Electronic Supplementary Material (ESI) for RSC Advances.

Supporting information

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Contents

1. Synthesis

1,5-Diazabicyclo[4.3.0]non-5-enium acetate [DBNH][OAc]

65.38 g (0.526 mol) of freshly distilled DBN was weighed into the reactor. The airspace was then replaced with argon gas followed by slow addition of 31.61 g (0.526 mol) of acetic acid. The temperature of the mildly exothermic reaction was kept below 70°C during the addition and after the last of the acetic acid the reactor was sealed and let to mix for 30 minutes in 100°C. The composition of the formed IL was characterized by NMR in d_6-DMSO and d_4-MeOH.

^{1}H NMR (in ppm, d_4-MeOH, 600 MHz, 27 °C): δ 3.70 (t, J = 7.3 Hz, 2H), 3.46 (s, J = 5.9 Hz, 2H), 3.41 (t, J = 5.8 Hz, 2H), 2.89 (t, J = 8.0 Hz, 2H), 2.17 (dt, J = 8.0, 7.3, 2H), 2.05 (dt, J = 5.9, 5.8 Hz, 2H), 1.89 (s, 3H).

^{13}C NMR (in ppm, d_4-MeOH, 151 MHz, 27 °C): δ 180.04, 165.88, 54.63, 43.52, 39.29, 31.13, 30.11, 24.39, 19.75

^{1}H NMR (in ppm, d_6-DMSO, 600 MHz, 27 °C): δ 3.49 (t, J = 7.1 Hz, 2H), 3.30 (t, J = 5.9 Hz, 2H), 3.24 (t, J = 5.8 Hz, 2H), 2.70 (t, J = 8.0 Hz, 2H), 1.97 (dt, J = 8.0, 7.1 Hz, 2H), 1.83 (dt, J = 5.9, 5.8 Hz, 2H), 1.64 (s, 1H).

^{13}C NMR (in ppm, d_6-DMSO, 151 MHz, 27 °C): δ 173.83, 162.87, 52.16, 41.94, 38.12, 29.43, 24.75, 18.72, 18.57

1-(3-Ammoniopropyl)-2-pyrrolidone acetate [APPH][OAc]

DBN was hydrolysed by refluxing in equal volume of distilled water for 18 hours. Water was removed from the mixture by rotary evaporation and the residue was distilled under oil pump vacuum to give 1-(3-aminopropyl)-2-pyrrolidone (APP) in excellent yield. Exactly one equivalent of acetic acid was added with cooling and stirring to the APP to give an oil which slowly solidified to give the title compound as a white crystalline mass. The product was characterized by NMR in d_6-DMSO and d_4-MeOH.

^{1}H NMR (in ppm, d_4-MeOH, 600 MHz, 27 °C): δ 3.49 (t, J = 7.1 Hz, 2H), 3.39 (t, J = 6.7 Hz, 2H), 2.90 (t, J = 7.3 Hz, 2H), 2.40 (t, J = 8.1 Hz, 2H), 2.08 (dt, J = 8.1, 7.1 Hz, 2H), 1.90 (dt, J = 7.3, 6.7 Hz, 2H), 1.89 (s, 3H)

^{13}C NMR (in ppm, d_4-MeOH, 151 MHz, 27 °C): δ 180.1, 178.40, 48.70, 40.50, 38.10, 21.80, 26.5, 24.20, 18.90

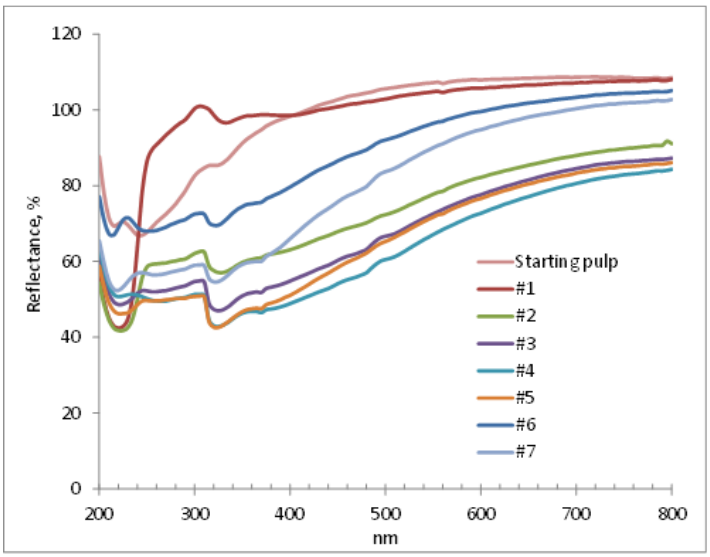
^{1}H NMR (in ppm, d_6-DMSO, 600 MHz, 27 °C): δ 5,41 (br, 3H), 3.31 (t, J = 7.0 Hz, 2H), 3.20 (t, J = 7.0 Hz, 2H), 2.53 (t, J = 7.0 Hz, 2H), 2.21 (t, J = 8.1, 2H), 1.91 (dt, J = 8.1, 7.0, 2H), 1.79 (s, 3H), 1.57 (p, J = 7.0 Hz, 2H).

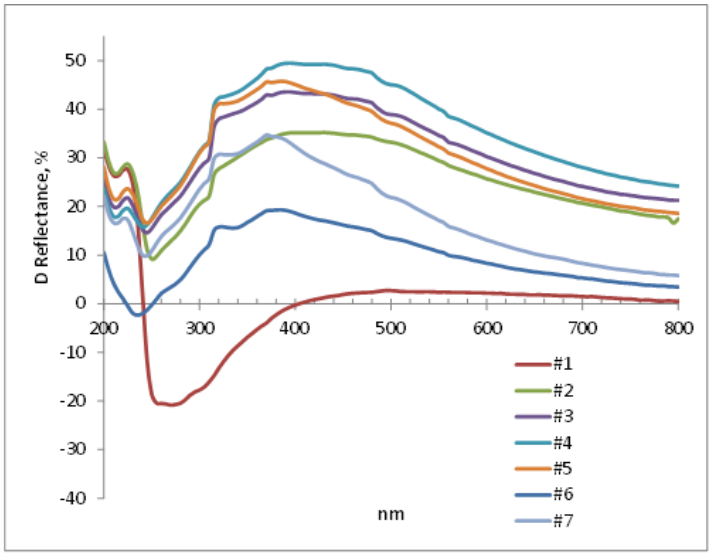
^{13}C NMR (in ppm, d_6-DMSO, 151 MHz, 27 °C): δ 174.08, 46.29, 39.12, 37.08, 30.39, 26.95, 23.80, 17.50

2. Spectra

a. S1. UV-VIS reflectance spectra

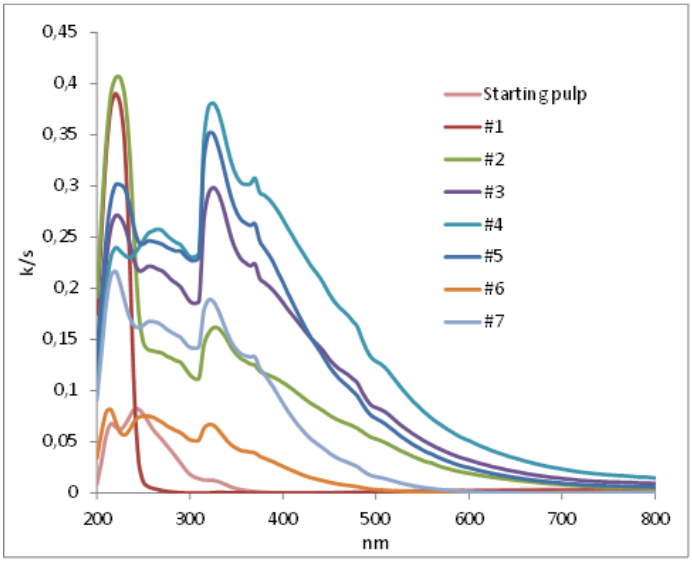
UV-Vis-reflectance (left) and difference reflectance (right) spectra of the untreated and in [DBNH][OAc] treated pulp samples. In difference reflectance spectra, the formed chromophores compared to the untreated pulp can be seen as positive signals and reacted/removed structures as negative signals.

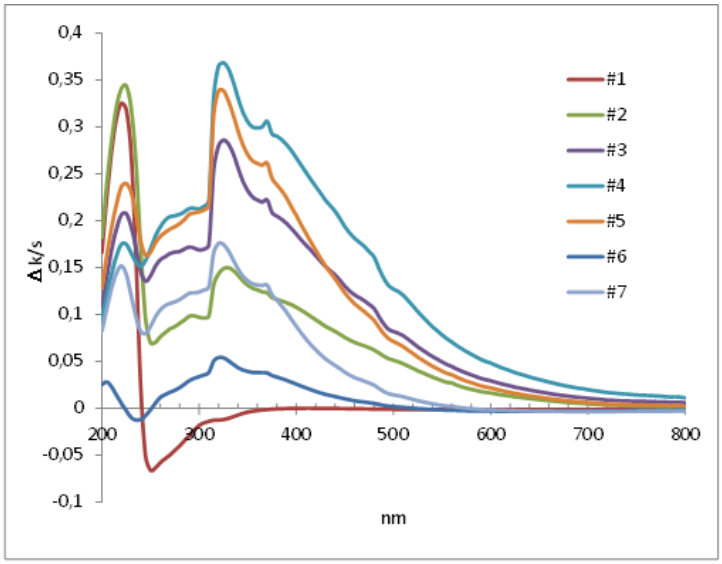




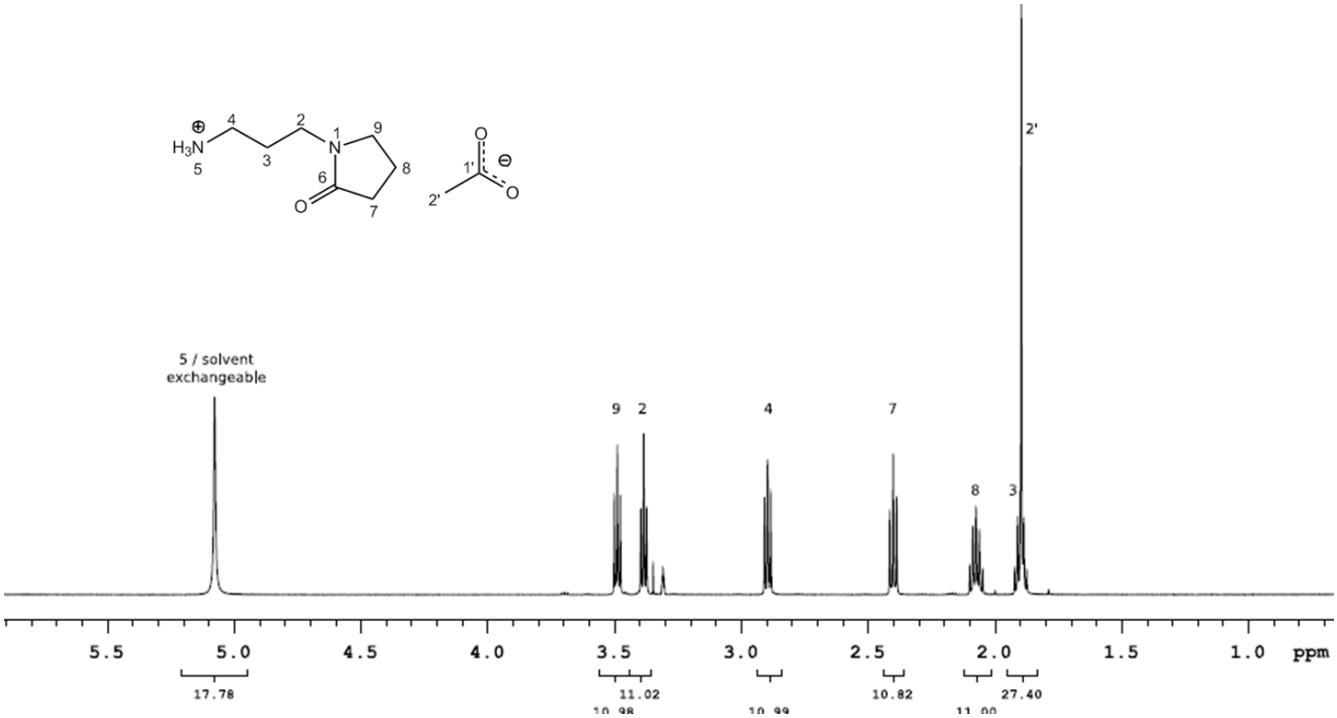
b. S2. K/s spectra

K/s absorbance (left) and difference absorbance spectra (right) of the untreated and in [DBNH]AcO treated samples.

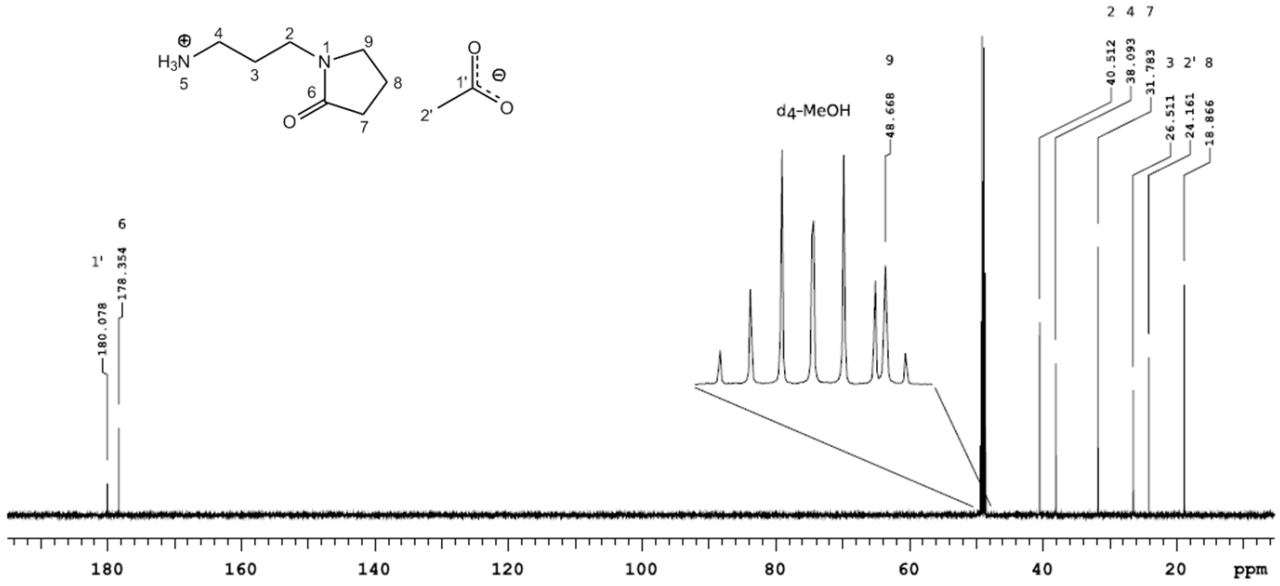




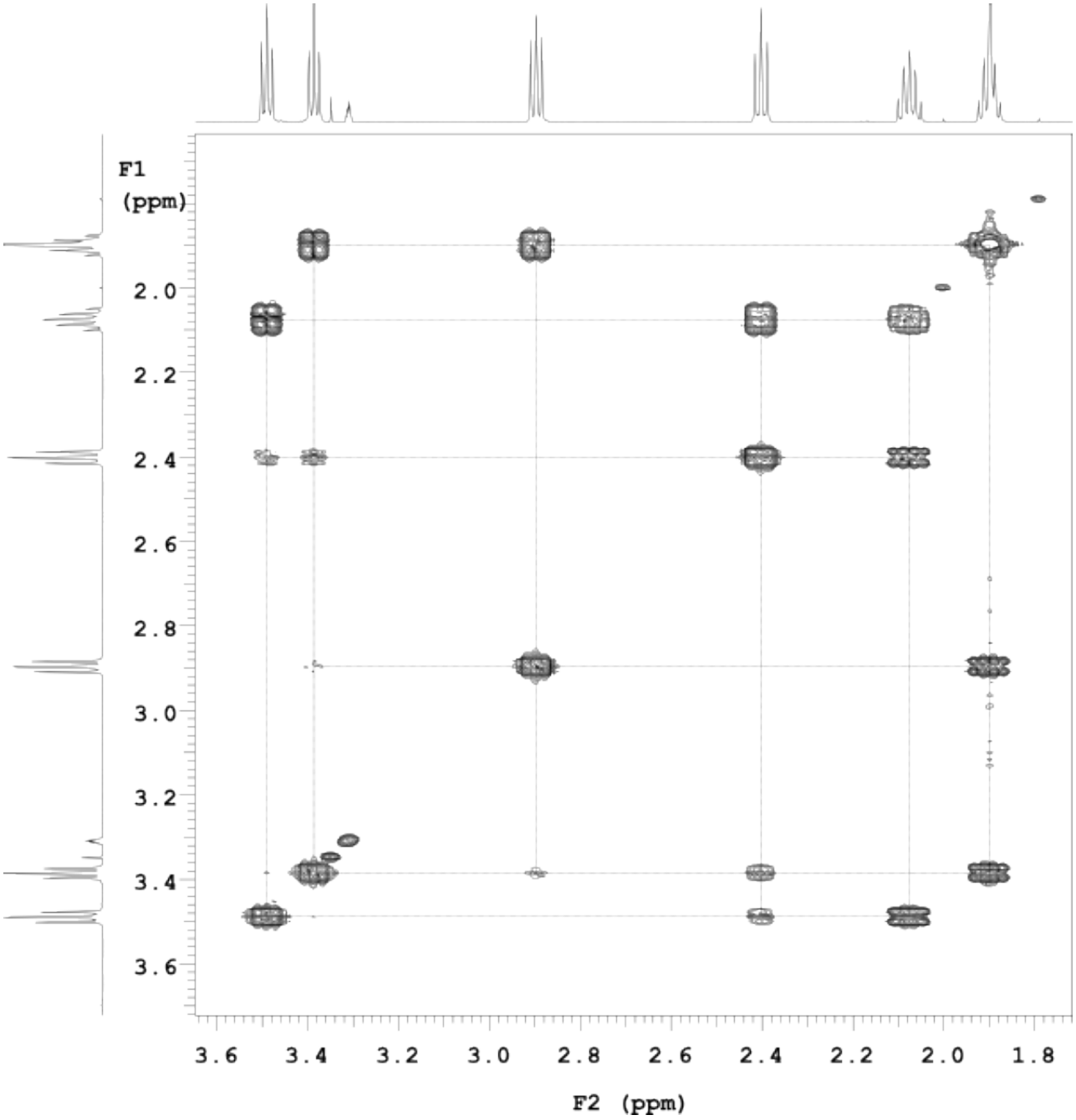
c. S3. [APPH][OAc] ^{1}H NMR in d_4-MeOH



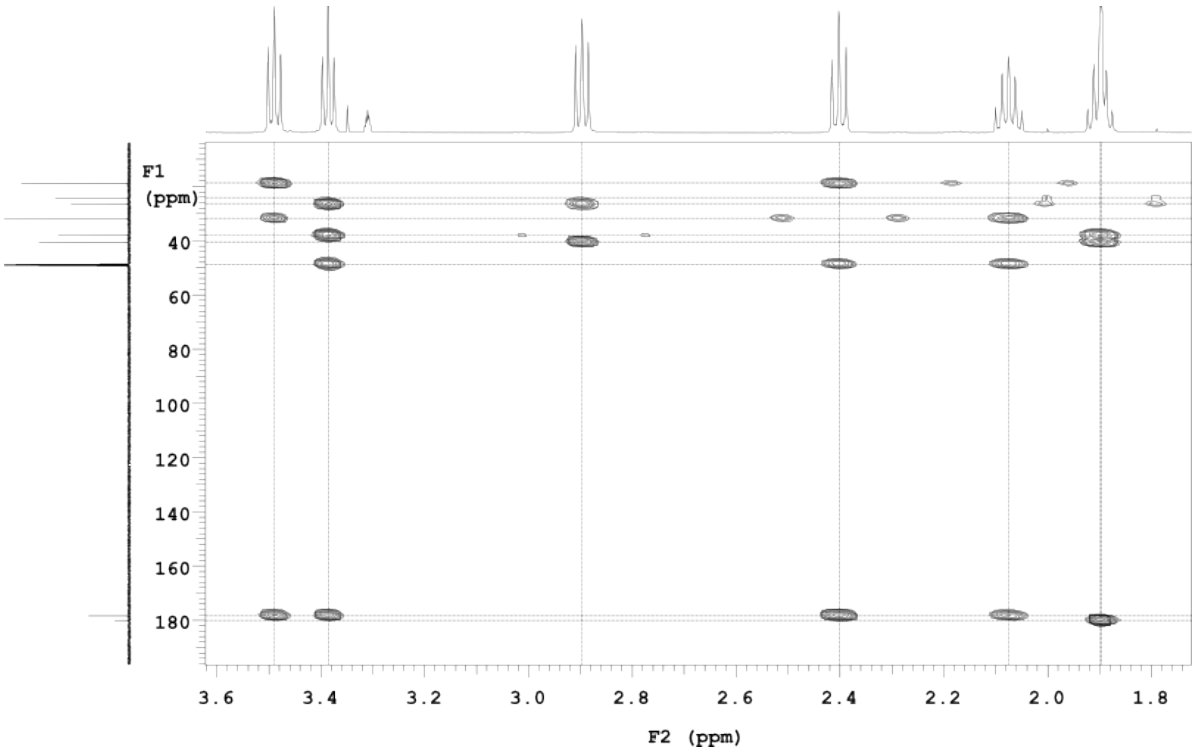
d. S4. [APPH][OAc] ^{13}C NMR in d_4-MeOH



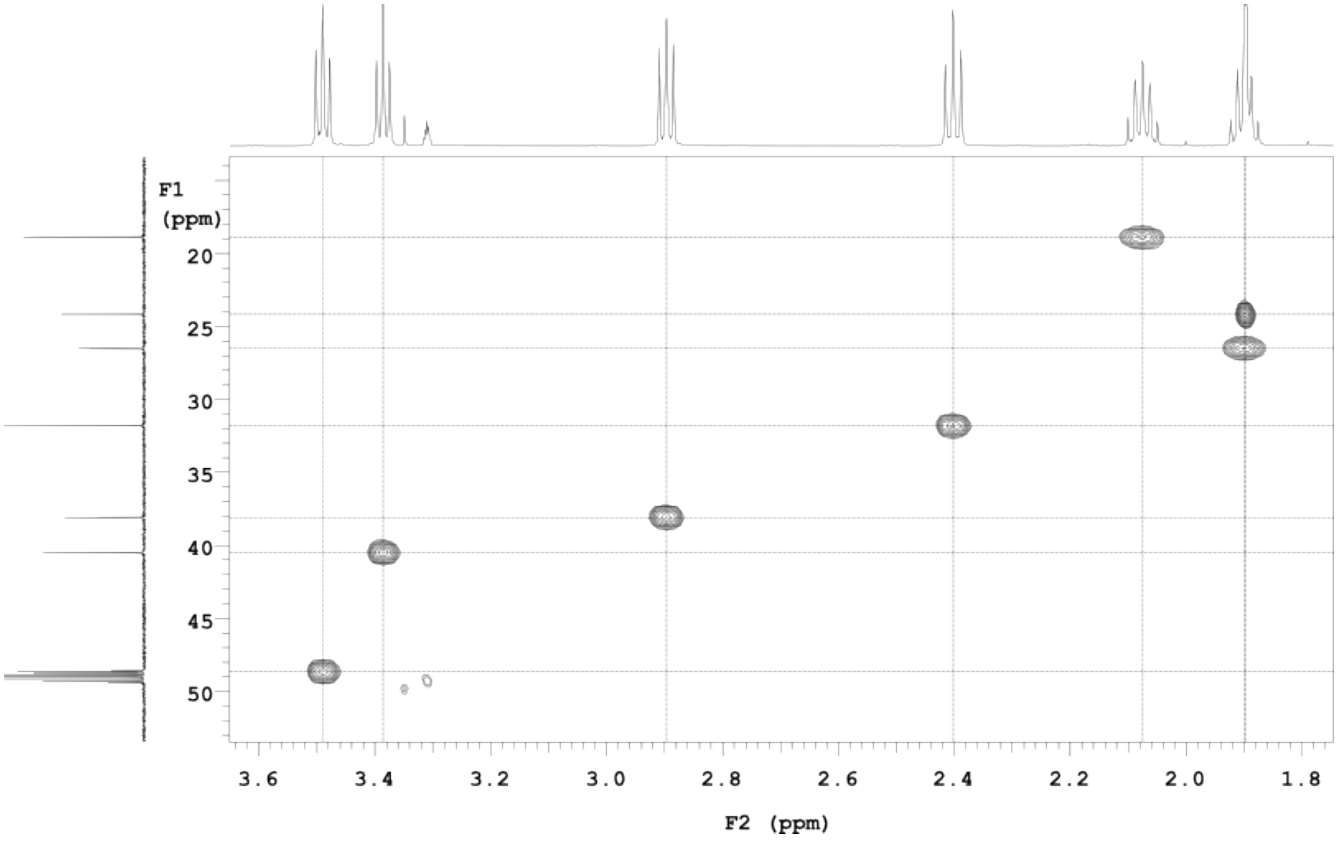
e. S5. [APPH][OAc] COSY in d_4-MeOH



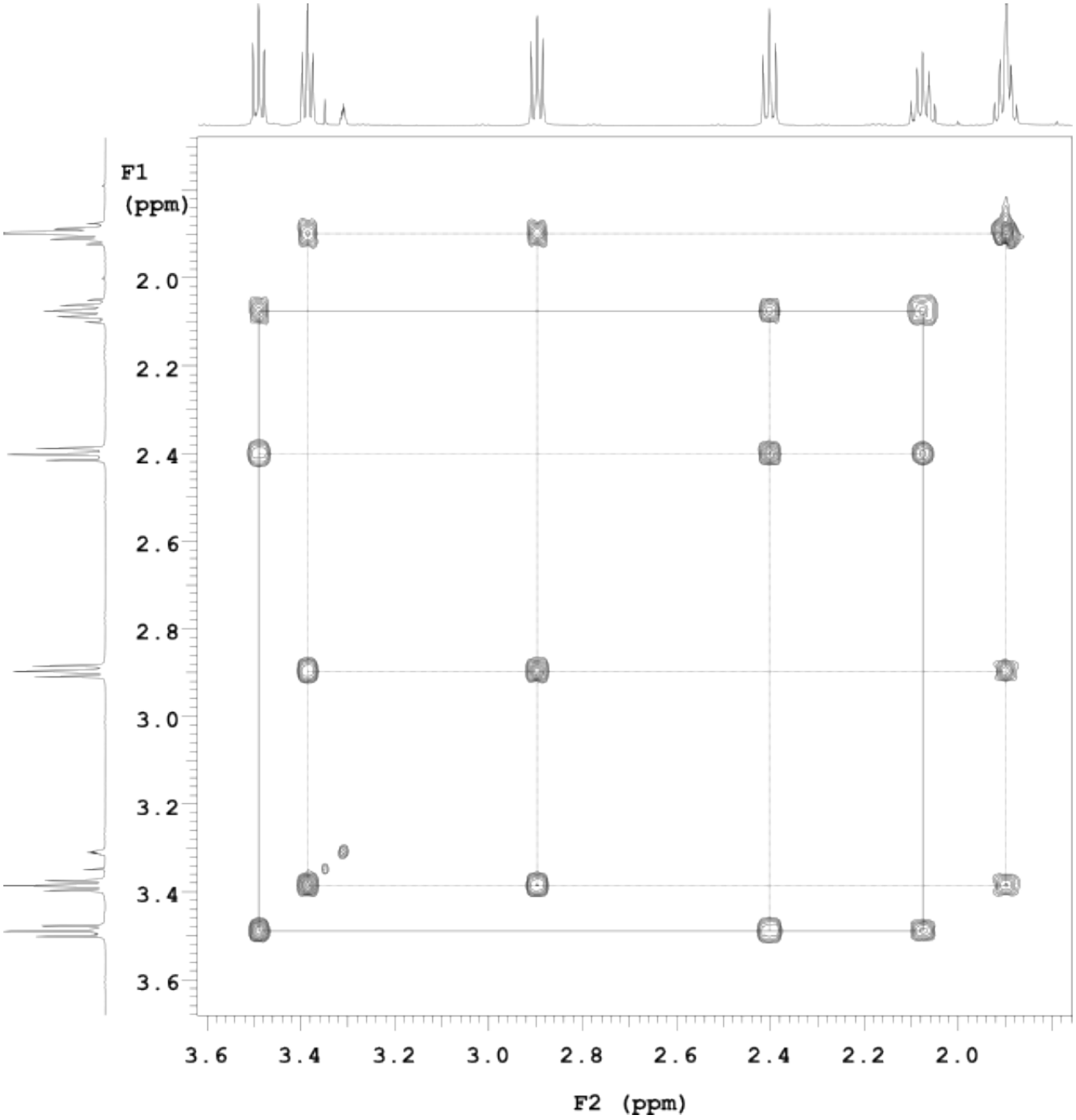
f. S6. [APPH][OAc] HMBC in d_4-MeOH



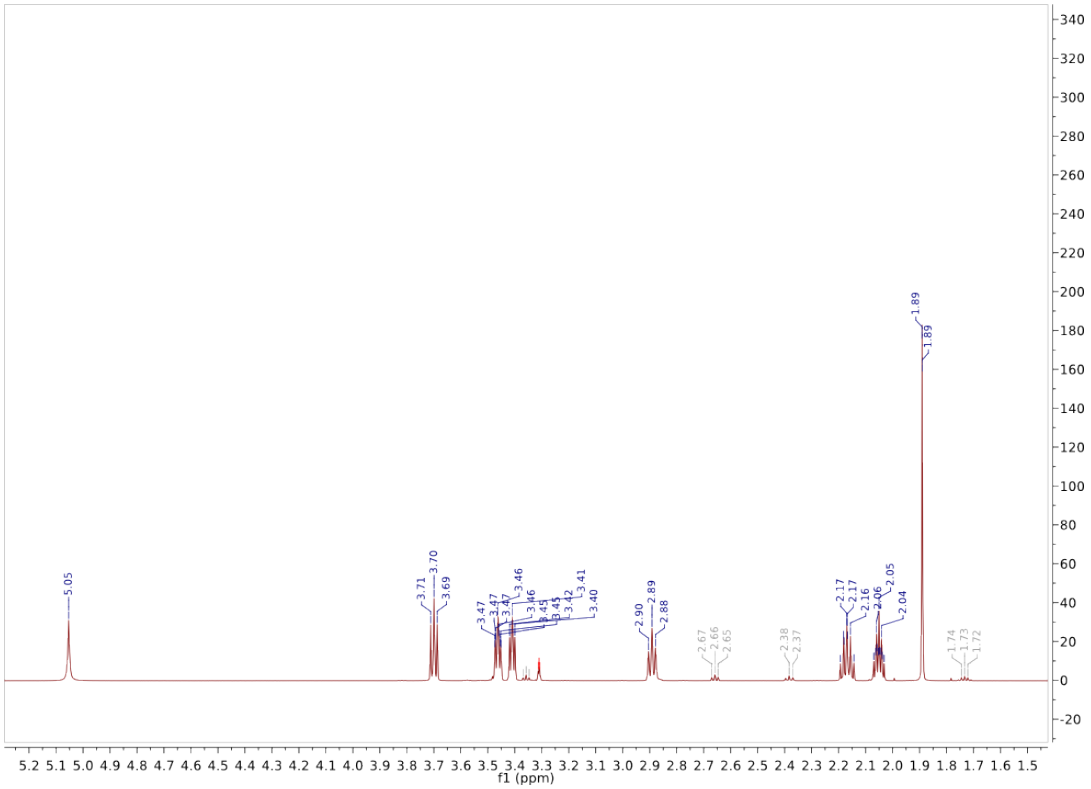
g. S7. [APPH][OAc] HSQC in d_4-MeOH



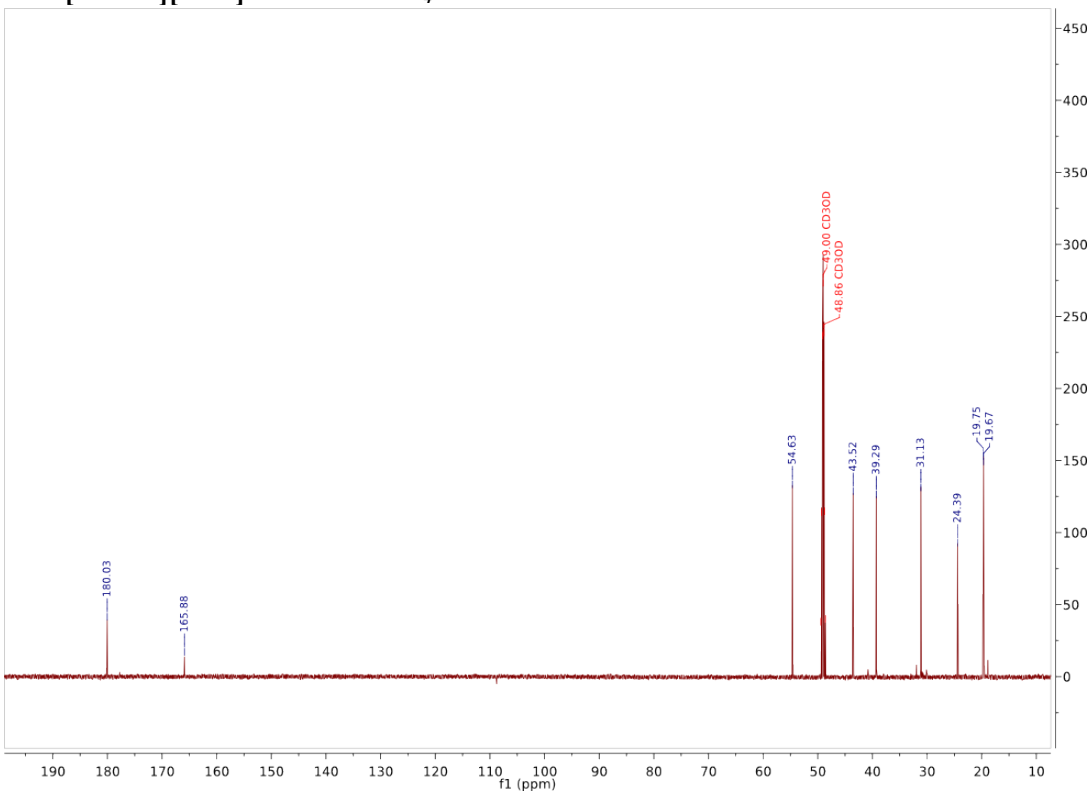
h. S8. [APPH][OAc] TOCSY in d_4-MeOH



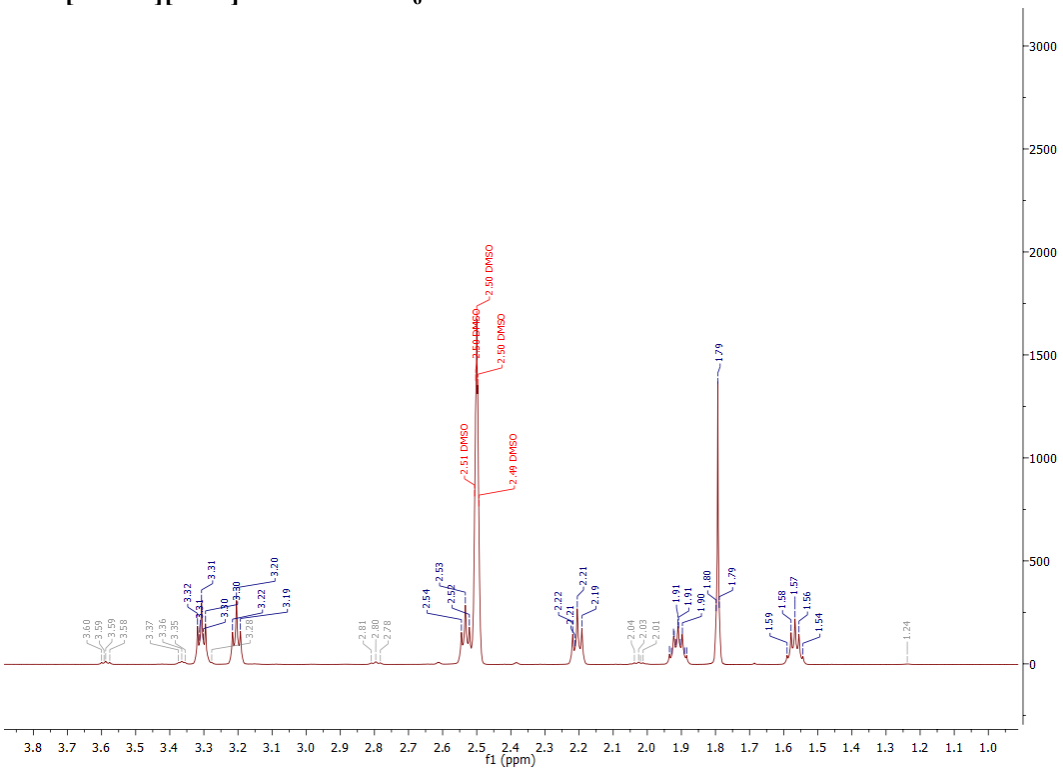
i. S9. [DBNH][OAc] ^{1}H NMR in d_4-MeOH



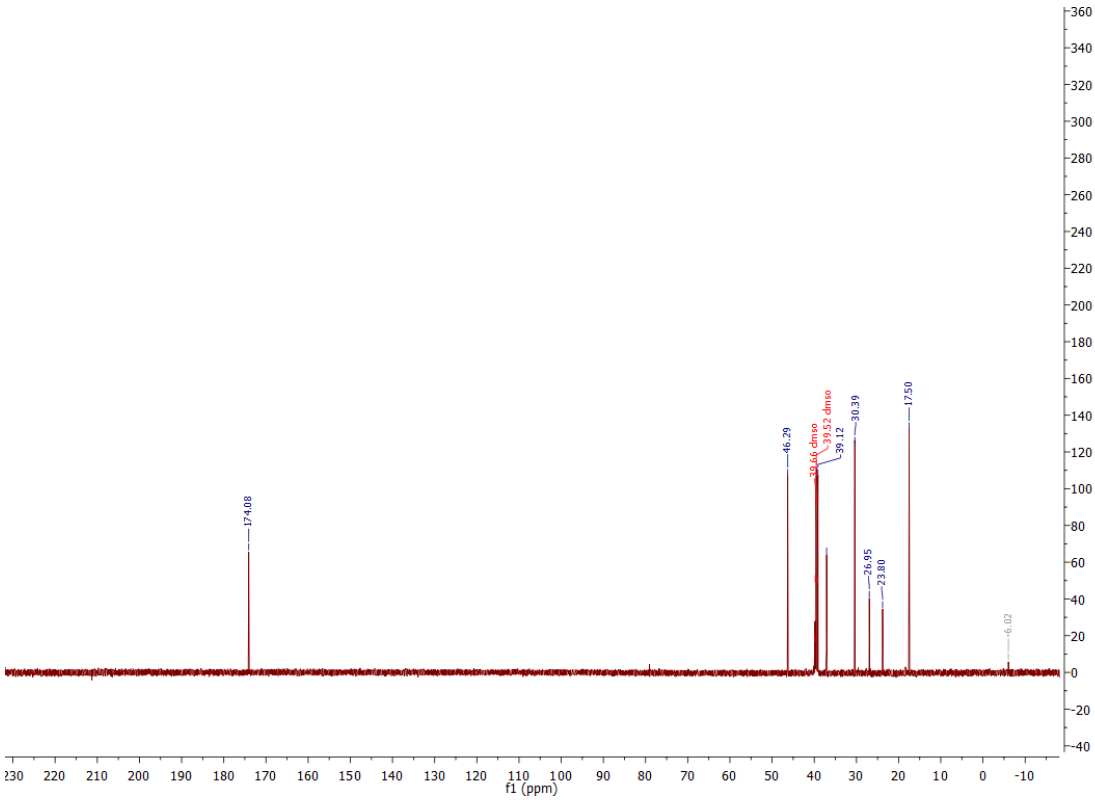
j. S10. [DBNH][OAc] ^{13}C NMR in d_4-MeOH



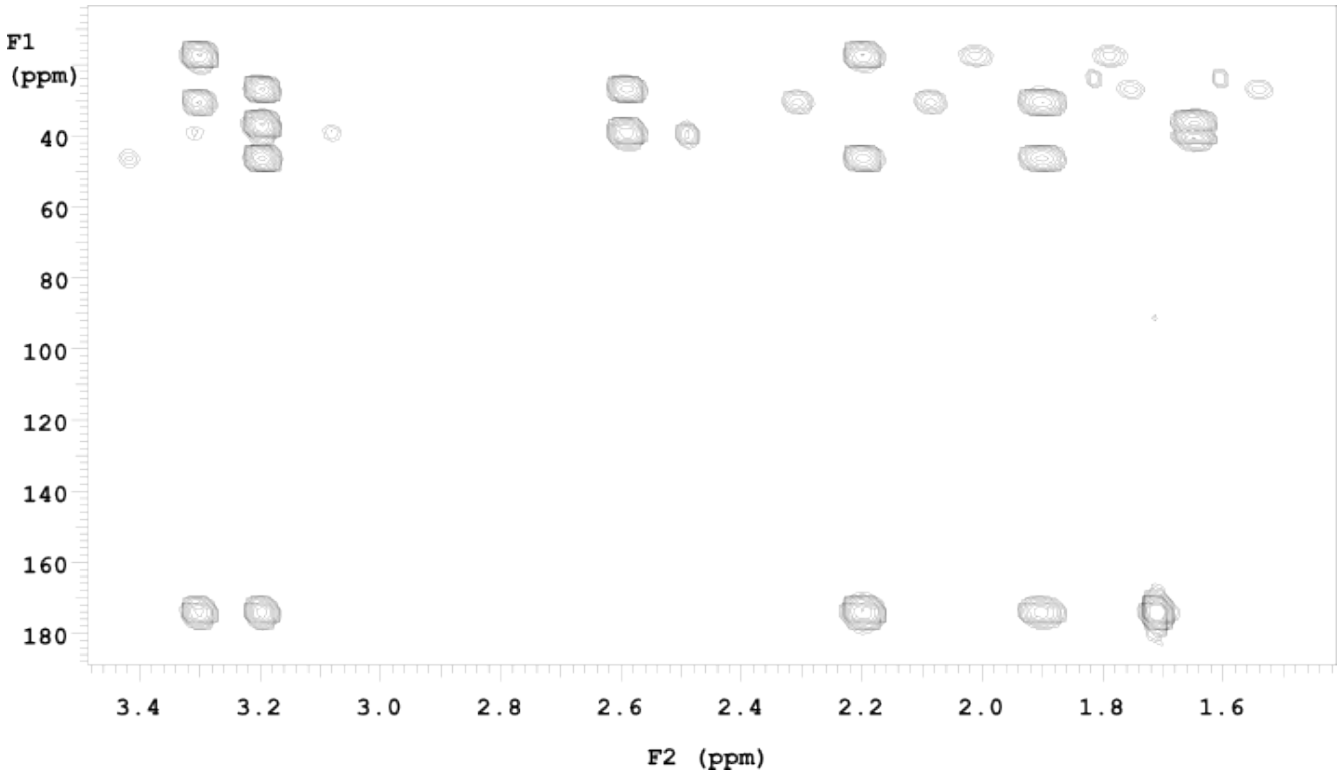
k. S11. [APPH][OAc] ^{1}H NMR in d_6-DMSO



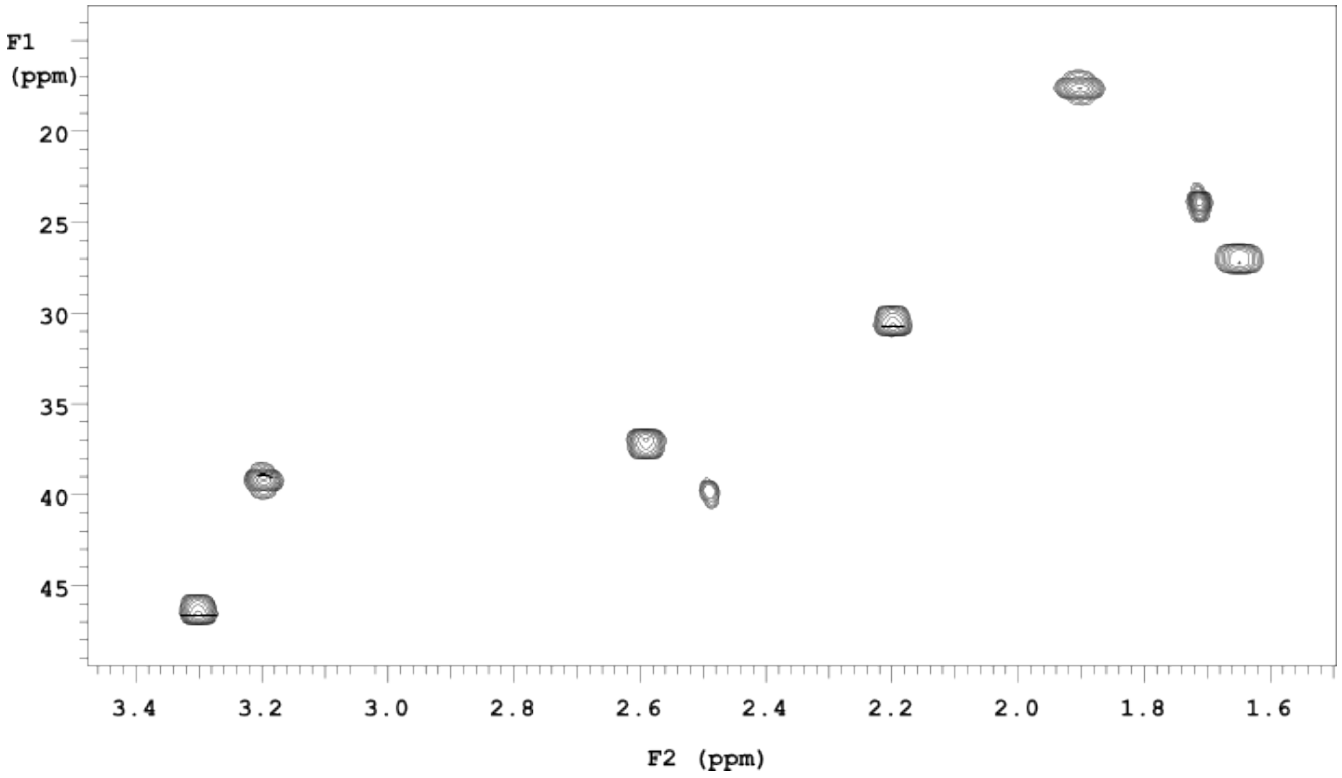
l. S12. [APPH][OAc] ^{13}C NMR in d_6-DMSO



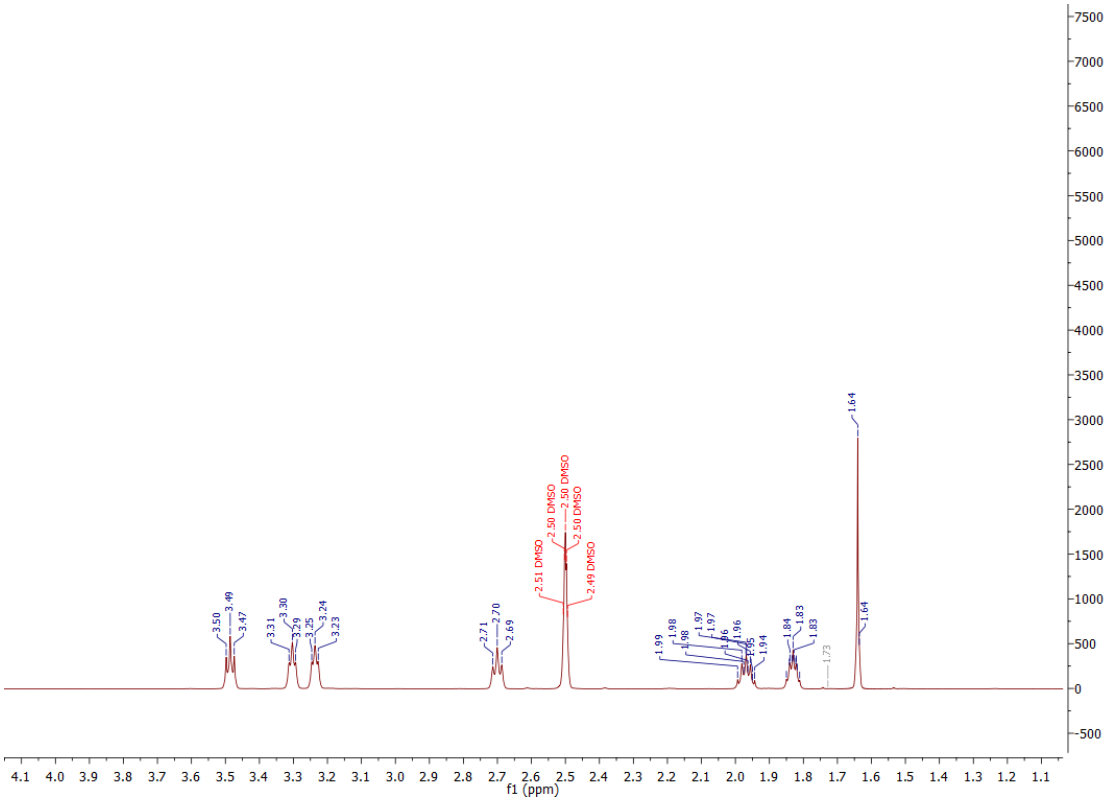
m. S13. [APPH][OAc] HMBC in d_6-DMSO



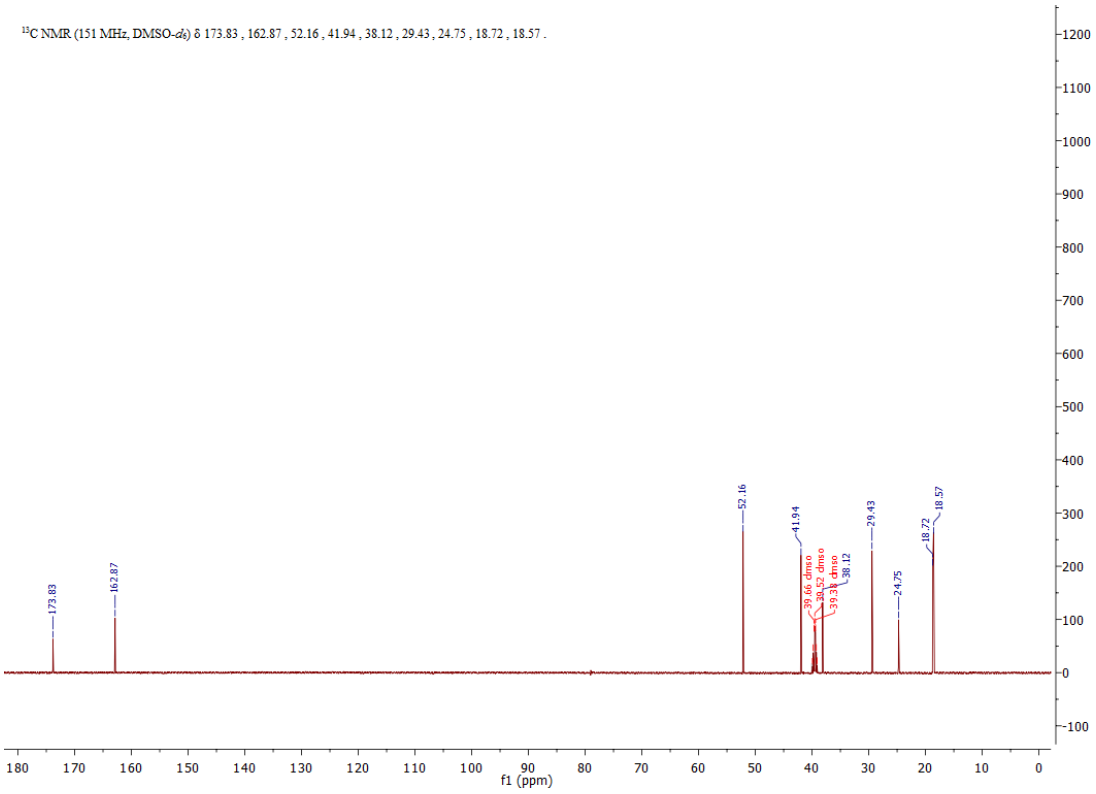
n. S14. [APPH][OAc] HSQC in d_6-DMSO


F1
(ppm)
20
25
30
35
40
45
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
F2 (ppm)

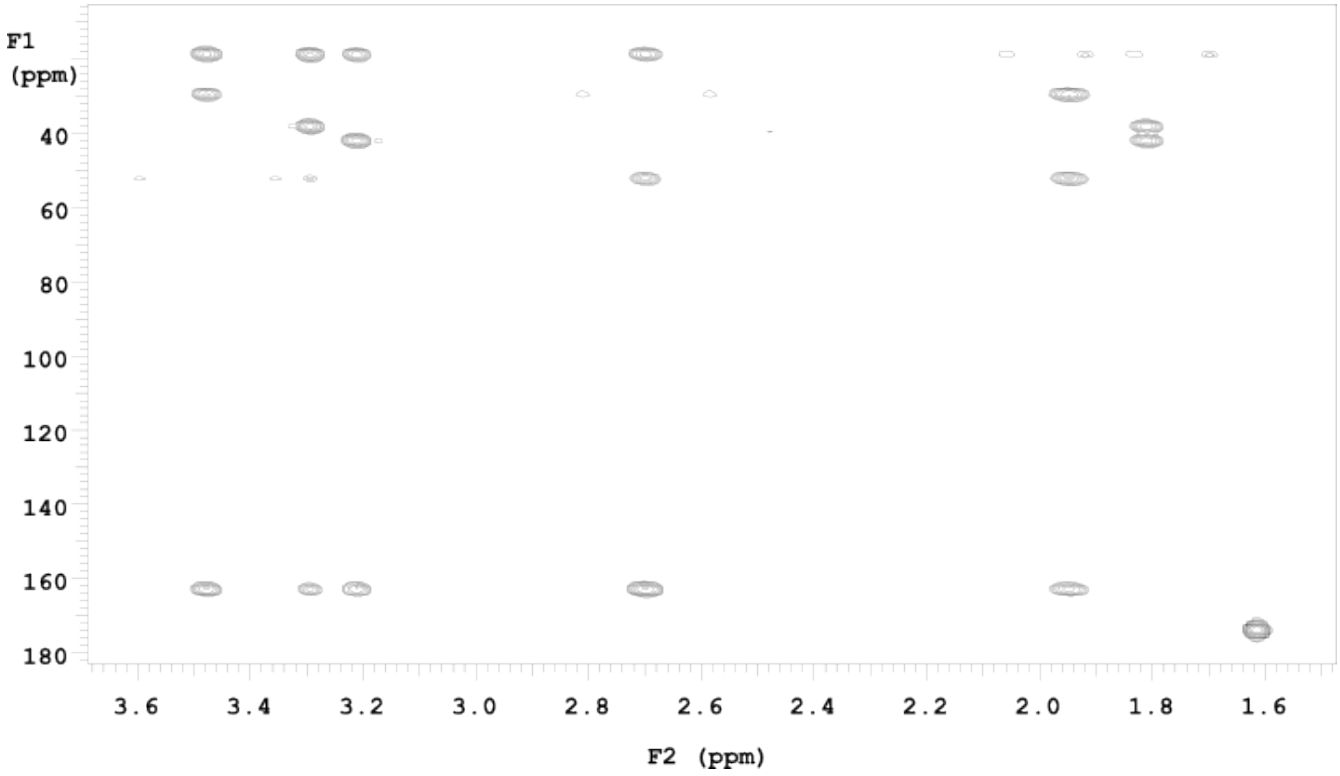
o. S15. [DBNH][OAc] ^{1}H NMR in d_6-DMSO



p. S16. [DBNH][OAc] ^{13}C NMR in d_6-DMSO



q. S17. [DBNH][OAc] HMBC in d_6-DMSO



r. S18. [DBNH][OAc] HSQC in d_6-DMSO