

# Synthesis and Application of Monodisperse Oligo(oxyethylene) Grafted Polystyrene Resins for Solid Phase Organic Synthesis

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## Gel-phase $^{13}\text{C}$ -NMR characterization of target PS-PEG resins (3a-f, 4a-f)

**PS-PEG(2)-O-TBDMS 3a.**  $^{13}\text{C}$  gel-phase NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 72.8$  (t), 69.9 (t), 67.3 (t), 62.8 (t,  $\text{CH}_2\text{-O-Si}$ ), 26.0 (q,  $\text{Si-C-(CH}_3)_3$ ), 18.4 (s,  $\text{Si-C-(CH}_3)_3$ ), -4.9 (q,  $\text{Si-CH}_3$ ) ppm.

**PS-PEG(4)-O-TBDMS 3b.**  $^{13}\text{C}$  gel-phase NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 72.6$  (t), 70.7 (t), 69.8 (t), 67.2 (t), 62.7 (t), 26.0 (q), 18.4 (s), -4.9 (q) ppm.

**PS-PEG(6)-O-TBDMS 3c.**  $^{13}\text{C}$  gel-phase NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 72.6$  (t), 70.7 (t), 69.8 (t), 67.2 (t), 62.7 (t), 26.0 (q), 18.4 (s), -4.9 (q) ppm.

**PS-PEG(8)-O-TBDMS 3d.**  $^{13}\text{C}$  gel-phase NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 72.7$  (t), 70.8 (t), 69.9 (t), 62.8 (t), 26.0 (q), 18.4 (s), -5.2 (q) ppm.

**PS-PEG(10)-O-TBDMS 3e.**  $^{13}\text{C}$  gel-phase NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 72.7$  (t), 70.6 (t), 69.9 (t), 62.8 (t), 26.0 (q), 18.4 (s), -5.2 (q) ppm.

**PS-PEG(12)-O-TBDMS 3f.**  $^{13}\text{C}$  gel-phase NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 72.7$  (t), 70.8 (t), 69.9 (t), 62.8 (t), 26.0 (q), 18.4 (s), -5.2 (q) ppm.

**PS-PEG(2)-OH 4a.**  $^{13}\text{C}$  gel-phase NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 72.6$  (t), 69.8 (t), 67.4 (t), 61.8 (t,  $\text{CH}_2\text{-OH}$ ) ppm.

**PS-PEG(4)-OH 4b.**  $^{13}\text{C}$  gel-phase NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 72.5$  (t), 70.7 (t), 70.6 (t), 70.4 (t), 69.9 (t), 67.3 (t), 61.7 (t) ppm.

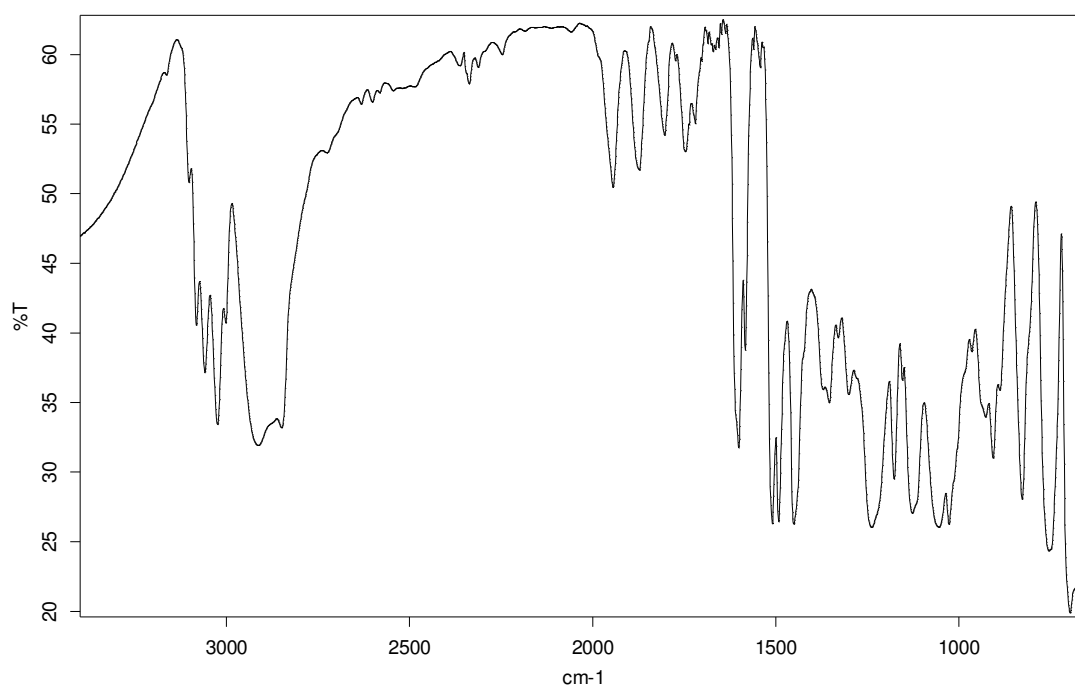
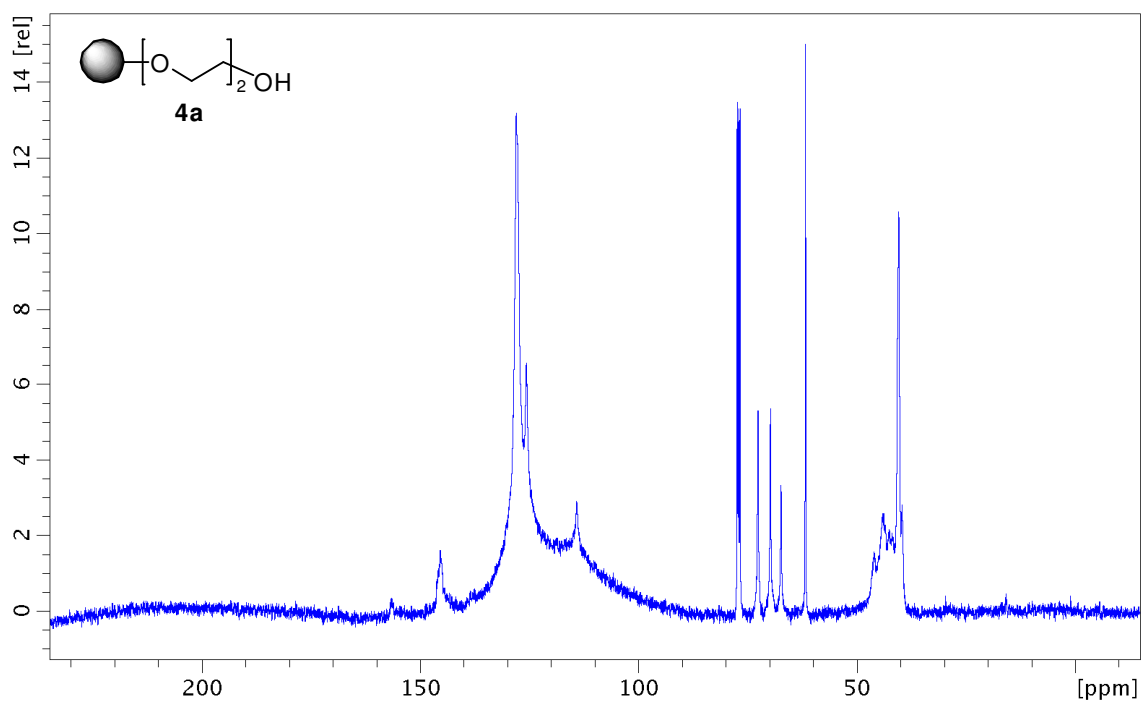
**PS-PEG(6)-OH 4c.**  $^{13}\text{C}$  gel-phase NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 72.5$  (t), 70.6 (t), 70.3 (t), 69.9 (t), 67.3 (t), 61.7 (t) ppm.

**PS-PEG(8)-OH 4d.**  $^{13}\text{C}$  gel-phase NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 72.6$  (t), 70.6 (t), 70.3 (t), 69.9 (t), 67.3 (t), 61.7 (t) ppm.

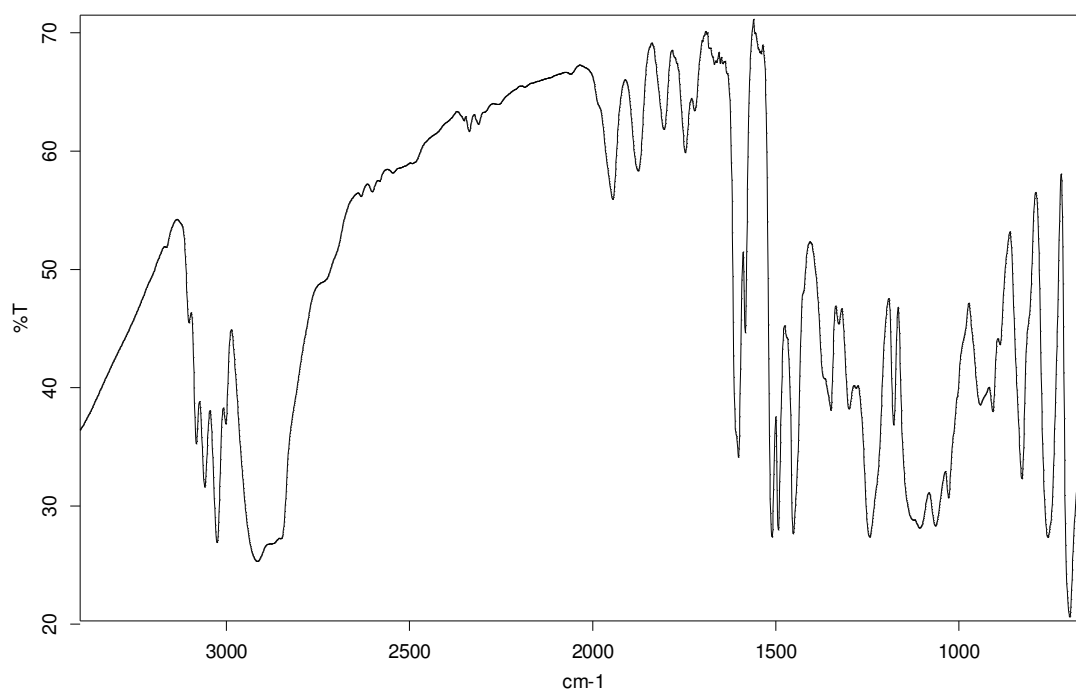
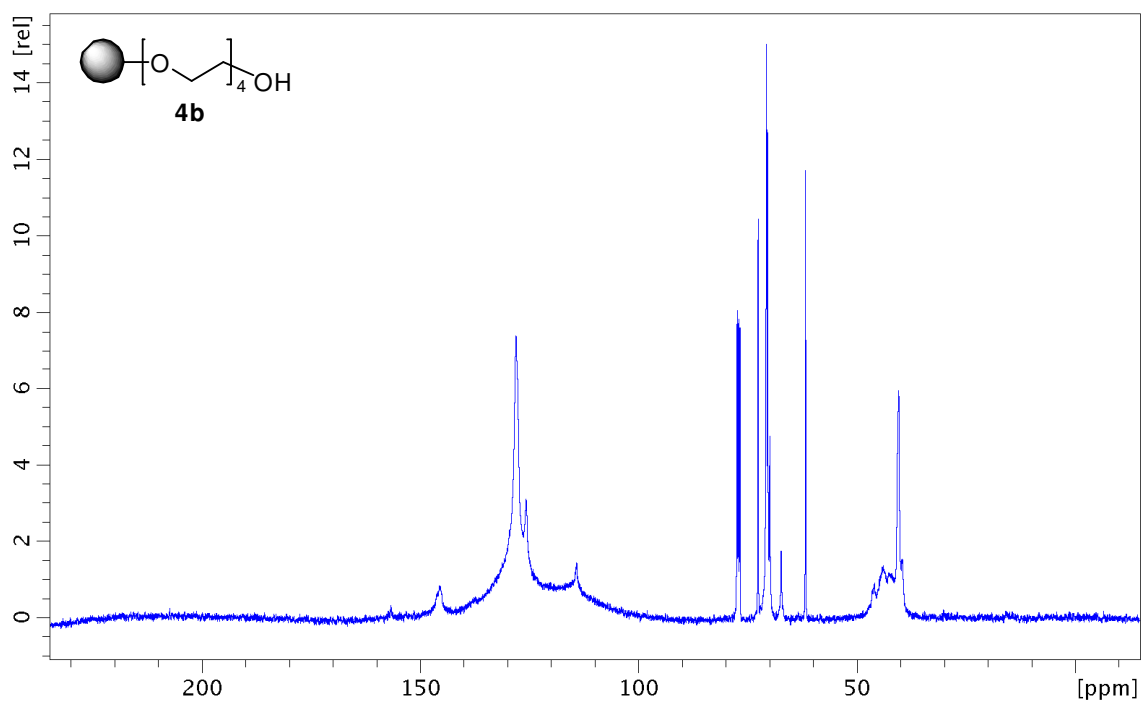
**PS-PEG(10)-OH 4e.**  $^{13}\text{C}$  gel-phase NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 72.6$  (t), 70.6 (t), 70.4 (t), 69.9 (t), 67.3 (t), 61.8 (t) ppm.

**PS-PEG(12)-OH 4f.**  $^{13}\text{C}$  gel-phase NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 72.6$  (t), 70.6 (t), 70.3 (t), 69.9 (t), 67.3 (t), 61.7 (t) ppm.

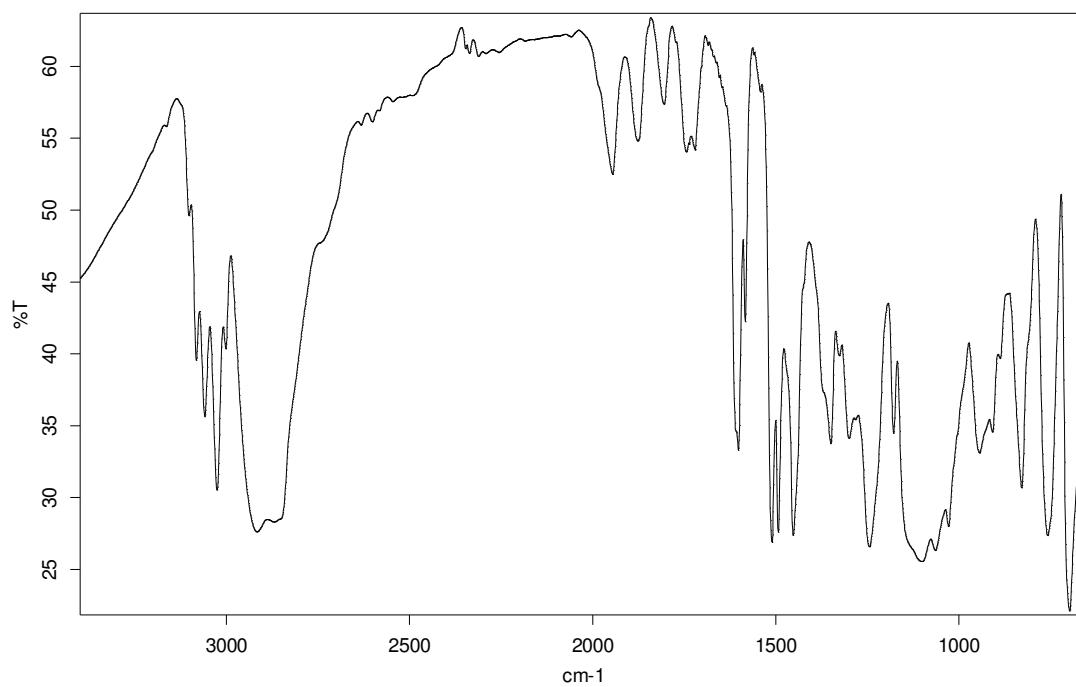
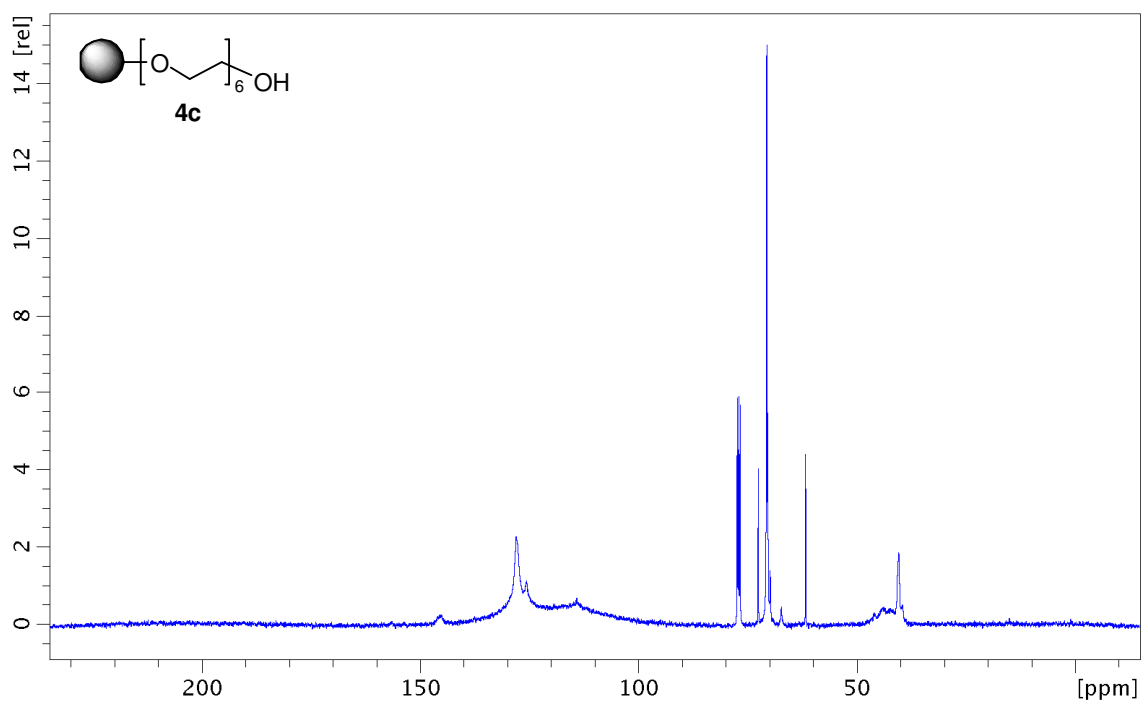
### Gel-phase $^{13}\text{C}$ -NMR and IR spectra of PS-PEG-2-OH (4a)



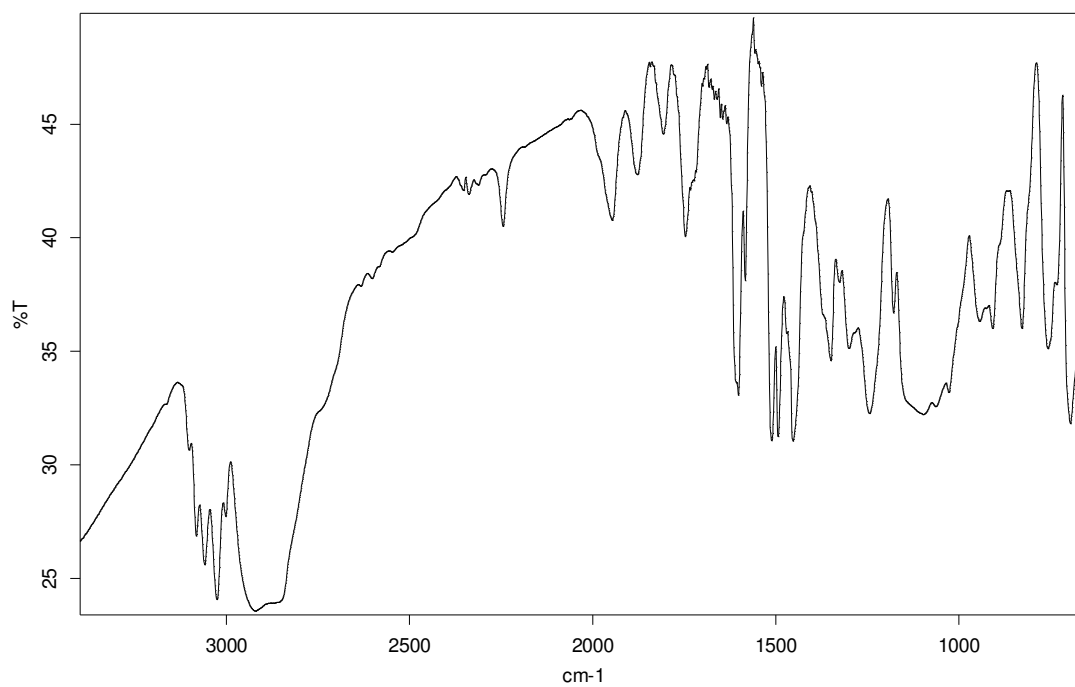
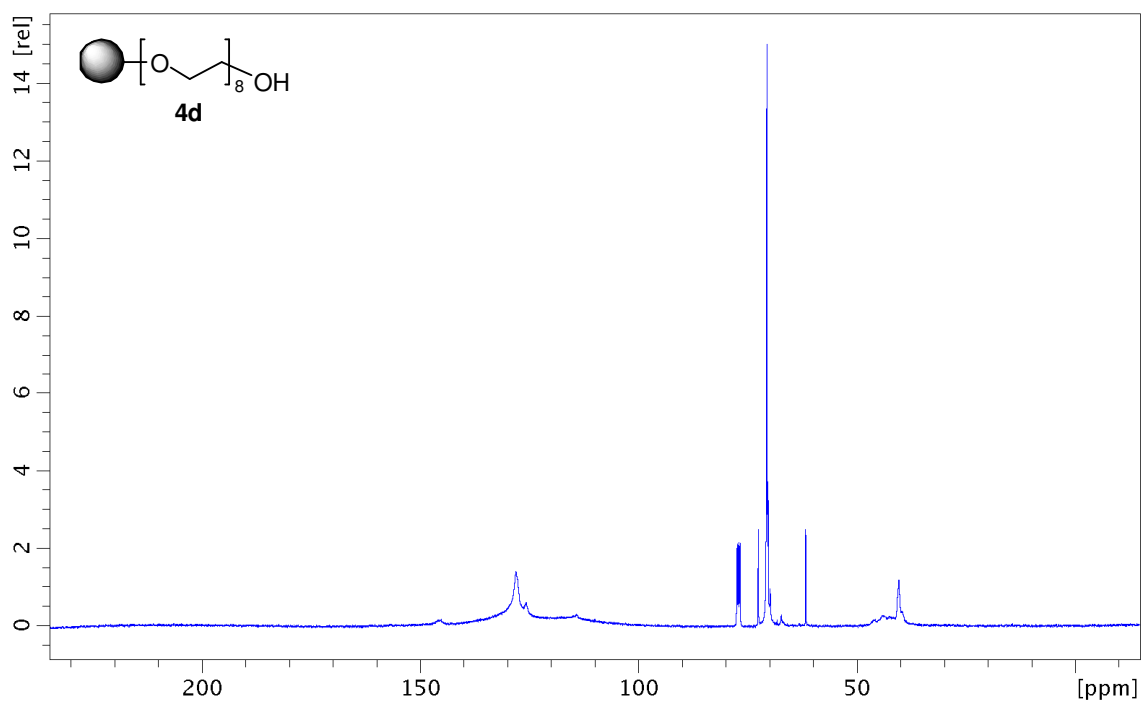
# Gel-phase $^{13}\text{C}$ -NMR and IR spectra of PS-PEG-4-OH (4b)



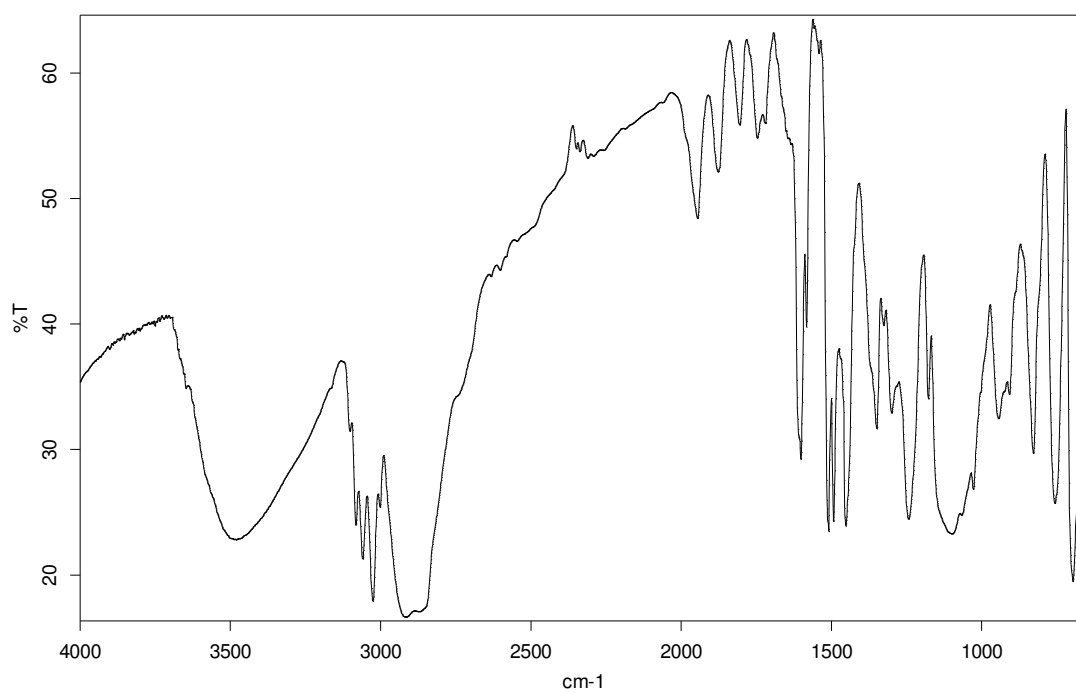
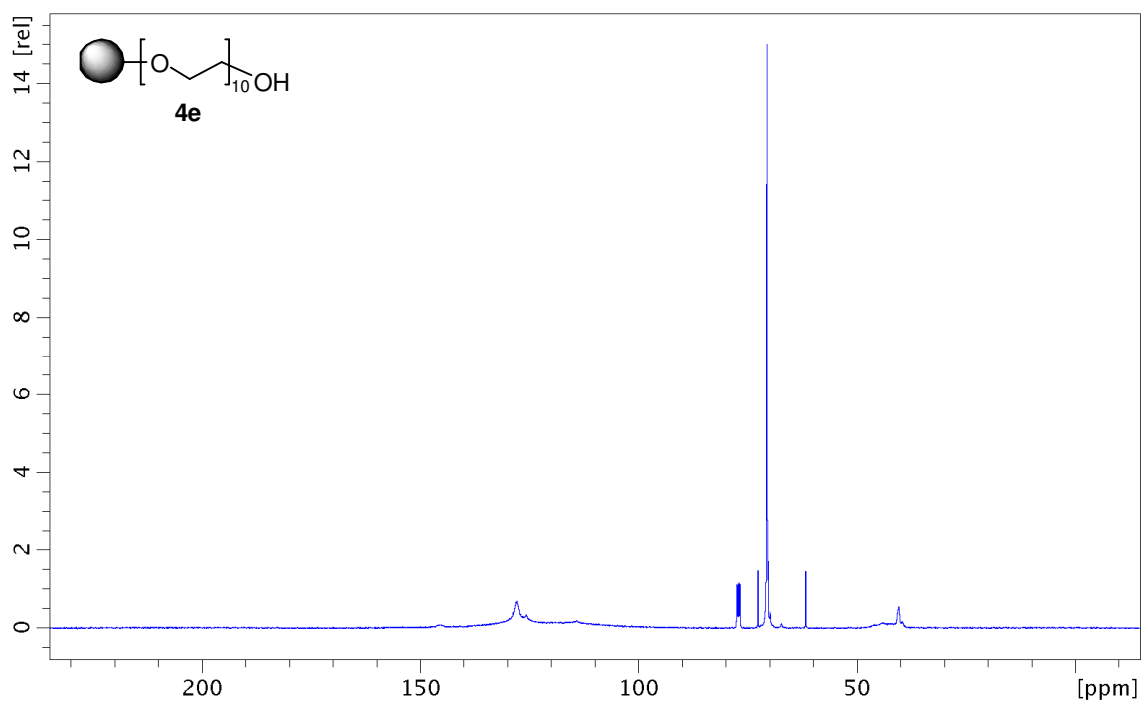
# Gel-phase $^{13}\text{C}$ -NMR and IR spectra of PS-PEG-6-OH (4c)



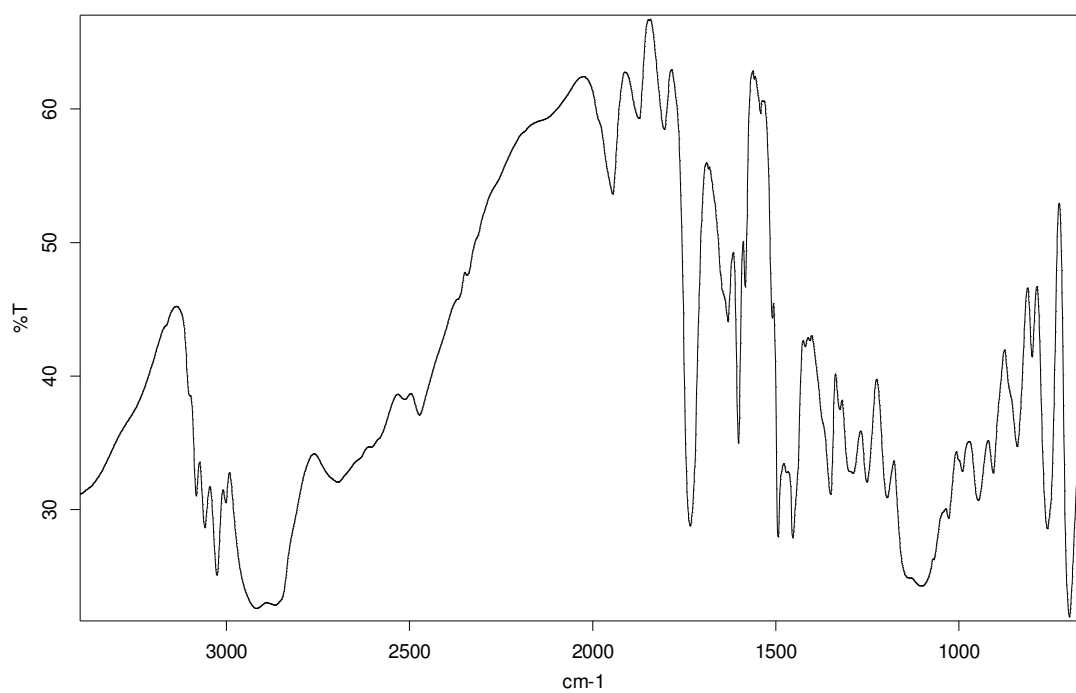
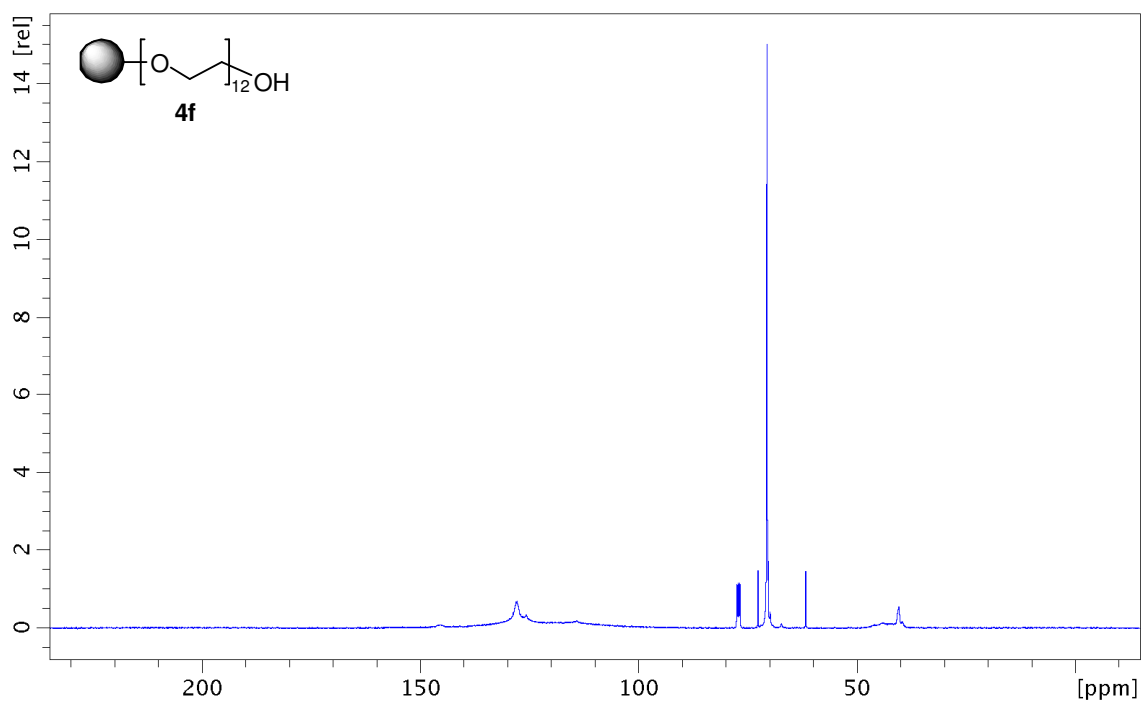
# Gel-phase $^{13}\text{C}$ -NMR and IR spectra of PS-PEG-8-OH (4d)



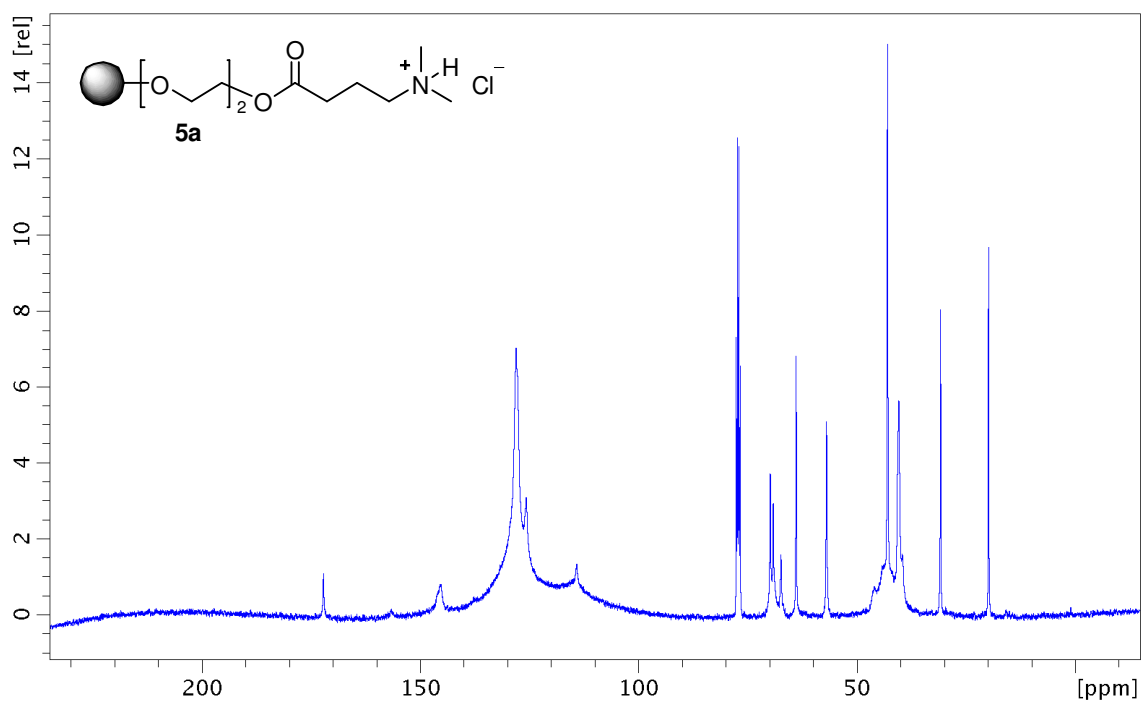
# Gel-phase $^{13}\text{C}$ -NMR and IR spectra of PS-PEG-10-OH (4e)



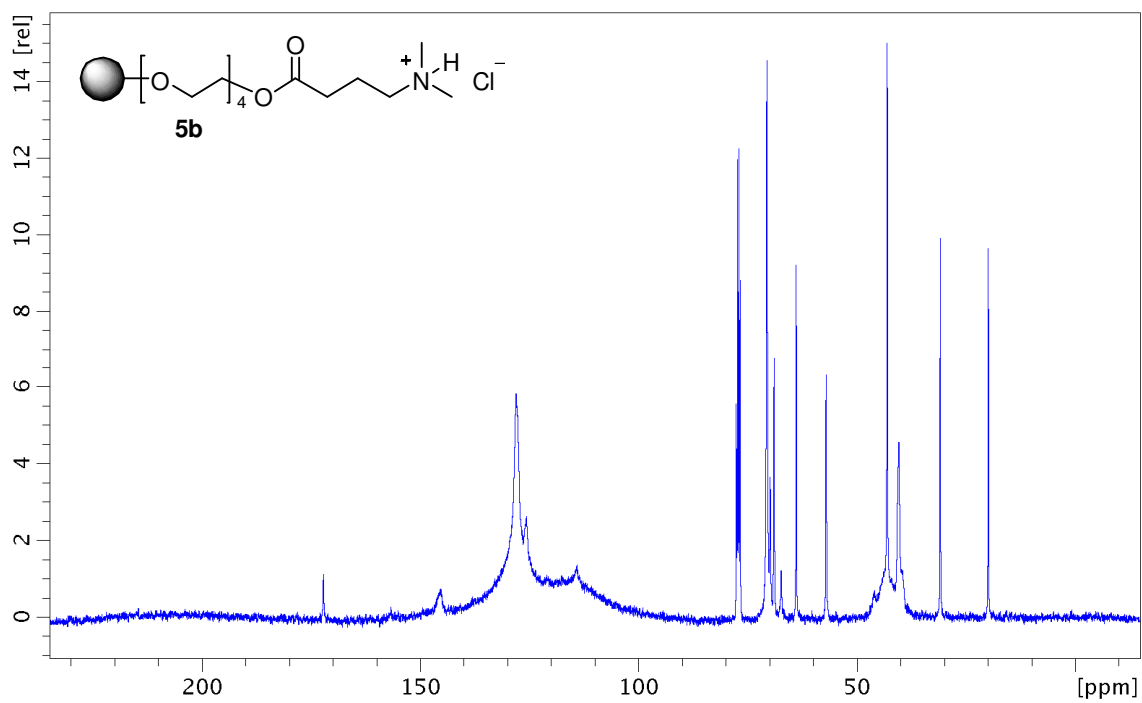
# Gel-phase $^{13}\text{C}$ -NMR and IR spectra of PS-PEG-12-OH (4f)



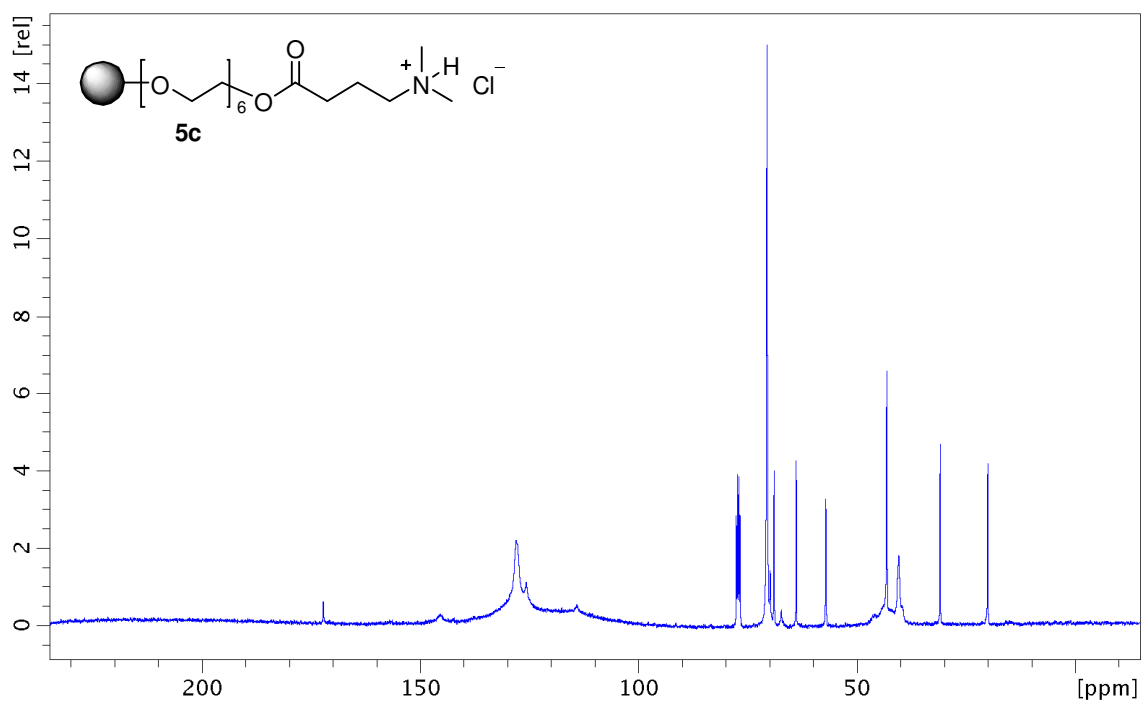
**Gel-phase  $^{13}\text{C}$ -NMR spectrum of PS-PEG-2-N1-C3-HCl (5a)**



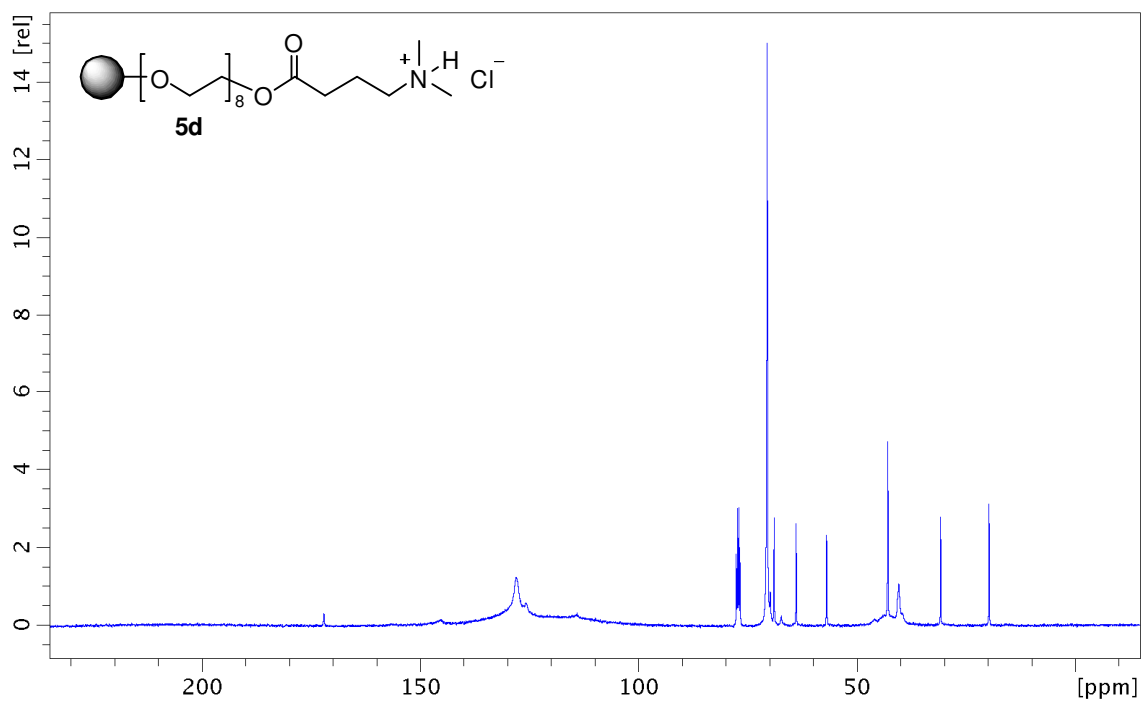
**Gel-phase  $^{13}\text{C}$ -NMR spectrum of PS-PEG-4-N1-C3-HCl (5b)**



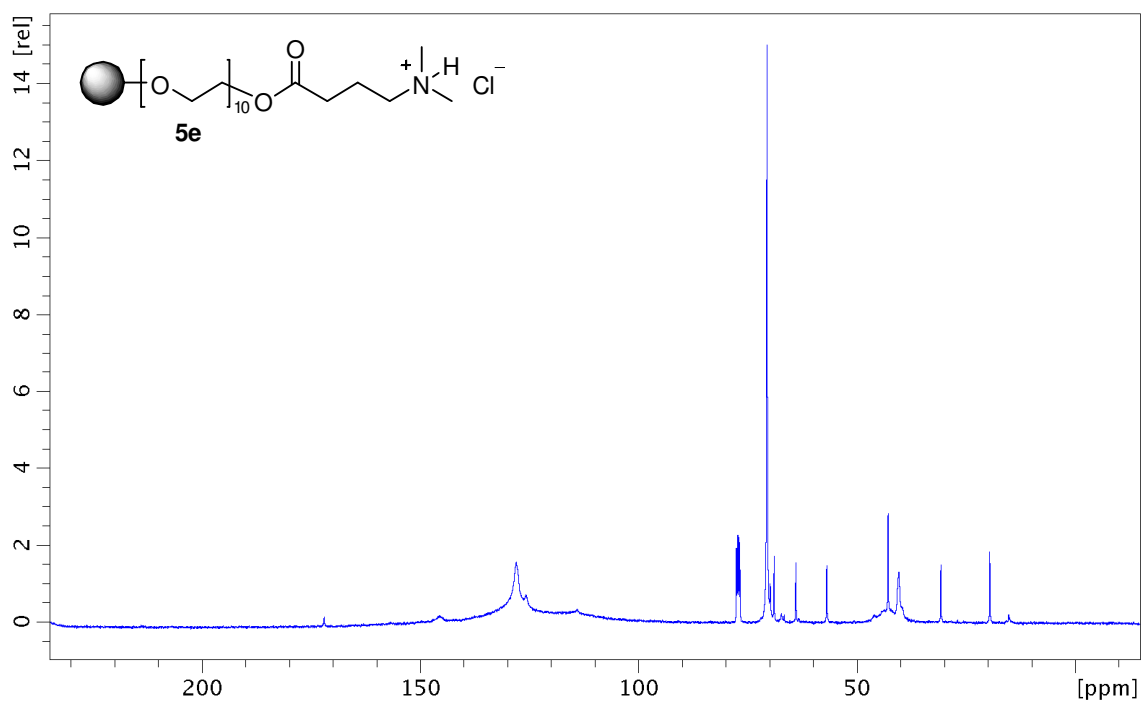
**Gel-phase  $^{13}\text{C}$ -NMR spectrum of PS-PEG-6-N1-C3-HCl (5c)**



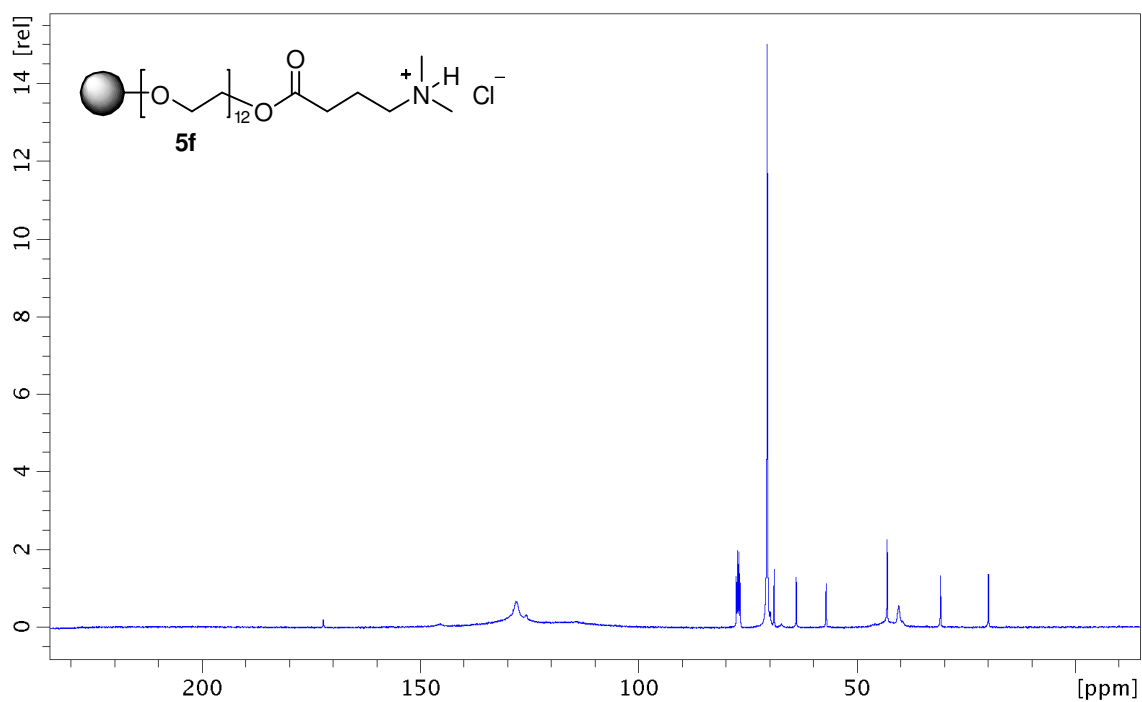
**Gel-phase  $^{13}\text{C}$ -NMR spectrum of PS-PEG-8-N1-C3-HCl (5d)**



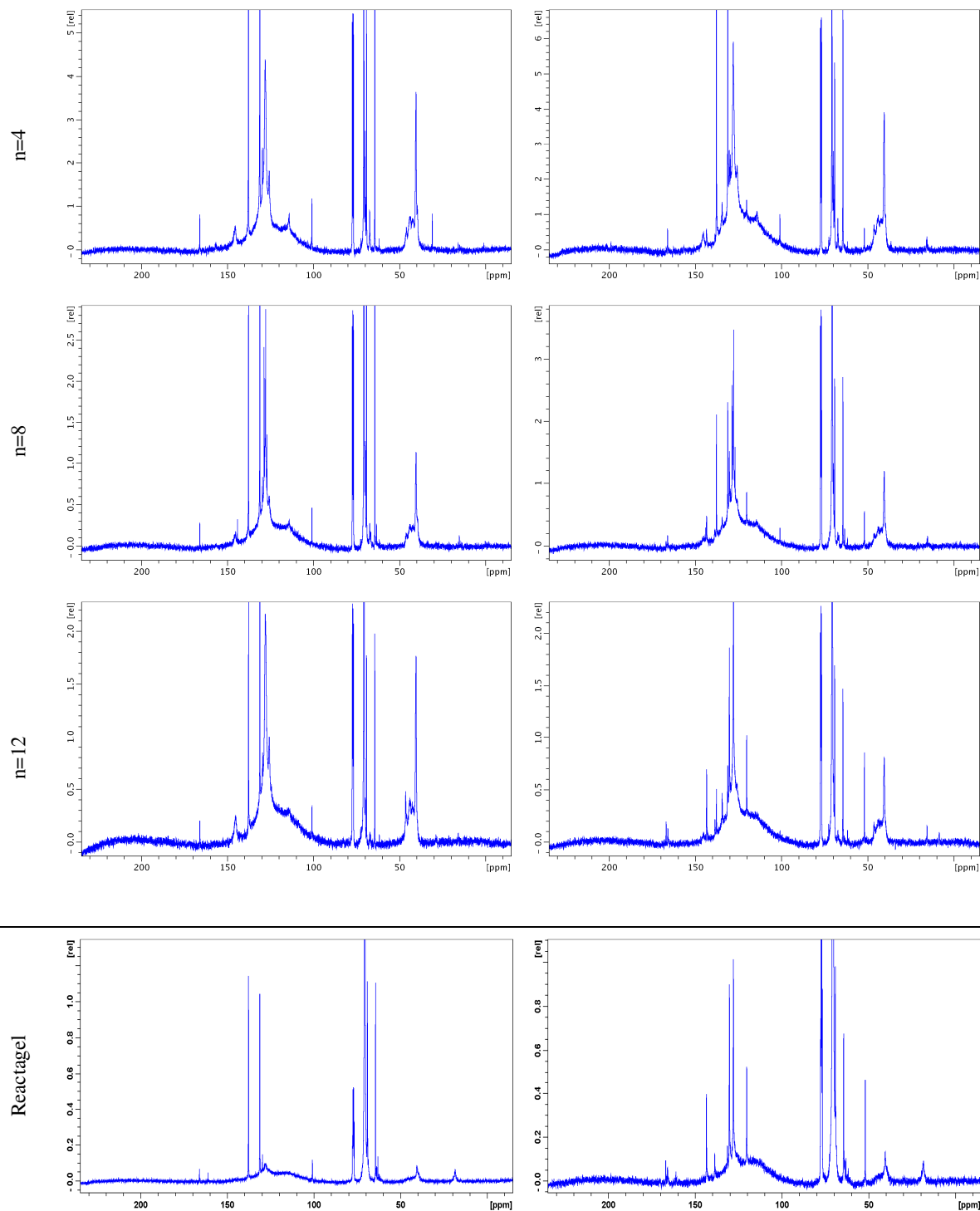
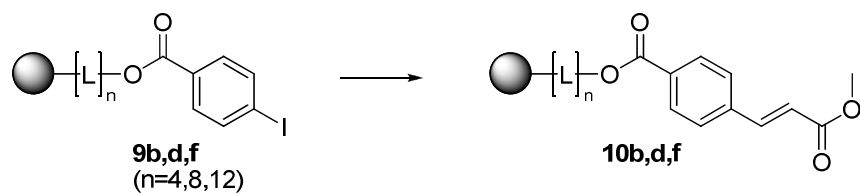
**Gel-phase  $^{13}\text{C}$ -NMR spectrum of PS-PEG-10-N1-C3-HCl (5e)**



**Gel-phase  $^{13}\text{C}$ -NMR spectrum of PS-PEG-12-N1-C3-HCl (5f)**



## Gel-phase $^{13}\text{C}$ -NMR spectra of application example 2 (Heck coupling)



### Gel phase $^{13}\text{C}$ -NMR reaction monitoring of resins 3d-5d

