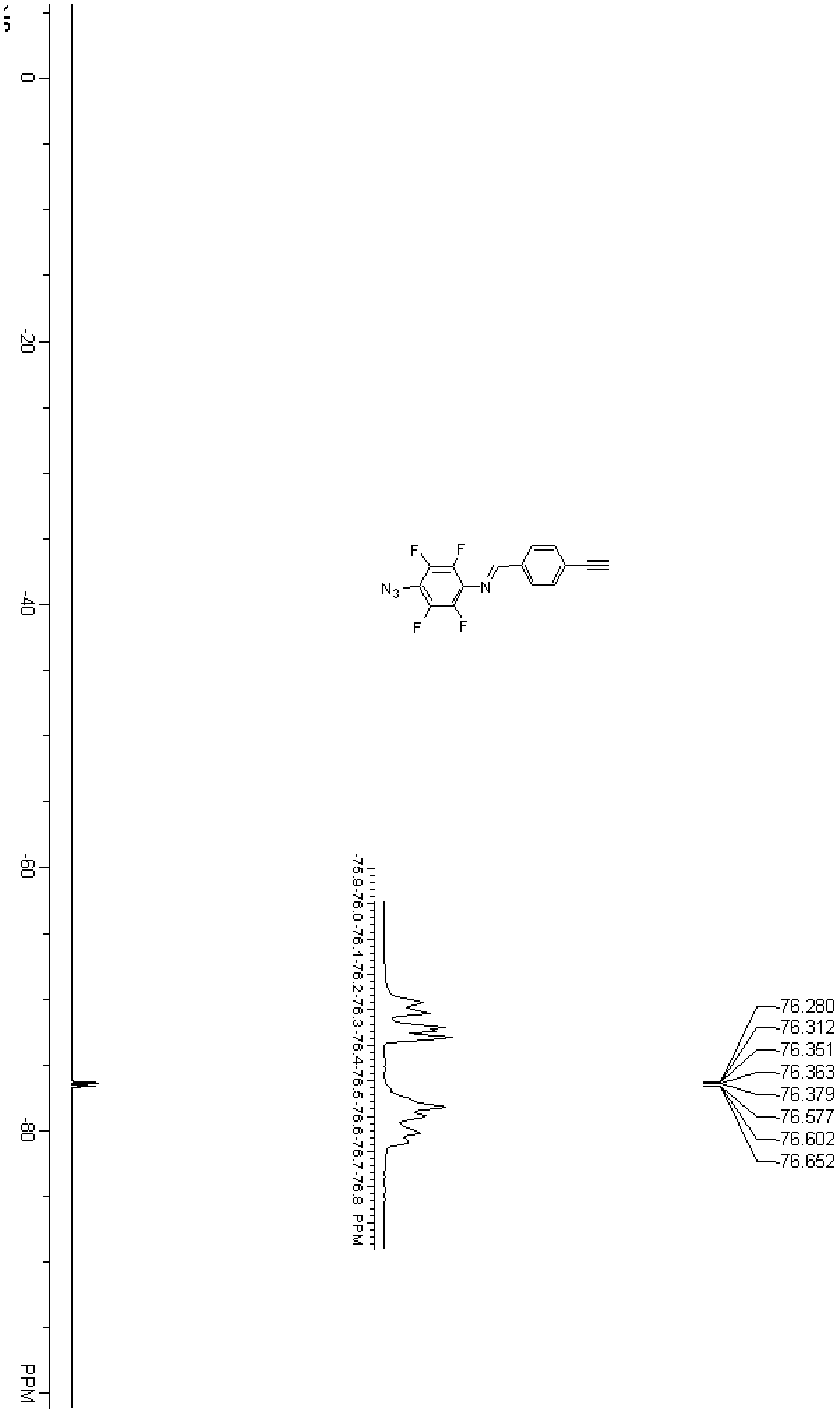


Figure S9. ^{19}F NMR spectrum of **1** in acetone-d_6

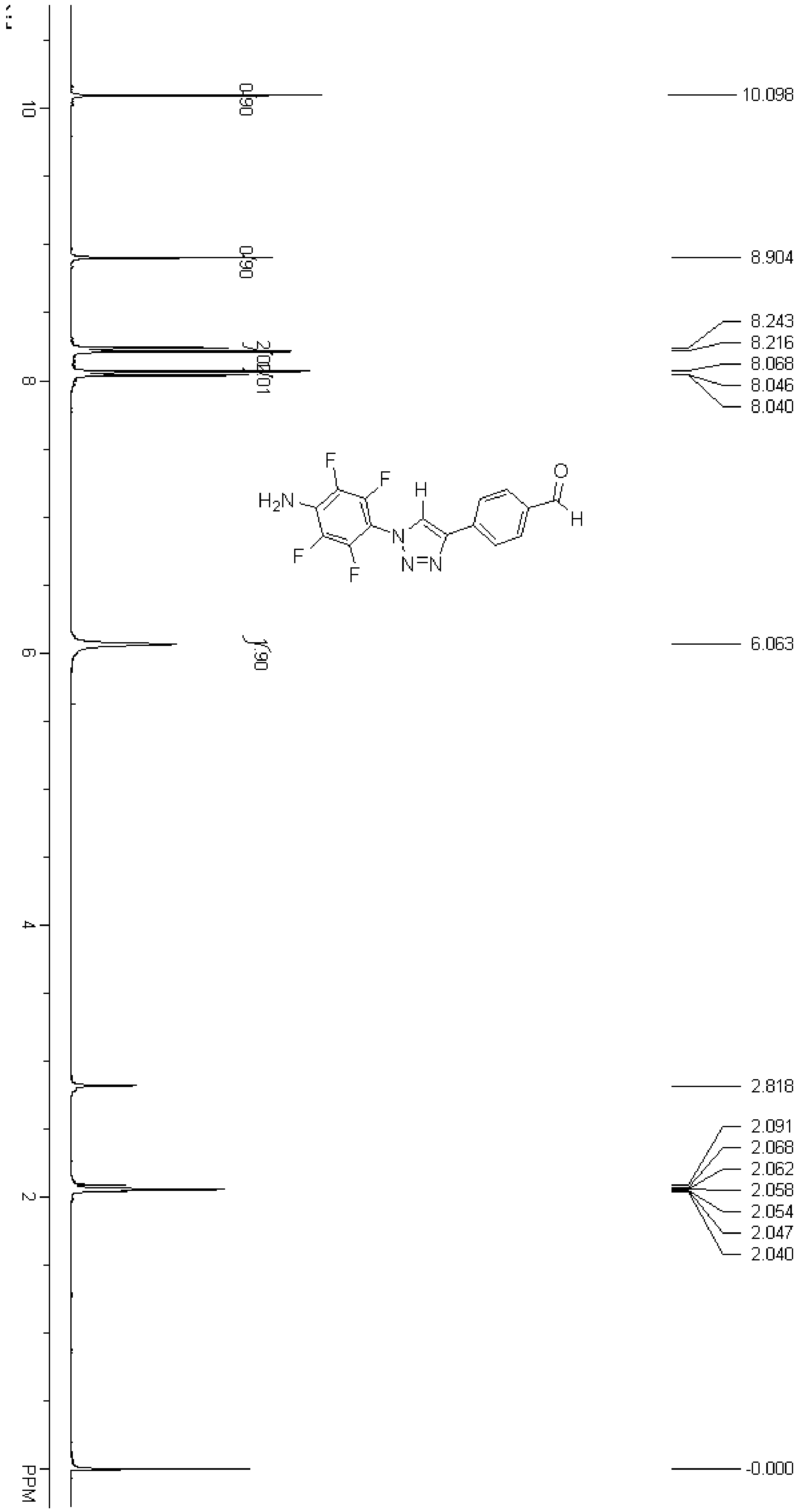


Figure S10. ^{1}H NMR spectrum of **3** in acetone-d_6

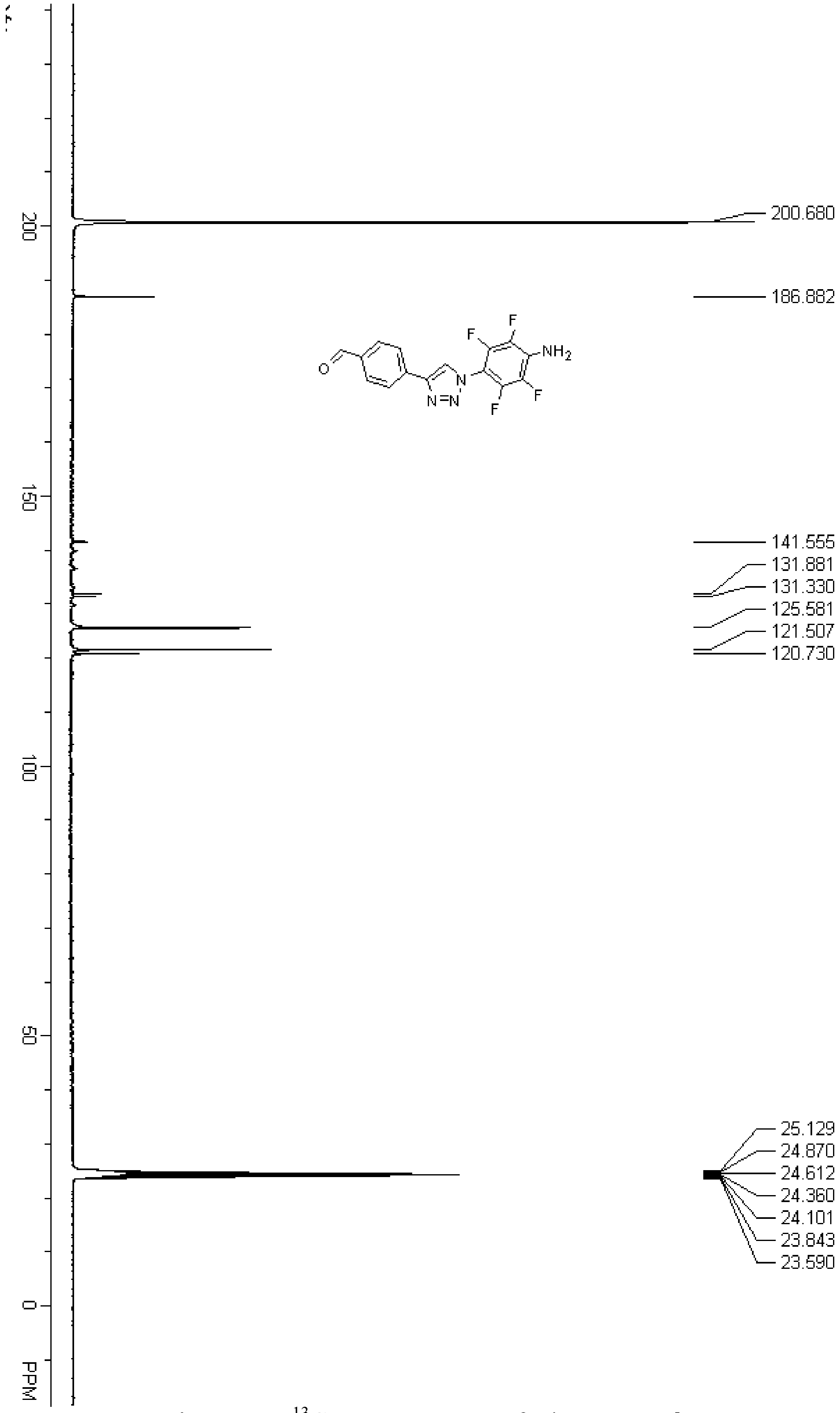


Figure S11. ^{13}C NMR spectrum of **3** in acetone-d_6

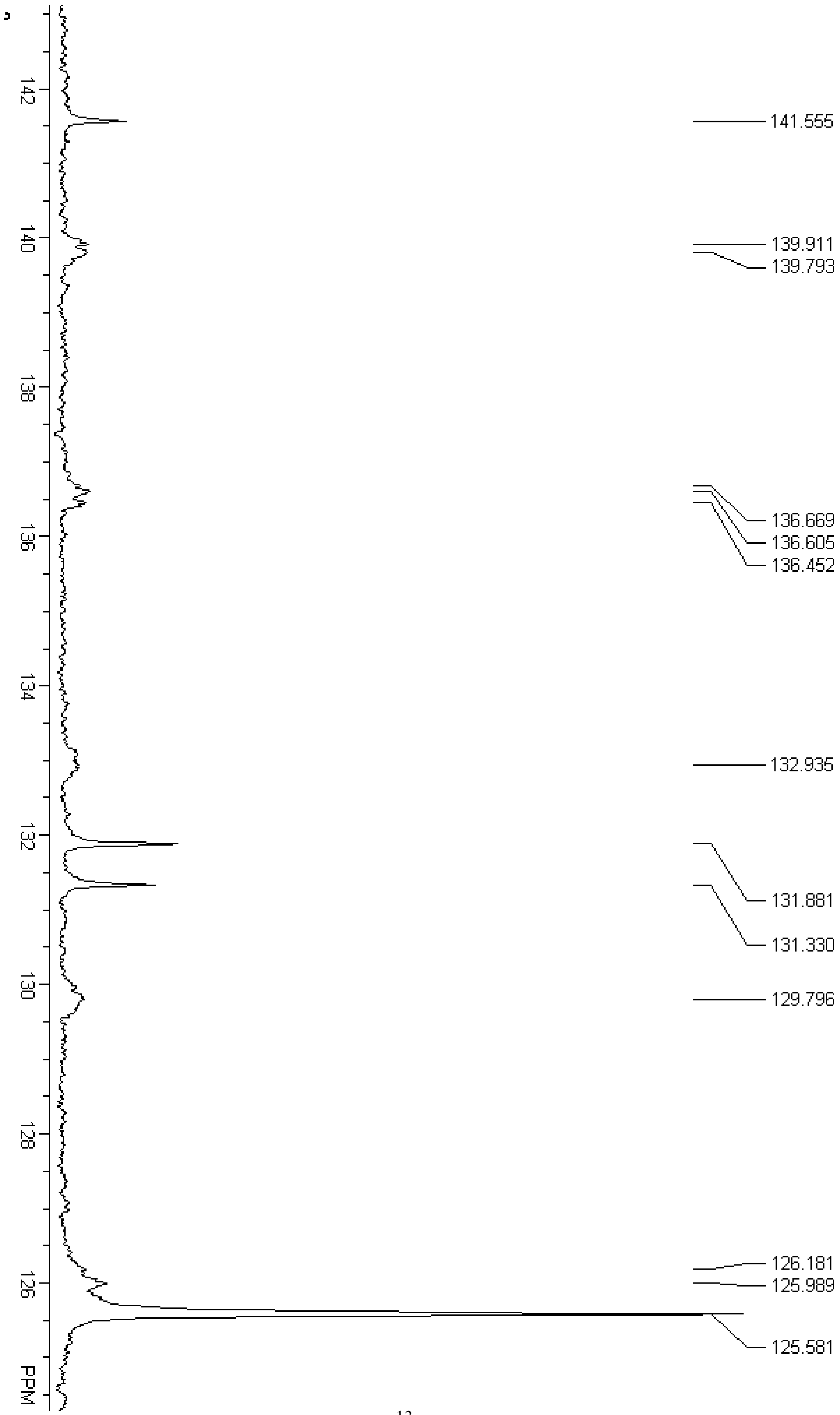


Figure S12. Aromatic region of ^{13}C NMR spectrum of **3** in acetone-d_6

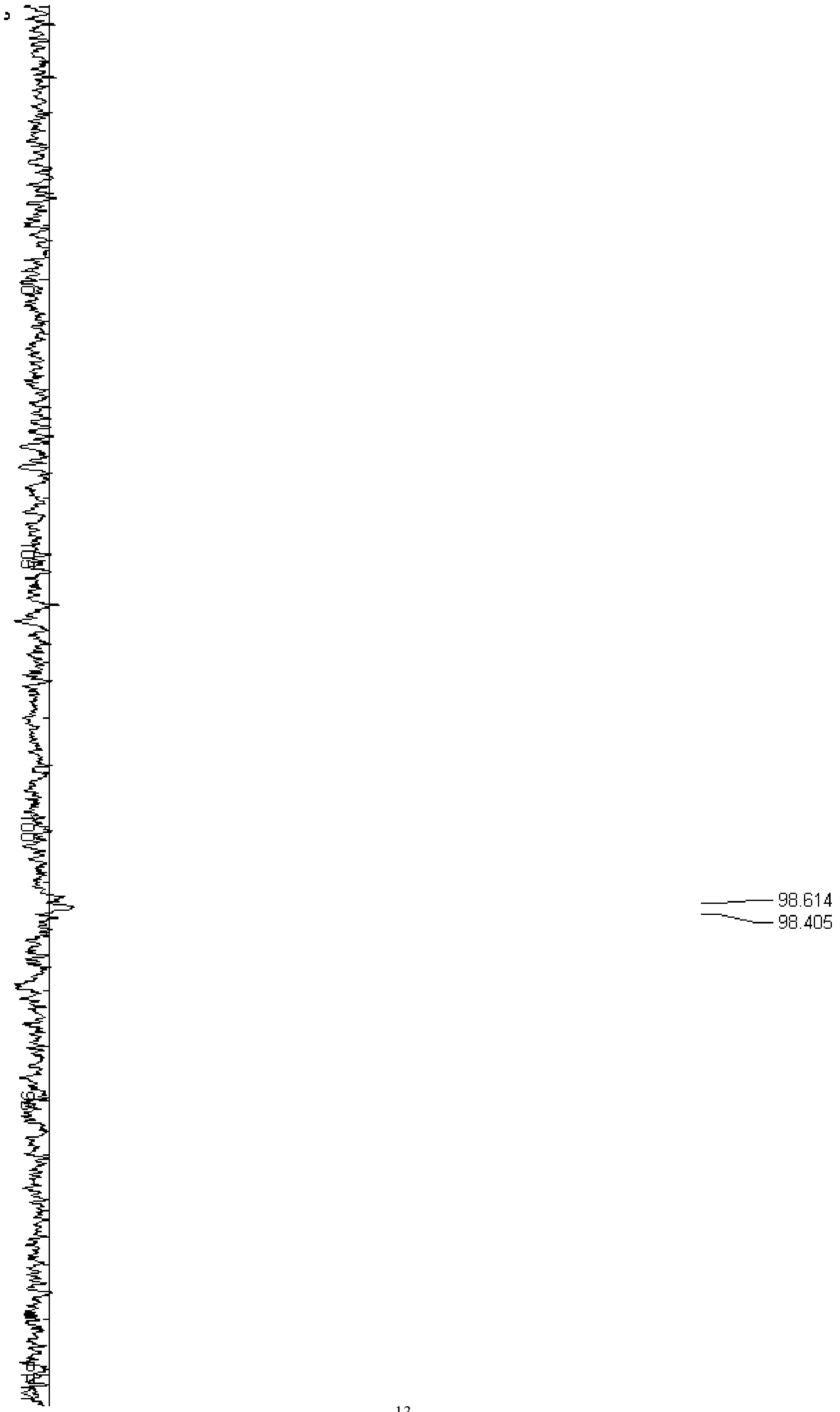


Figure S13. Aromatic region of ^{13}C NMR spectrum of **3** in acetone-d_6

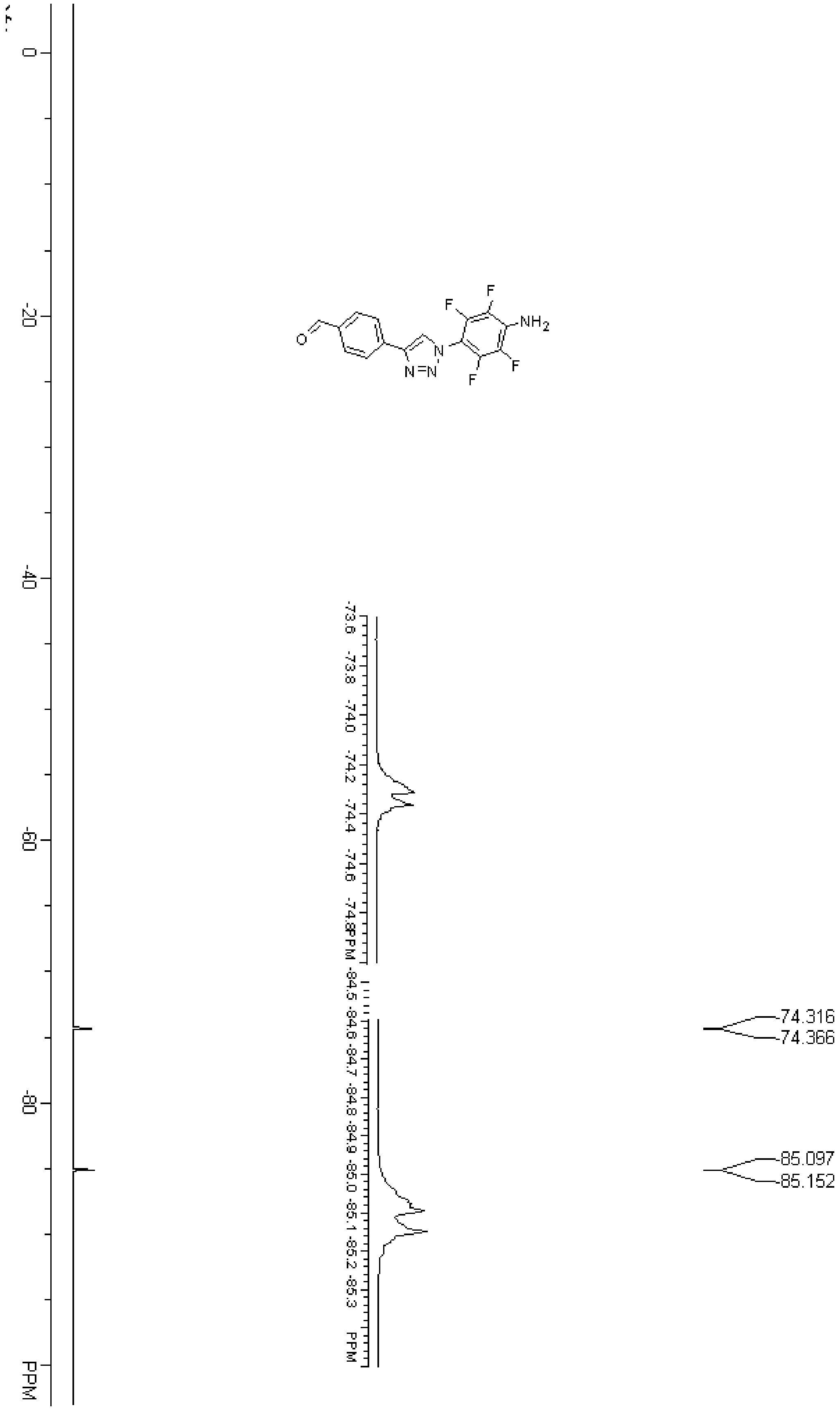


Figure S14. ^{19}F NMR spectrum of **3** in acetone-d_6

3. FT-IR and Raman Spectra

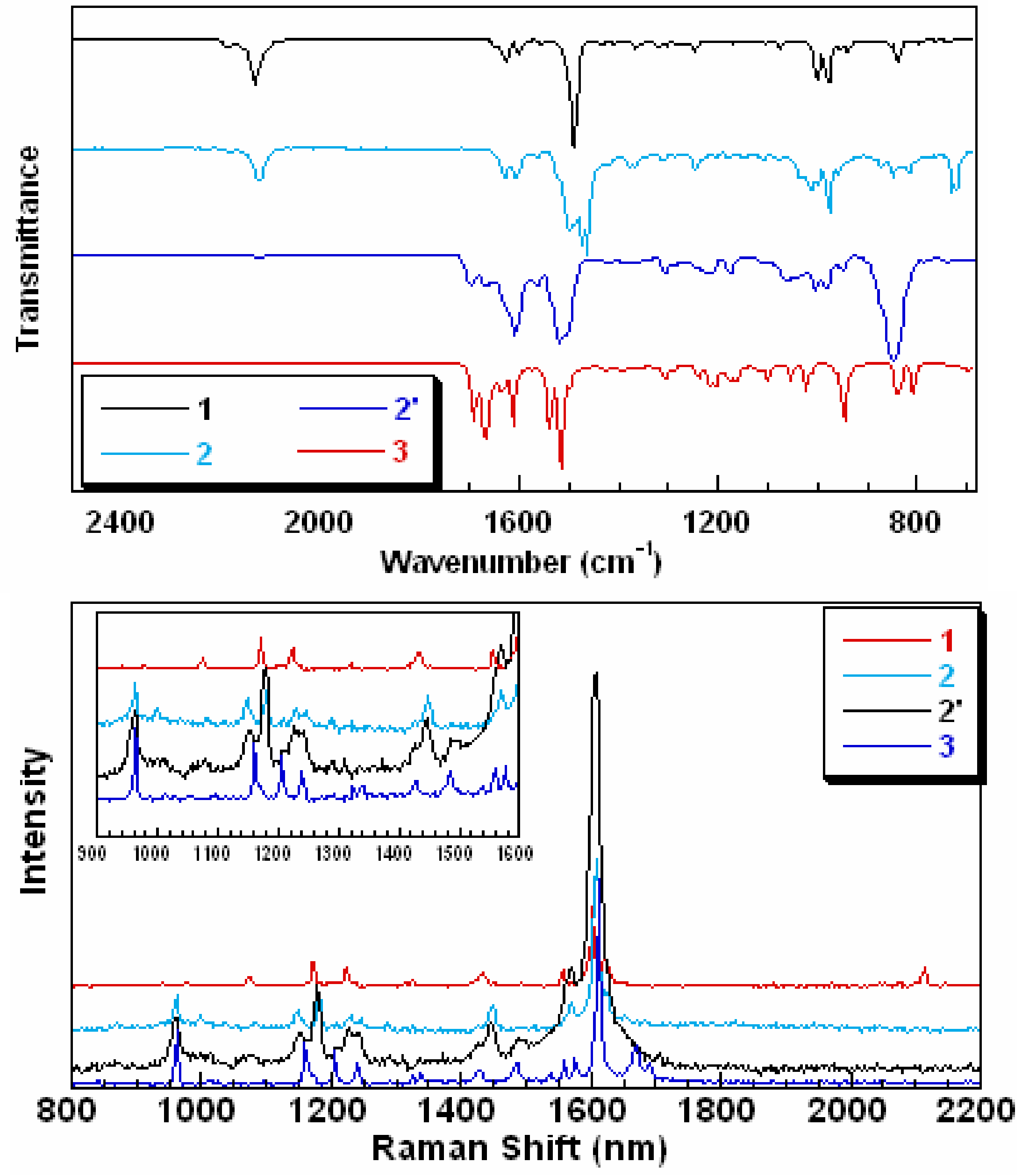


Figure S15. FT-IR spectra (top) and Raman spectra (bottom) of imine **1**, polymer **2**, control polymer **2'** and triazole **3**.

FT-IR spectra of **1**, **2**, **2'** and **3** indicate that after 1,3-dipolar cycloaddition of azide and alkyne in crystal (**2**) and catalyzed by Cu(I) in solution (**2**'), the signal of azide stretching mode decreased in intensity and disappeared, respectively. The residue signal at 2133 cm^{-1} of polymer **2** came from the end group of resulting insoluble polymer. In the FT-IR spectrum of **3**, two bands emerged at 807 and 838 cm^{-1} that can be assigned to triazole. Raman spectrum of polymer **2** showed a characteristic band at 962 cm^{-1}, which can be assigned as in-plane ring bending band of triazole ring.[2,3] Control polymer **2'** and hydrolysis product **3** also showed similar bands at 962 and 963 cm^{-1} in their Raman spectra, respectively, which reveals that 1,3-dipolar cycloaddition of azide and alkyne proceeded in the crystal of imine **1** and formed polymer **2**. However, the characteristic band at about 963 cm^{-1} in the Raman spectra of **2**, **2'** and **3** cannot provide distinction of 1,4-triazole and 1,5-triazole.

4. X-ray Diffraction Patterns

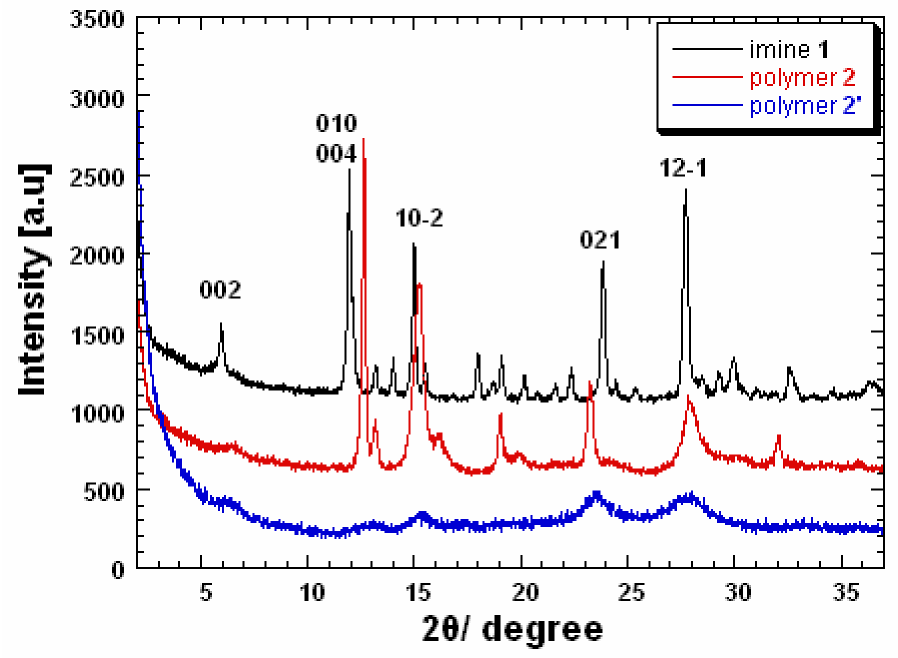


Figure S16. X-ray diffraction patterns of imine **1**, polymer **2** (obtained from **1** in crystals) and polymer **2'** (obtained from **1** in THF, catalyzed by $Cu(CH_3CN)_4PF_6$). Strong diffraction peaks of imine **1** were assigned in the figure.

XRD pattern of imine **1** shows multiple diffraction peaks in the 2θ range of 2 to 37 degree. Polymer **2** obtained from imine **1** in crystal also gave strong diffraction. The similarity of XRD patterns of **1** and polymer **2** indicates that the crystal parameter did not change significantly after 1,3-dipolar cycloaddition. Although polymer **2'** gave low diffraction signal probably due to poor degree of crystallinity, it gave four diffraction peaks consistent with those of polymer **2**. The broad peak at ca. 6^{o} was from aluminium foil.

5. Crystal Data and Structure Refinement

Table S1-1. Crystal data and structure refinement for imine **1**.

Identification code	Imine **1**	
Empirical formula	$C_{15}H_6F_4N_4$	
Formula weight	318.24	
Temperature	113(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 6.1856(12) Å	α = 90°
	b = 7.2446(14) Å	β = 92.57(3)°
	c = 29.281(6) Å	γ = 90°
Volume	1310.8(4) Å^3	
Z	4	
Density (calculated)	1.613 Mg/m^3	
Absorption coefficient	0.141 mm^{-1}	
F(000)	640	
Crystal size	0.18 x 0.12 x 0.10 mm^3	
Theta range for data collection	1.39 to 25.02°	
Index ranges	$-6 \le h \le 7$, $-8 \le k \le 8$, $-34 \le l \le 34$	
Reflections collected	9472	
Independent reflections	2317 [R(int) = 0.043]	
Completeness to theta = 25.02°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9861 and 0.9751	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2317 / 0 / 208	
Goodness-of-fit on F^2	1.08	
Final R indices [I>2sigma(I)]	R1 = 0.0450, wR2 = 0.1170	
R indices (all data)	R1 = 0.0545, wR2 = 0.1244	
Largest diff. peak and hole	0.209 and -0.235 e. Å^{-3}	

Table S1-2. Atomic coordinates (× 10^4) and equivalent isotropic displacement parameters ($Å^2$ × 10^4) for imine **1** at 113K. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
F(1)	0.98231(17)	0.54116(16)	0.30217(4)	0.0290(3)
F(2)	1.10399(17)	0.54625(16)	0.39079(4)	0.0294(3)
F(3)	0.44329(18)	0.26584(16)	0.42653(3)	0.0293(3)
F(4)	0.31940(17)	0.26381(15)	0.33829(3)	0.0260(3)
N(1)	0.8680(3)	0.4072(3)	0.45636(5)	0.0300(4)
N(2)	0.7488(3)	0.3713(3)	0.48857(6)	0.0340(5)
N(3)	0.6691(3)	0.3429(3)	0.52161(6)	0.0533(6)
N(4)	0.6068(3)	0.3880(2)	0.26939(5)	0.0235(4)
C(1)	0.6473(3)	0.4026(3)	0.31691 (6)	0.0221(4)
C(2)	0.8469(3)	0.4710(3)	0.33265(6)	0.0225(4)
C(3)	0.9102(3)	0.4738(3)	0.37803(6)	0.0233(5)
C(4)	0.7785(3)	0.4048(3)	0.41129(6)	0.0237(5)
C(5)	0.5781(3)	0.3372(3)	0.39620(6)	0.0235(5)
C(6)	0.5138(3)	0.3378(3)	0.35041(6)	0.0221(4)
C(7)	0.4214(3)	0.4316(3)	0.25199(6)	0.0232(5)
H(7)	0.3154	0.4745	0.2709	0.028*
C(8)	0.3721(3)	0.4155(3)	0.20273(6)	0.0227(4)
C(9)	0.1676(3)	0.4666(3)	0.18518(7)	0.0256(5)
H(9)	0.0632	0.5064	0.2049	0.031*
C(10)	0.1189(3)	0.4582(3)	0.13856(6)	0.0265(5)
H(10)	0.0177	0.4929	0.1270	0.032*
C(11)	0.2740(3)	0.3980(3)	0.10903(6)	0.0242(5)
C(12)	0.4786(3)	0.3443(3)	0.12652(6)	0.0252(5)
H(12)	0.5824	0.3029	0.1069	0.030*
C(13)	0.5258(3)	0.3530(3)	0.17302(6)	0.0236(4)
H(13)	0.6617	0.3168	0.1846	0.028*
C(14)	0.2293(3)	0.3900(3)	0.06010(7)	0.0299(5)
C(15)	0.2031(4)	0.3809(3)	0.02020(7)	0.0404(6)
H(15	0.1824	0.3736	0.0114	0.048*

Table S1-3. Bond lengths [Å] and angles [°] for imine **1** at 113K.

F1—C2	1.350(2)	C7—C8	1.466(3)
F2—C3	1.346(2)	C7—H7	0.9300
F3—C5	1.348(2)	8—C13	1.393(3)
F4—C6	1.349(2)	C8—C9	1.394(3)
N1—N2	1.250(2)	C9—C10	1.386(3)
N1—C4	1.408(2)	C9—H9	0.9300
N2—N3	1.124(2)	C10—C11	1.390(3)
N4—C7	1.274(3)	C10—H10	0.9300
N4—C1	1.407(2)	C11—C12	1.399(3)
C1—C2	1.390(3)	11—C14	1.448(3)
C1—C6	1.392(3)	C12—C13	1.381(3)
C2—C3	1.369(3)	C12—H12	0.9300

C3—C4	1.391(3)	C13—H13	0.9300
C4—C5	1.386(3)	C14—C15	1.174(3)
C5—C6	1.382(3)	C15—H15	0.9300
N2—N1—C4	118.96(17)	N4—C7—H7	119.4
N3—N2—N1	169.5(2)	C8—C7—H7	119.4
C7—N4—C1	119.60(17)	13—C8—C9	119.33(17)
C2—C1—C6	115.66(17)	C13—C8—C7	121.51(17)
C2—C1—N4	118.09(17)	C9—C8—C7	119.15(17)
C6—C1—N4	125.93(18)	C10—C9—C8	120.33(18)
F1—C2—C3	118.48(17)	C10—C9—H9	119.8
F1—C2—C1	118.91(16)	C8—C9—H9	119.8
C3—C2—C1	122.59(18)	C9—C10—C11	120.05 (18)
F2—C3—C2	119.20(17)	C9—C10—H10	120.0
F2—C3—C4	119.23(16)	11—C10—H10	120.0
C2—C3—C4	121.57(18)	C10—C11—C12	119.85(17)
C5—C4—C3	116.57(17)	C10—C11—C14	121.43(18)
C5—C4—N1	127.68(18)	C12—C11—C14	118.72(18)
C3—C4—N1	115.72(18)	C13—C12—C11	119.75(18)
F3—C5—C6	118.73(17)	C13—C12—H12	120.1
F3—C5—C4	119.74(16)	C11—C12—H12	120.1
C6—C5—C4	121.52(17)	C12—C13—C8	120.67(18)
F4—C6—C5	117.94(17)	C12—C13—H13	119.7
F4—C6—C1	119.90(16)	C8—C13—H13	119.7
C5—C6—C1	122.07(18)	C15—C14—C11	176.8(2)
N4—C7—C8	121.18(17)	C14—C15—H15	180.0
C4—N1—N2—N3	179.3(13)	C4—C5—C6—F4	177.77(17)
C7—N4—C1—C2	139.37(19)	F3—C5—C6—C1	177.51(17)
C7—N4—C1—C6	47.4(3)	C4—C5—C6—C1	1.3(3)
C6—C1—C2—F1	178.11(16)	C2—C1—C6—F4	178.07(16)
N4—C1—C2—F1	8.0(3)	N4—C1—C6—F4	4.7(3)
C6—C1—C2—C3	0.5(3)	C2—C1—C6—C5	1.6(3)
N4—C1—C2—C3	173.42(17)	N4—C1—C6—C5	171.74(18)
F1—C2—C3—F2	0.2(3)	C1—N4—C7—C8	179.05(17)
C1—C2—C3—F2	178.43(17)	N4—C7—C8—C13	0.4(3)
F1—C2—C3—C4	179.66(17)	N4—C7—C8—C9	179.39(18)
C1—C2—C3—C4	1.1(3)	C13—C8—C9—C10	1.1(3)
F2—C3—C4—C5	178.05(16)	C7—C8—C9—C10	177.97(18)
C2—C3—C4—C5	1.4(3)	C8—C9—C10—C11	0.2(3)
F2—C3—C4—N1	3.7(3)	C9—C10—C11—C12	0.6(3)
C2—C3—C4—N1	176.76(17)	C9—C10—C11—C14	179.17(18)
N2—N1—C4—C5	11.8(3)	C10—C11—C12—C13	0.6(3)
N2—N1—C4—C3	170.25(19)	C11—C12—C13—C8	−0.3(3)
N1—C4—C5—F3	1.1(3)	C9—C8—C13—C12	1.1(3)
C3—C4—C5—C6	0.3(3)	C7—C8—C13—C12	177.92(18)
N1—C4—C5—C6	177.64(18)	C10—C11—C14—C15	177(4)
F3—C5—C6—F4	1.0(3)	C12—C11—C14—C15	3(4)

Table S1-4. Anisotropic displacement parameters ($Å^2 \times 10^3$) for imine **1** at 113K. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2hka^*b^*U^{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
F(1)	0.0233(6)	0.0369(7)	0.0275 (6)	0.0006(5)	0.0078(5)	0.0017(5)
F(2)	0.0211(6)	0.0356(7)	0.0314 (6)	0.0031(5)	0.0016(5)	0.0031(5)
F(3)	0.0295(7)	0.0353(7)	0.0236 (6)	0.0041(5)	0.0053(5)	0.0044(5)
F(4)	0.0222(6)	0.0277(7)	0.0279 (6)	0.0030(5)	0.0000(5)	0.0002(5)
N(1)	0.0285(10)	0.0385(11)	0.0228 (8)	0.0026(8)	0.0010(7)	0.0020(7)
N(2)	0.0297(10)	0.0453(12)	0.0265 (10)	0.0056(9)	0.0036(8)	0.0018(8)
N(3)	0.0422(13)	0.0918(19)	0.0258 (10)	0.0234(12)	0.0012(9)	0.0022(11)
N(4)	0.0256(9)	0.0235(9)	0.0214 (8)	0.0015(7)	0.0001(7)	0.0013(6)
C(1)	0.0248(11)	0.0194(10)	0.0223 (10)	0.0049(8)	0.0027(8)	0.0014(8)
C(2)	0.0216(10)	0.0213(11)	0.0251 (10)	0.0041(8)	0.0057(8)	0.0014(8)
C(3)	0.0177(10)	0.0214(11)	0.0305 (11)	0.0030(8)	0.0012(8)	0.0029(8)
C(4)	0.0280(11)	0.0208(11)	0.0222 (9)	0.0038(8)	0.0011(8)	0.0009(8)
C(5)	0.0234(11)	0.0237(11)	0.0238 (10)	0.0025(8)	0.0039(8)	0.0021(8)
C(6)	0.0186(10)	0.0196(10)	0.0277 (10)	0.0021(8)	0.0021(8)	0.0011(8)
C(7)	0.0245(11)	0.0206(10)	0.0248 (10)	0.0007(8)	0.0052(8)	0.0007(8)
C(8)	0.0235(11)	0.0207(10)	0.0240 (10)	0.0033(8)	0.0011(8)	0.0007(8)
C(9)	0.0225(11)	0.0244(11)	0.0300 (10)	0.0006(8)	0.0034(8)	0.0002(8)
C(10)	0.0213(11)	0.0254(11)	0.0322 (11)	0.0031(9)	0.0048(9)	0.0016(9)
C(11)	0.0268(11)	0.0202(11)	0.0254 (10)	0.0023(8)	0.0013(8)	0.0006(8)
C(12)	0.0266(11)	0.0223(11)	0.0269 (10)	0.0011(9)	0.0031(8)	0.0013(8)
C(13)	0.0216(10)	0.0219(11)	0.0270 (10)	0.0000(8)	0.0016(8)	0.0025(8)
C(14)	0.0293(12)	0.0271(12)	0.0329 (12)	0.0020(9)	0.0036(9)	0.0007(9)
C(15)	0.0407(14)	0.0501(15)	0.0296 (12)	0.0100(11)	0.0074(10)	0.0033 (10)

References:

[1] W. B. Austin, N. Bilow, W. J. Kelleghan, K. S. Y. Lau, *J. Org. Chem.* **1981**, *46*, 2280-2286.
[2] C. Törnkvist, J. Bergman, B. Liedberg, *J. Phys. Chem.* **1991**, *95*, 3119.
[3] A. A. Jbarah, K. Banert, R. Holze, *Vib. Spectrosc.* **2007**, *44*, 142.