Supplementary material

Facile synthesis of *N,N'*-alkylidene bisamides catalyzed by trimethyl-tris(4-pyridinium)benzene trifluoroacetate: an efficient and rapid approach

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Materials and Apparatuses

All solvents and reactants were obtained from Sigma-Aldrich or Fluka Chemical Companies. A Thermo Scientific 9200 device was used to measure melting points. The progress of the reactions was supervised by TLC using SIL G/UV 254 silica gel plates. A Thermo device (AVATAR model) was used to record the FT-IR spectrum. A Heraeus CHN-OS Elemental Analyzer was used to determine the contents of carbon, hydrogen and nitrogen elements. The ^{19}F NMR (235 MHz), ^{13}C NMR (125.7 MHz) and ^{1}H NMR (500 MHz) spectra were recorded on a Bruker Avance DPX FT-NMR spectrometer. TGA was performed using a TA instrument (model Q600), at temperatures of 25–600°C, with a temperature increase rate of 10°C/min in an argon atmosphere. MS QP 1100 EX mass spectrometer operating at an ionization potential of 70 eV. The UV spectra were recorded in the range of 250–600 nm on an Avantes-Avaspec-2048 spectrophotometer at 25 °C.

Selected spectral data of the *N,N'-alkylidene bisamides*

Product 3a. ^{1}H NMR (500 MHz, DMSO-d_6): δ (ppm) 9.10 (d, *J* = 7.7 Hz, 2H, 2NH), 7.91 (d, *J* = 7.5 Hz, 4H, H_{Ar}), 7.50 (t, *J* = 7.4 Hz, 2H, H_{Ar}), 7.42-7.46 (m, 6H, H_{Ar}), 7.36 (t, *J* = 7.6 Hz, 2H, H_{Ar}), 7.25 (t, *J* = 7.4 Hz, 1H, H_{Ar}), 7.05 (t, *J* = 7.7 Hz, 1H, CH) (Fig. S1); ^{13}C NMR (125 MHz, DMSO-d_6): δ (ppm) 59.56, 126.33, 127.02, 127.86, 128.48, 129.06, 132.34, 134.60, 140.95, 166.93 (Fig. S2)

Product 8a. ^{1}H NMR (500 MHz, DMSO-d_6): δ (ppm) 9.18 (d, *J* = 7.5 Hz, 2H, 2NH), 7.91 (d, *J* = 7.6 Hz, 4H, H_{Ar}), 7.57-7.63 (m, 6H, H_{Ar}), 7.37-7.40 (m, 4H, H_{Ar}), 7.11-7.15 (m, 4H, H_{Ar}), 7.04 (t, *J* = 7.5 Hz, 1H, CH), 2.29 (s, 3H, CH_3) (Fig. S3); ^{13}C NMR (125 MHz, DMSO-d_6): δ (ppm) 166.84, 144.06, 139.39, 135.80, 134.93, 133.98, 133.06, 130.94, 129.94, 129.27, 128.69, 127.95, 127.13, 58.64, 20.79 (Fig. S4); FT-IR (KBr): 710, 1540, and 3275 (NH), 1490 (C=C), 1642 (C=O), 2895 (SP^3-CH), 3066 (SP^2-CH) cm^{-1}; Mass: *m/z* 420 [M^+], 421 [M^+ + 1] (Fig. S5); Anal. Calcd for $C_{28}H_{24}N_2O_2$: C, 79.98; H, 5.75; N, 6.66, %; Found C, 79.86; H, 5.68; N, 6.58 %.

Product 9a. ^{1}H NMR (500 MHz, DMSO-d_6): δ (ppm) 8.74 (d, *J* = 7.7 Hz, 2H, 2NH), 7.33-7.37 (m, 4H, H_{Ar}), 7.13-7.16 (m, 4H, H_{Ar}), 6.56 (t, *J* = 7.7 Hz, 1H, CH), 2.29 (s, 3H, CH_3), 1.84 (s, 6H, $2CH_3$) (Fig. S6); ^{13}C NMR (125 MHz, DMSO-d_6): δ (ppm) 168.91, 143.24, 138.64, 135.46, 134.58, 130.47, 128.92, 128.11, 127.37, 57.10, 23.80, 20.21 (Fig. S7); FT-IR (KBr): 712, 1556, and 3275 (NH), 1513 (C=C), 1664 (C=O, Stretching), 2963 (SP^3-CH), 3044 (SP^2-CH) cm^{-1}; Mass: *m/z* 296 [M^+], 297 [M^+ + 1], 298 [M^+ + 2] (Fig. S8); Anal. Calcd for $C_{18}H_{20}N_2O_2$: C, 72.95; H, 6.80; N, 9.45, %; Found C, 73.04; H, 6.72; N, 9.39%.

Product 10a. ^{1}H NMR (500 MHz, DMSO-d_6): δ (ppm) 8.75 (d, *J* = 7.7 Hz, 2H, 2NH), 8.23 (d, *J* = 8.7 Hz, 2H, H_{Ar}), 7.57 (d, *J* = 8.6 Hz, H_{Ar}), 6.55 (t, *J* = 7.7 Hz, 1H, CH), 1.88 (s, 6H,

$2CH_3$) (Fig. S9); ^{13}C NMR (125 MHz, DMSO-d_6): δ (ppm) 169.65, 148.61, 147.73, 128.21, 123.85, 57.72, 23.18 (Fig. S10)

Original spectrums of the *N,N'-alkylidene bisamides*

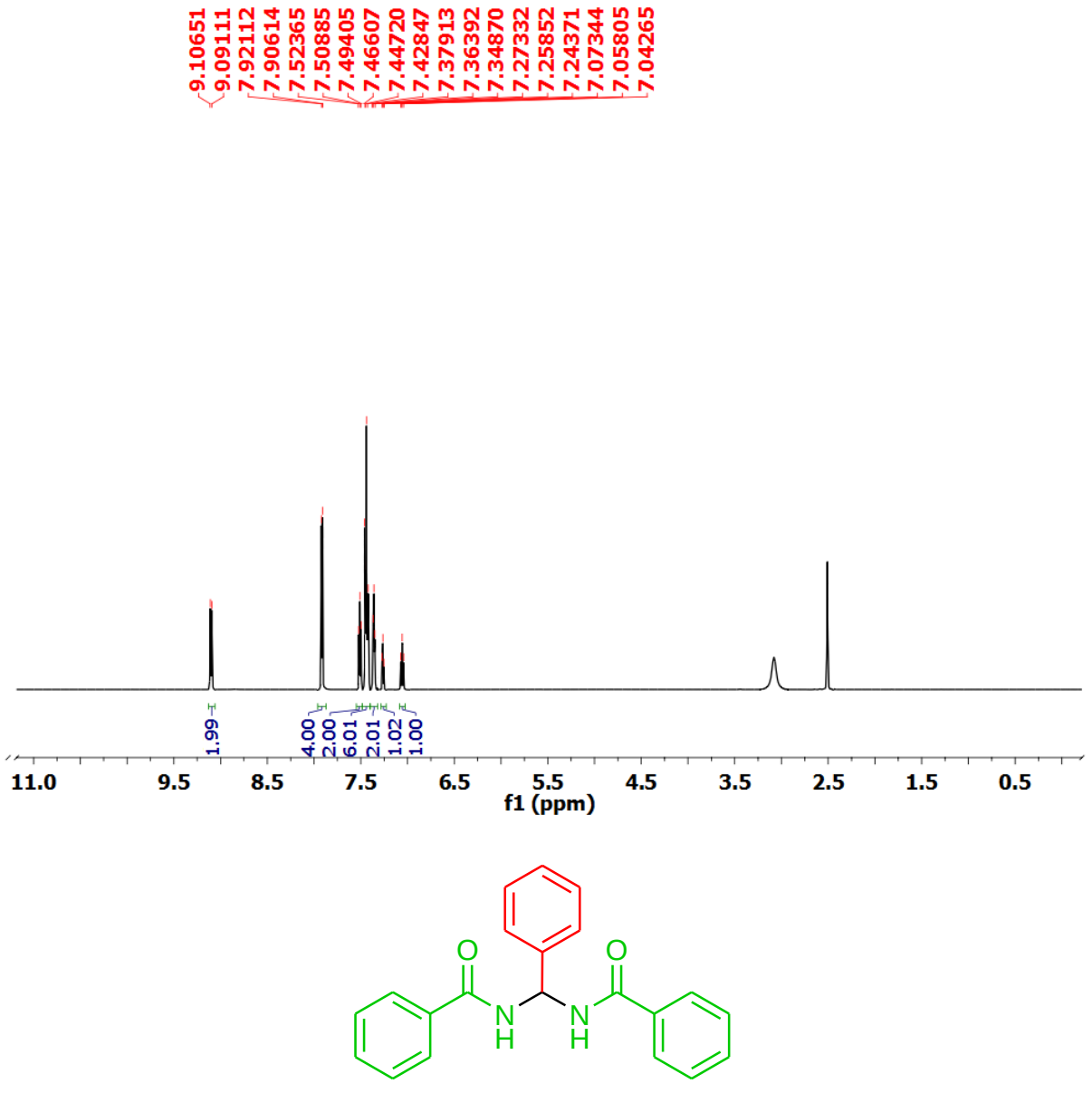


Fig. S1 The ^1H NMR spectrum of product **3a**

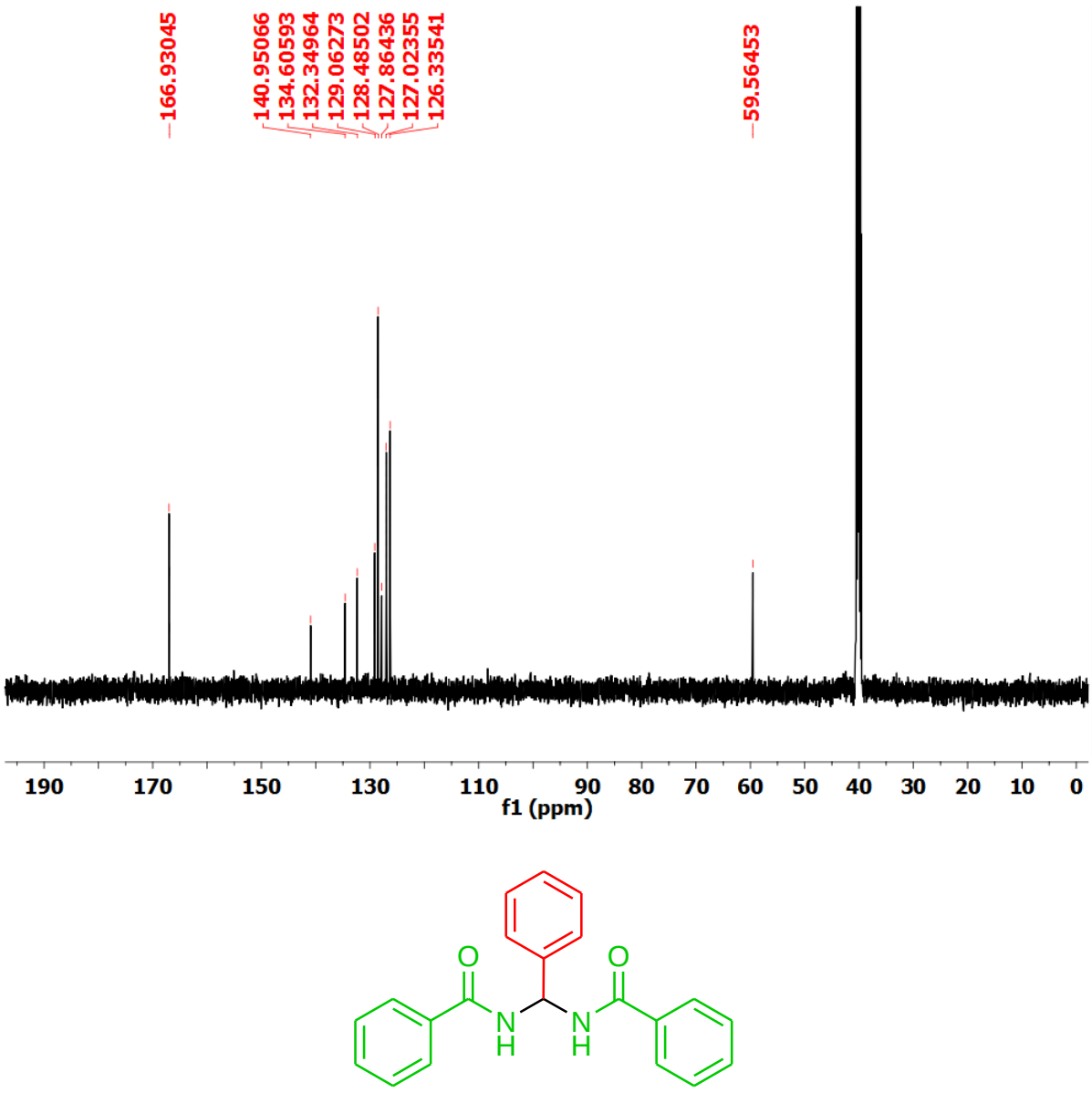


Fig. S2 The ^{13}C NMR spectrum of product **3a**

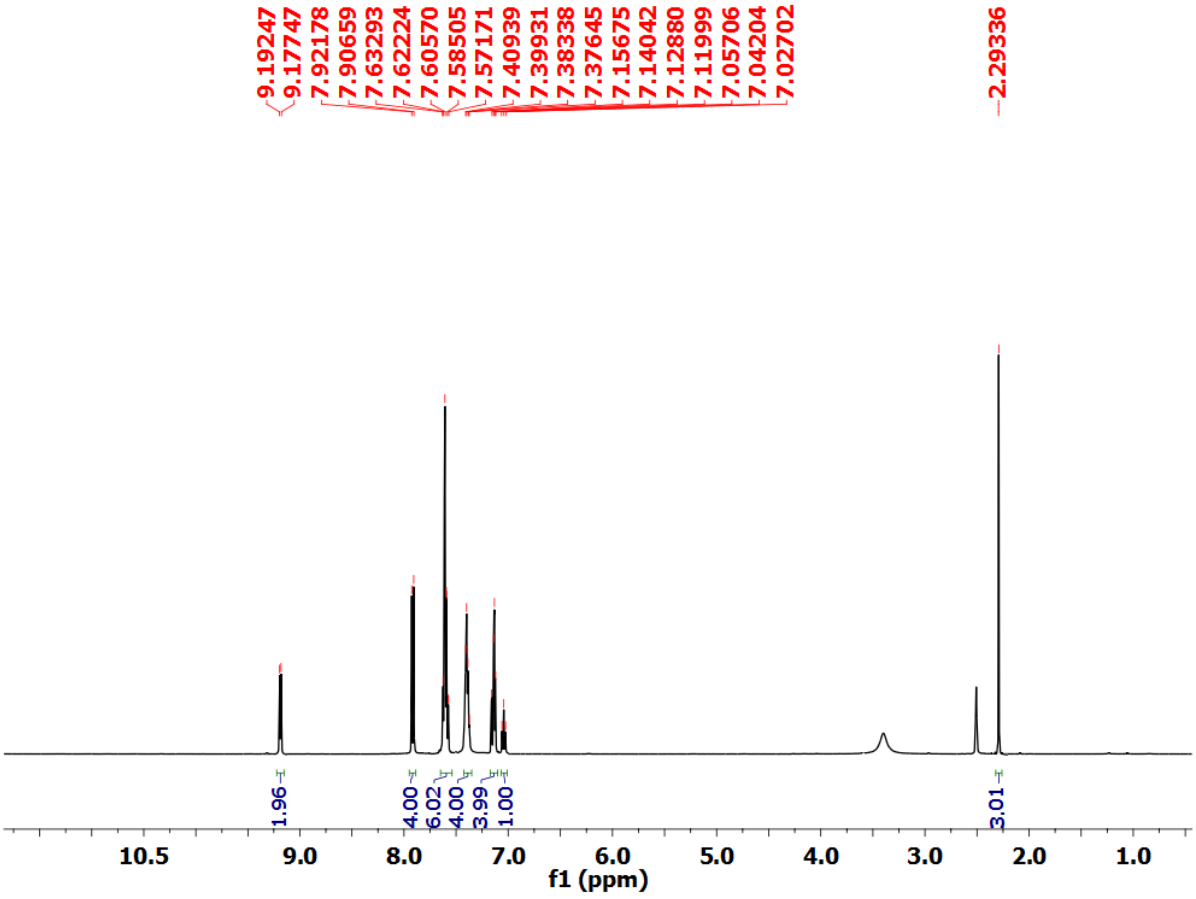
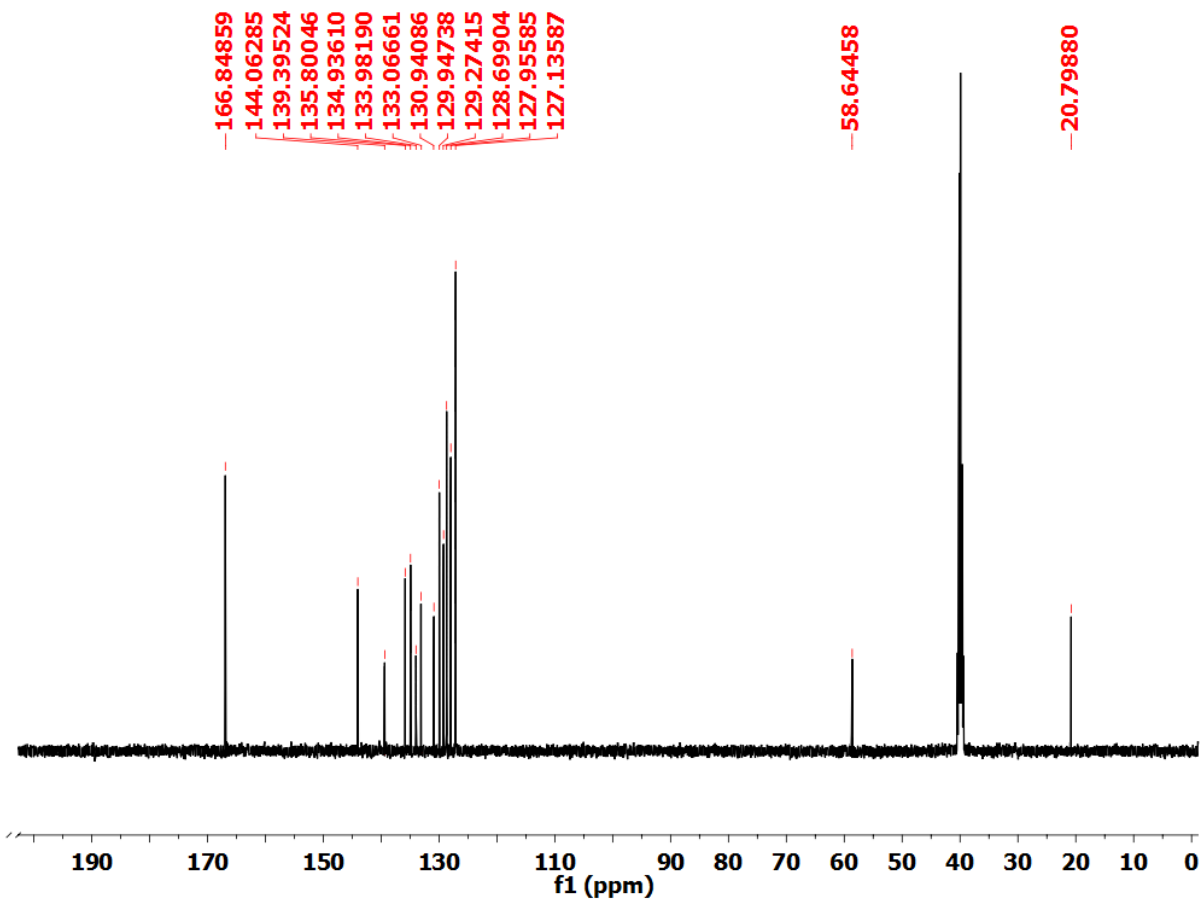


Fig. S3 The ^{1}H NMR spectrum of product **8a**



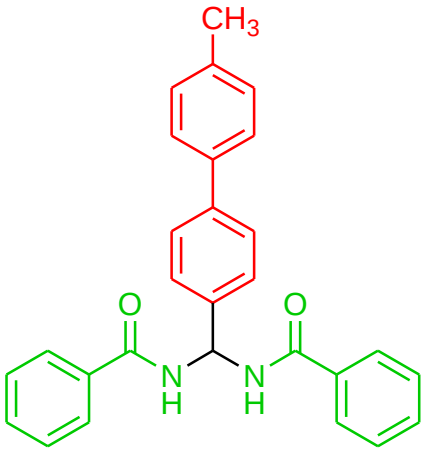
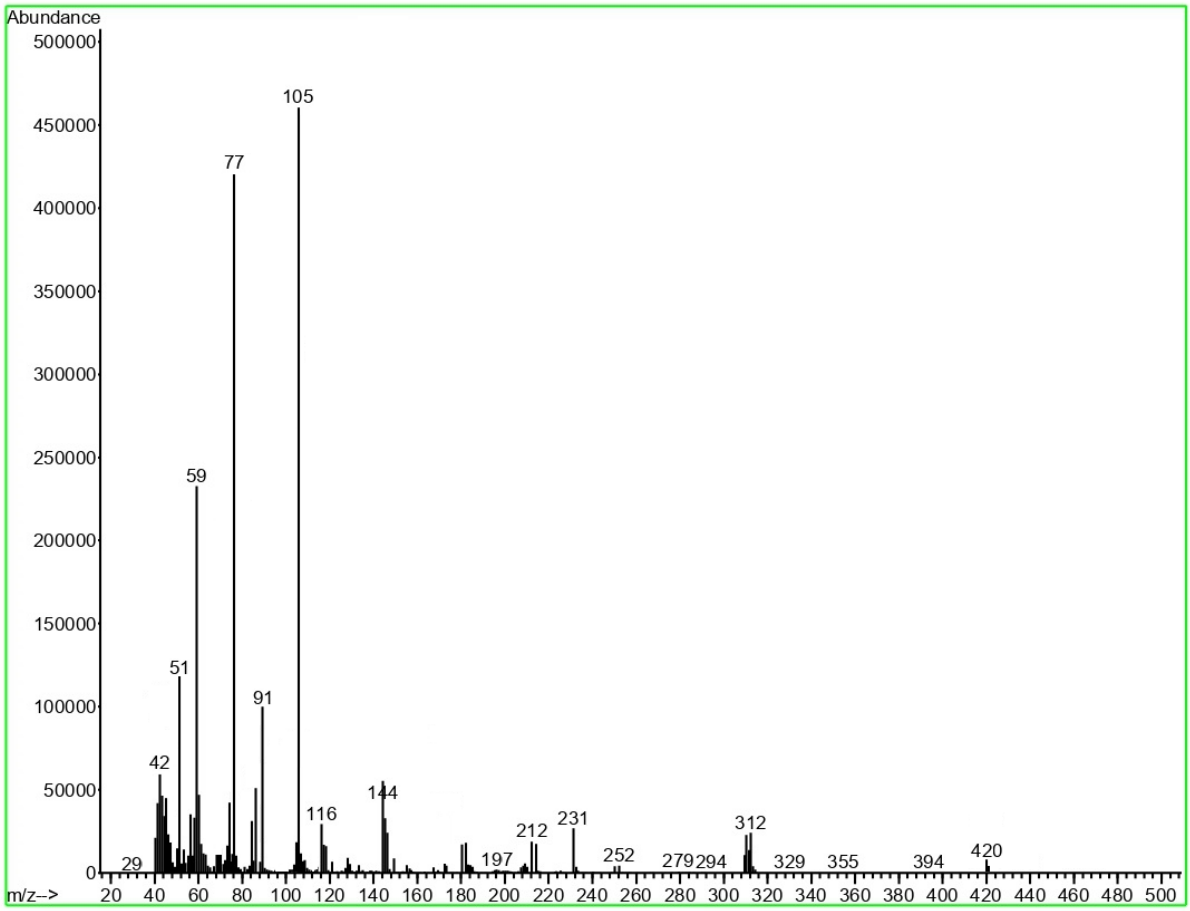


Fig. S4 The ^{13}C NMR spectrum of product **8a**



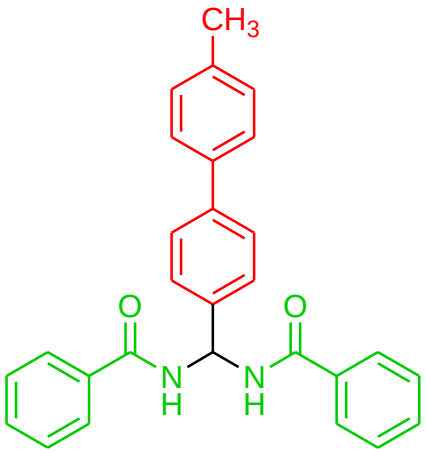


Fig. S5 The mass spectrum of product **8a**

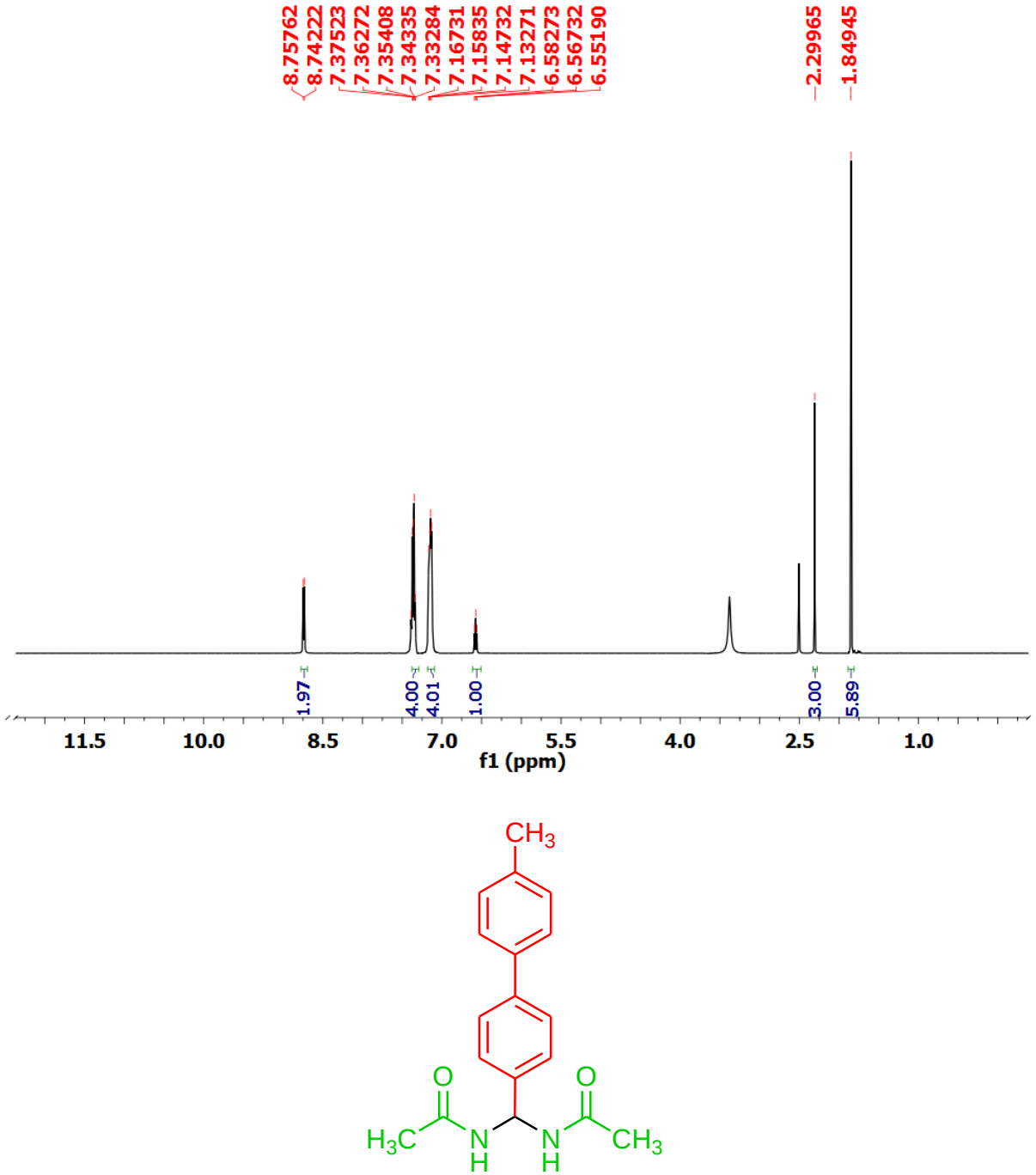


Fig. S6 The ^{1}H NMR spectrum of product **9a**

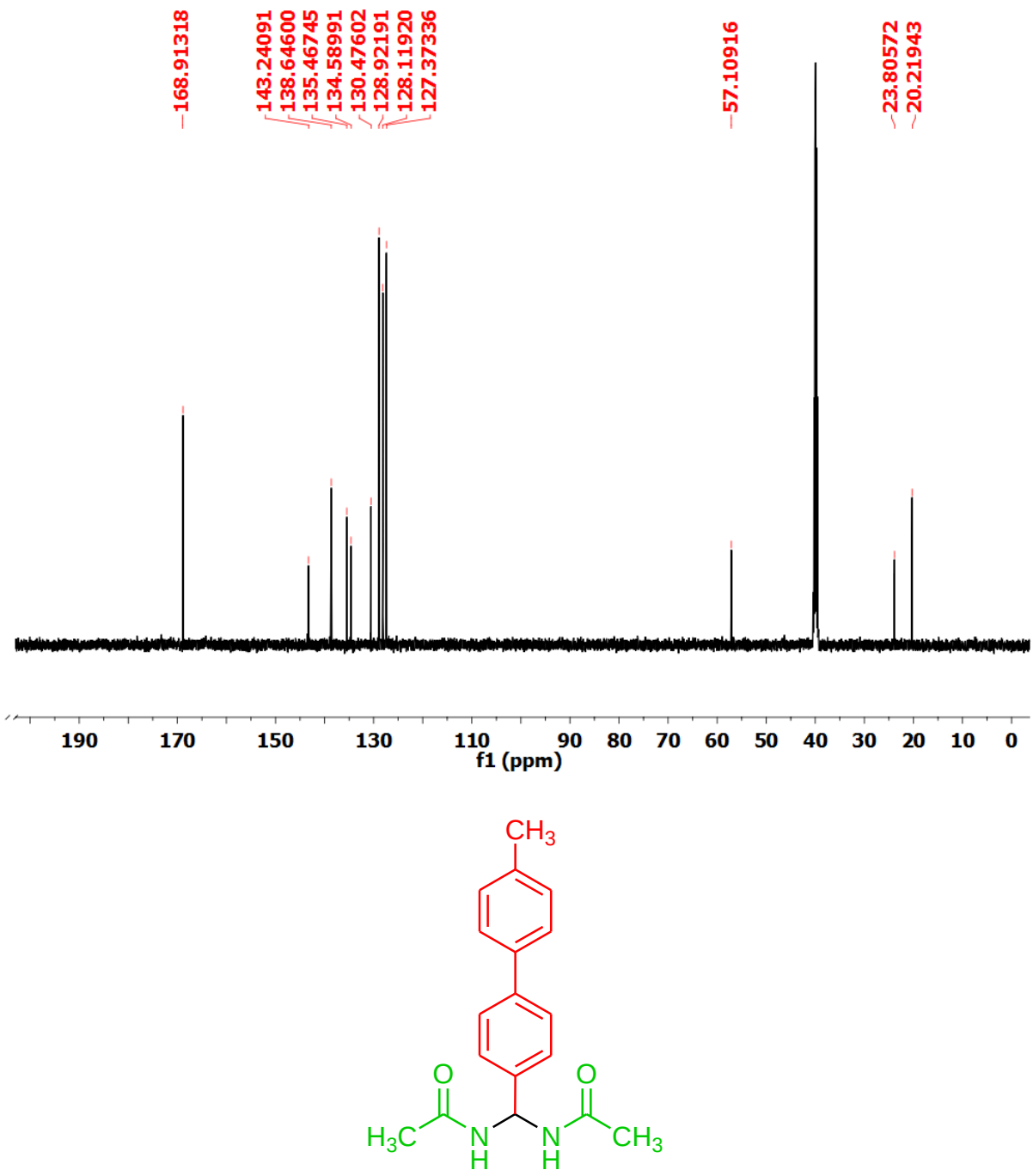


Fig. S7 The ^{13}C NMR spectrum of product **9a**

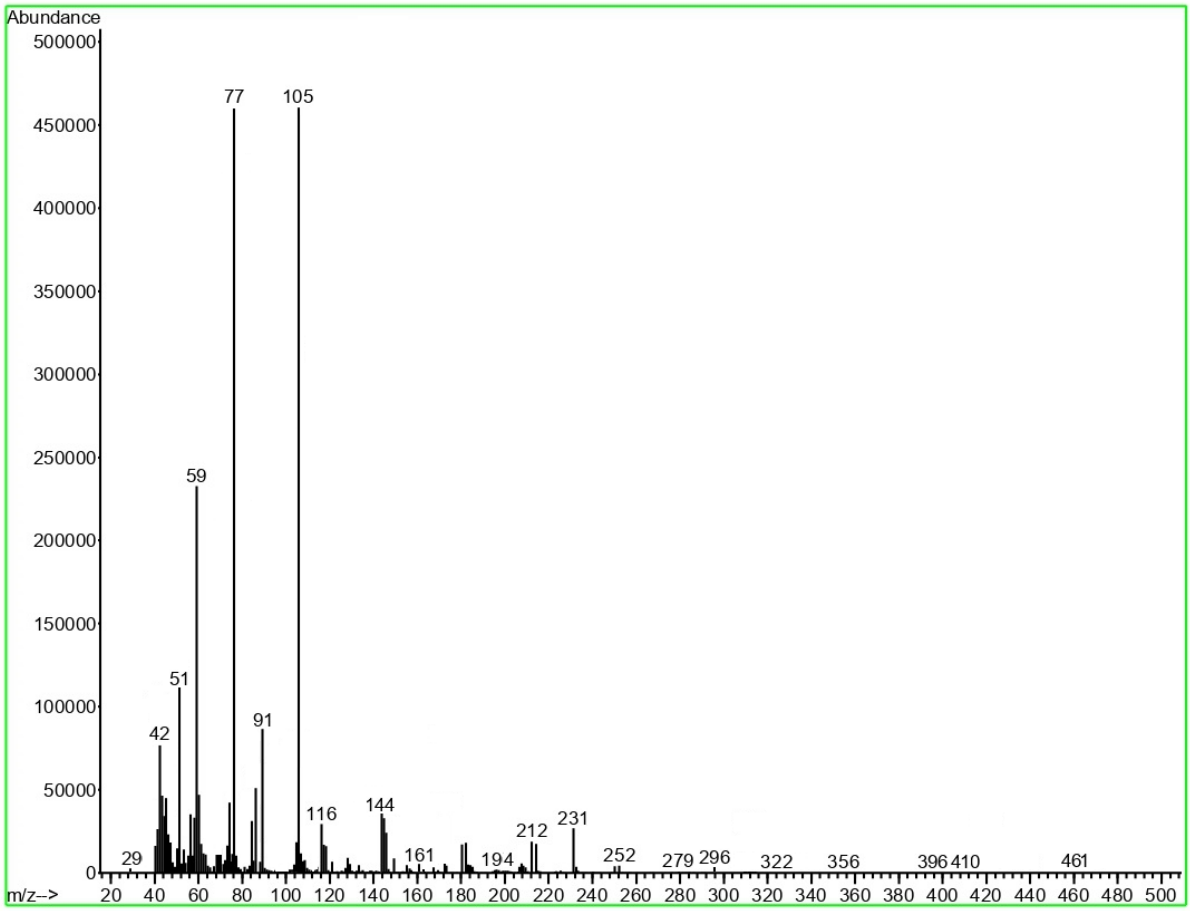


Fig. S8 The mass spectrum of product **9a**

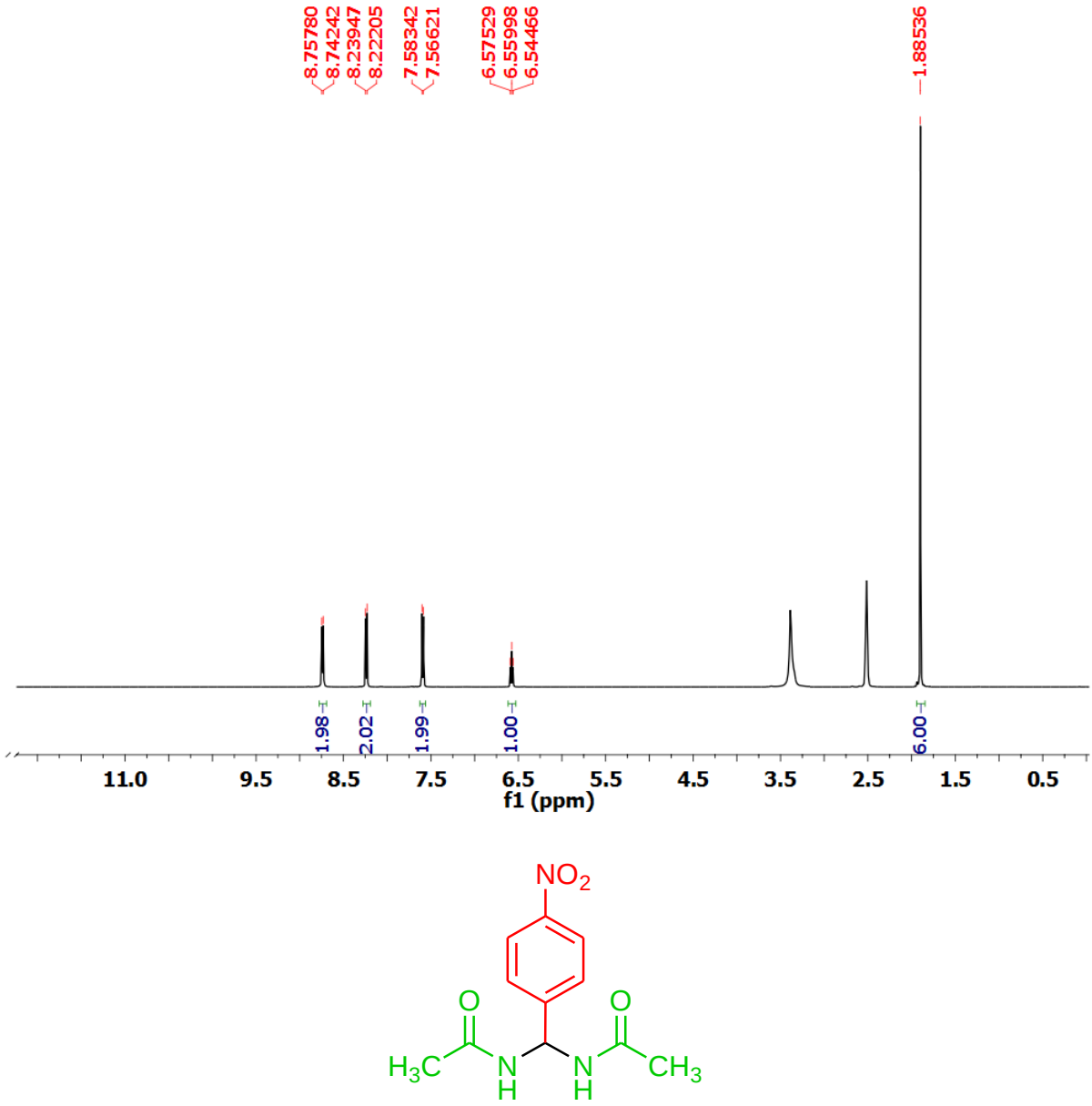
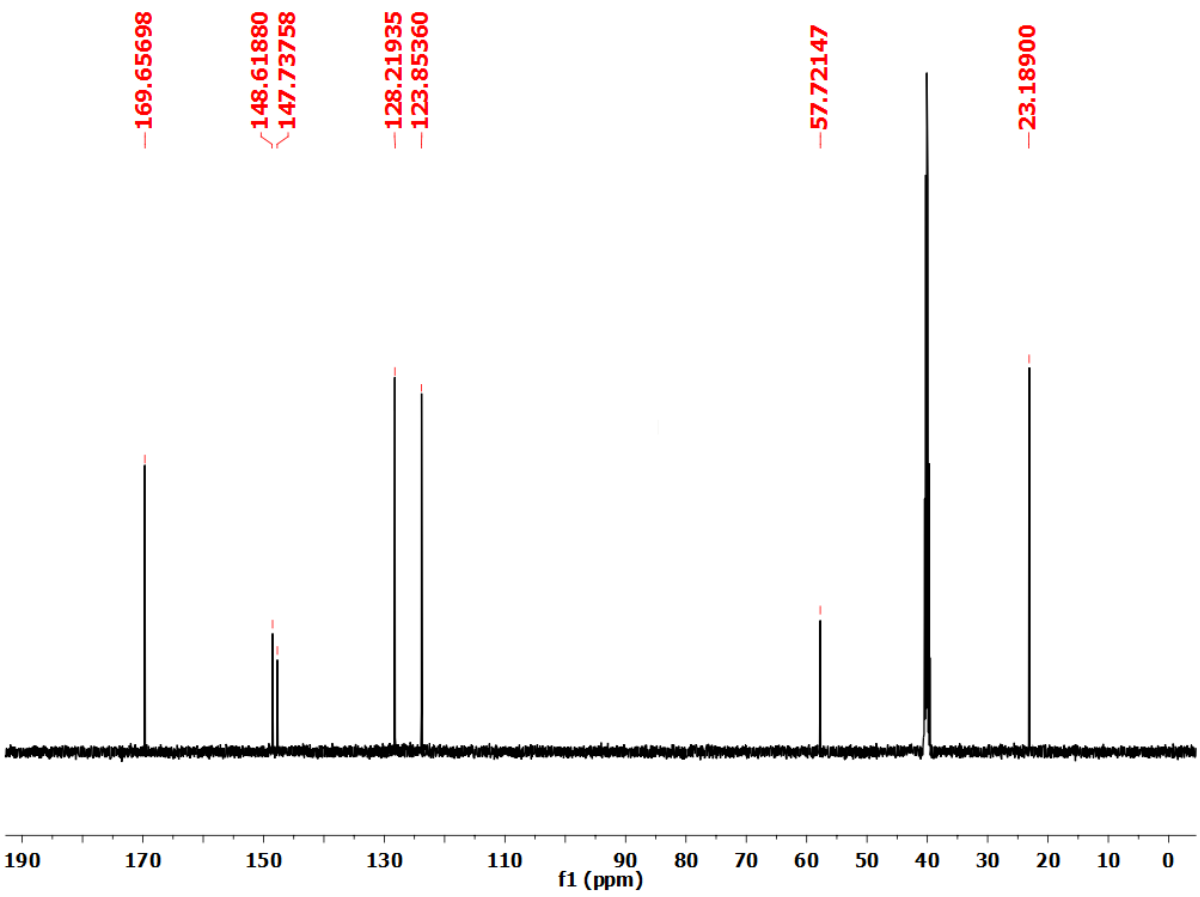


Fig. S9 The ^{1}H NMR spectrum of product **10a**



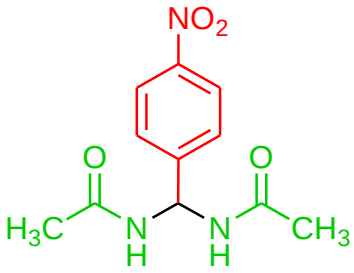


Fig. S10 The ^{13}C NMR spectrum of product **10a**