

Poly(4-Dodecylstyrene) as a Phase-Selectively Soluble Polymer Support in Homogeneous Catalysis

Tatyana V. Khamaturova and David E. Bergbreiter*

Department of Chemistry, Texas A&M University, College Station, Texas 77842-3012, USA

Supporting Information

Phase Selective Solubility of Copolymers 5 and 6. Copolymers **5** ($M_n = 32000$ Da, PDI = 2.22, with the loading of the dansyl groups = 0.32 mmol/g) and **6** ($M_n = 22000$ Da, PDI = 2.86, with the loading of dansyl groups = 0.46 mmol/g) were each dissolved in 10 mL of heptane (10mL) and their solutions were diluted by 1000-fold and the fluorescence ($\lambda_{EX} = 355$ nm, $\lambda_{EM} = 500$ nm) of the resulting solutions were measured. These heptane solutions were then used to prepare a calibration curve that was used to determine the concentration of dansyl fluorophore-labeled **5** or **6** in acetonitrile phases from original polymer containing heptane solutions that had been 1.4 mM and 1.3 mM. Four consecutive washings of polymer containing heptane phase with acetonitrile phase were performed for each of the poly(4-alkylstyrene) copolymers. Fluorescence spectra of acetonitrile solutions of polymers **5** and **6** are shown in Figure 1.

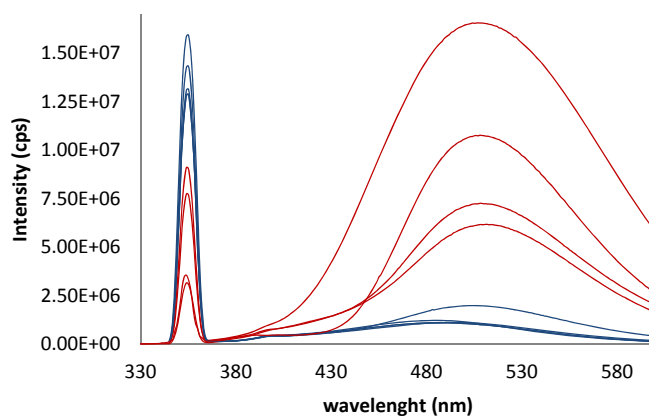
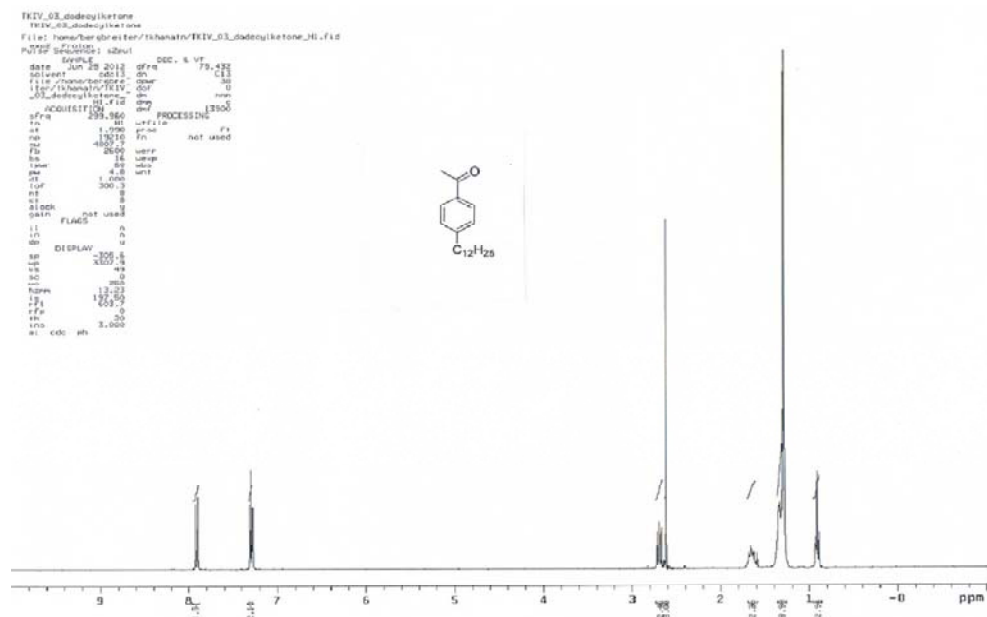
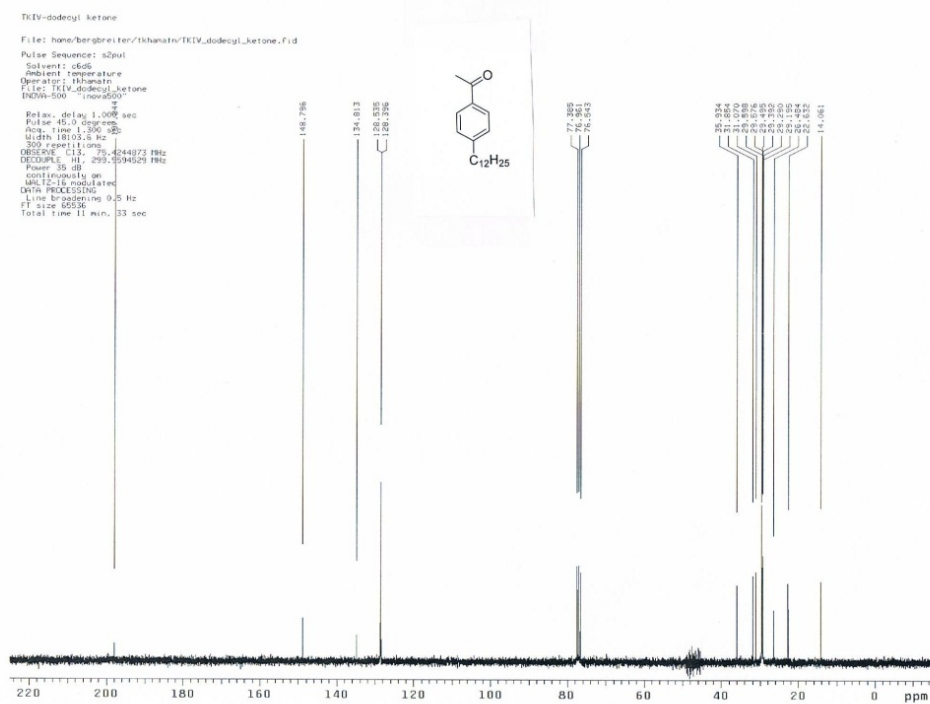


Figure 1. Fluorescence spectra of acetonitrile solutions for poly(4-dodecylstyrene) copolymer **5** blue and for poly(4-tert-butylstyrene) copolymer **6** red.

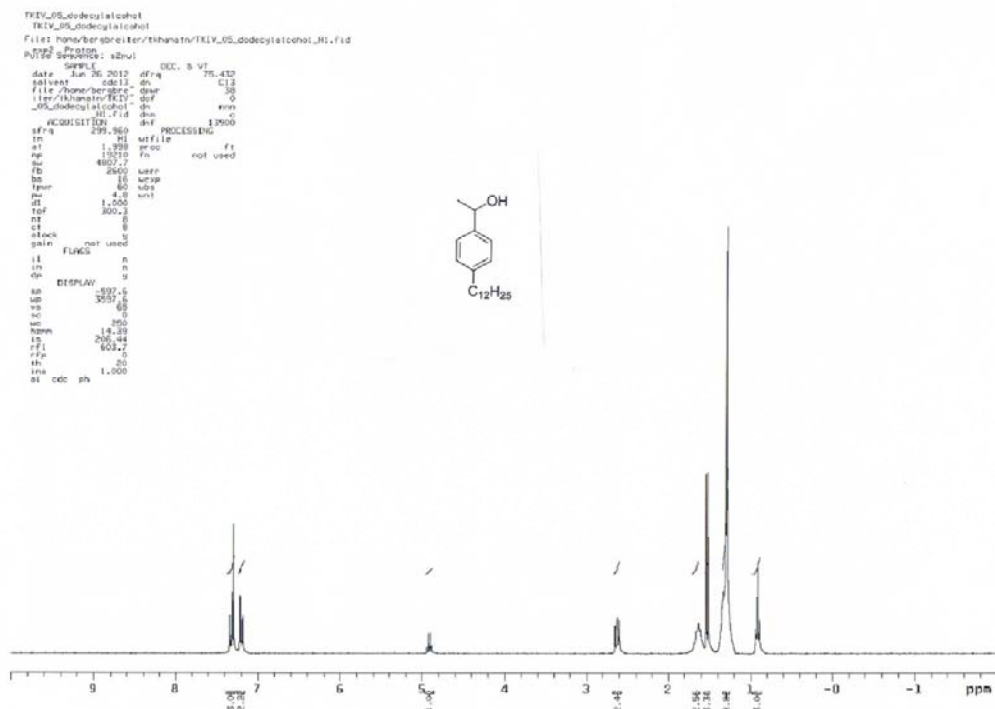
¹H NMR Spectrum of 4-Dodecylacetophenone



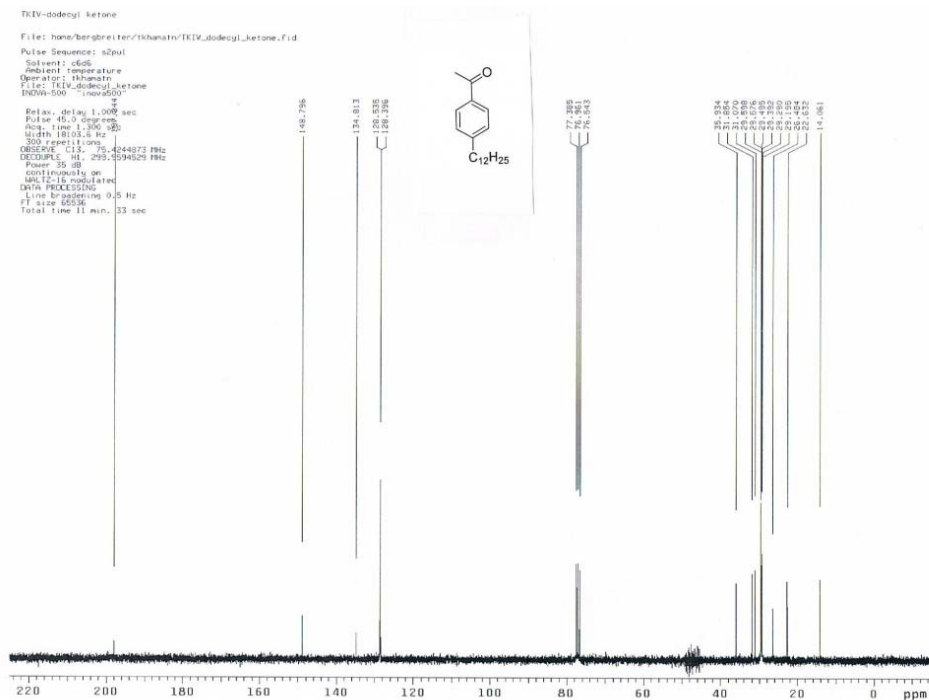
¹³C NMR Spectrum of 4-Dodecylacetophenone



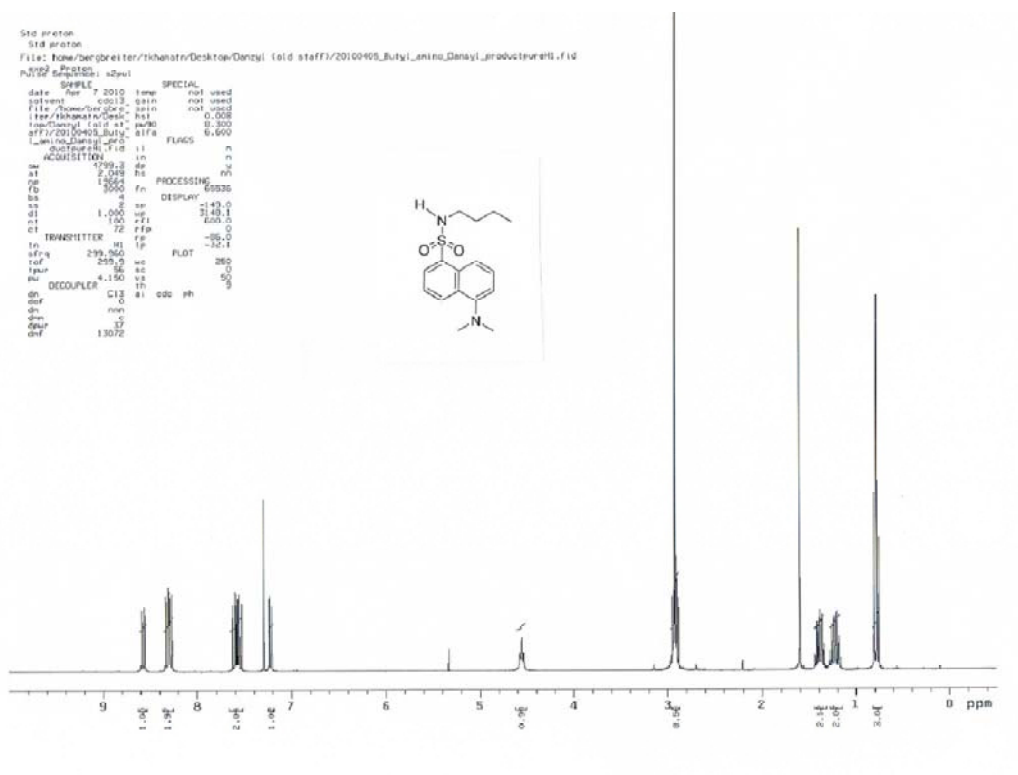
¹H NMR of (4-n-dodecylphenyl)ethanol



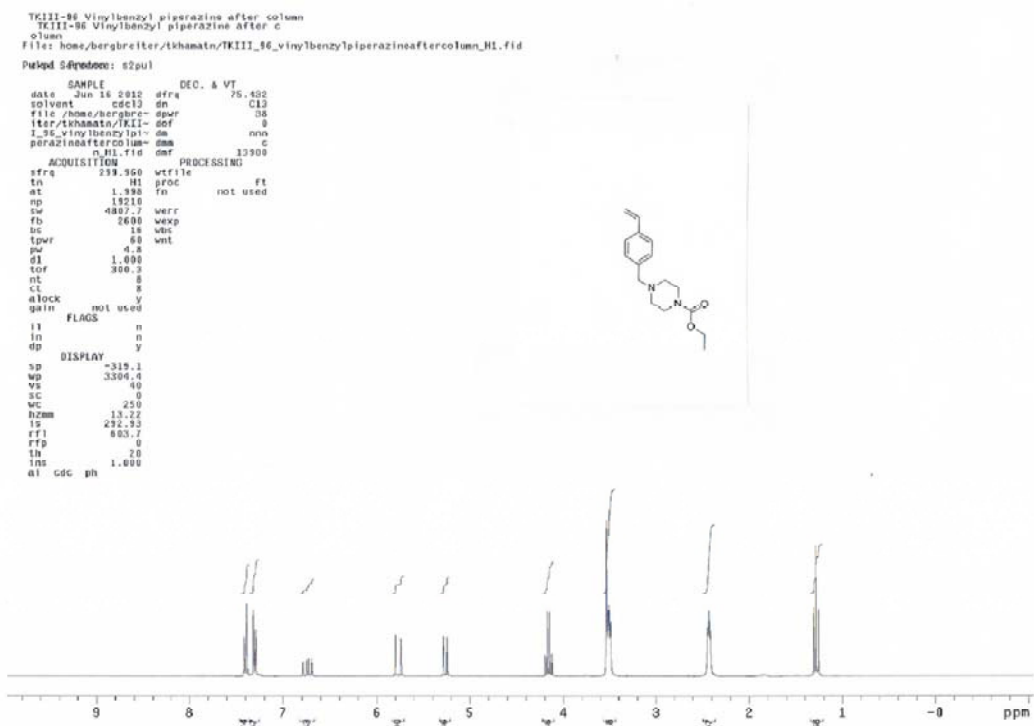
¹³C NMR of (4-n-dodecylphenyl)ethanol



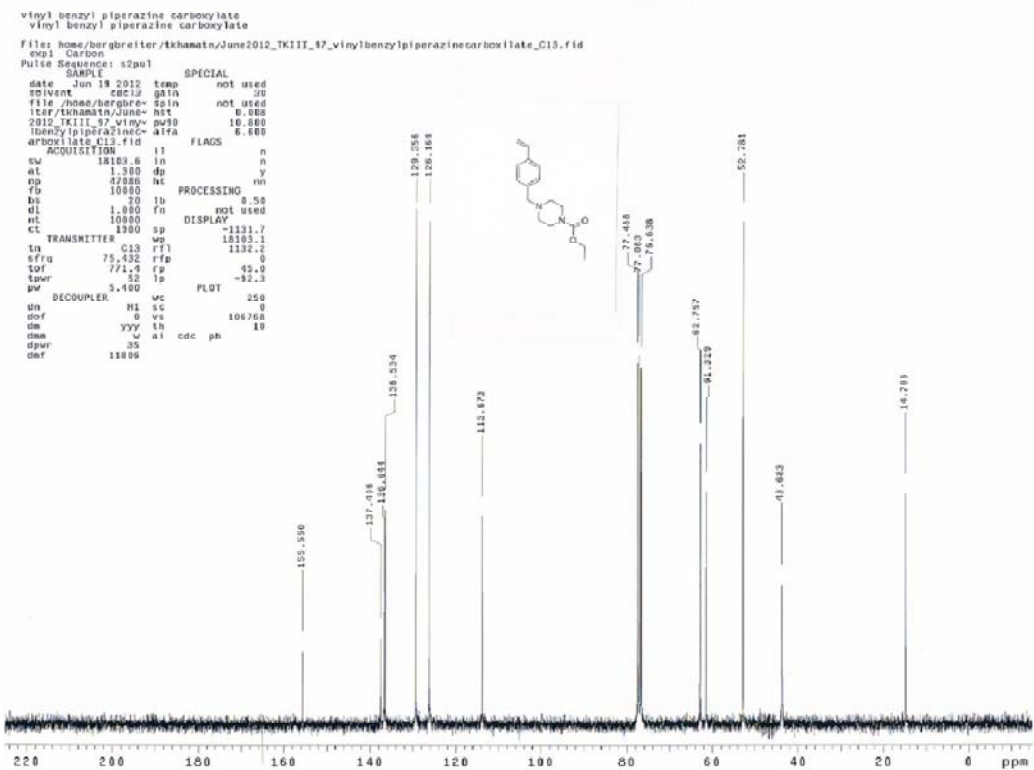
^1H NMR of *N*-*n*-butyl dansylsulfonamide



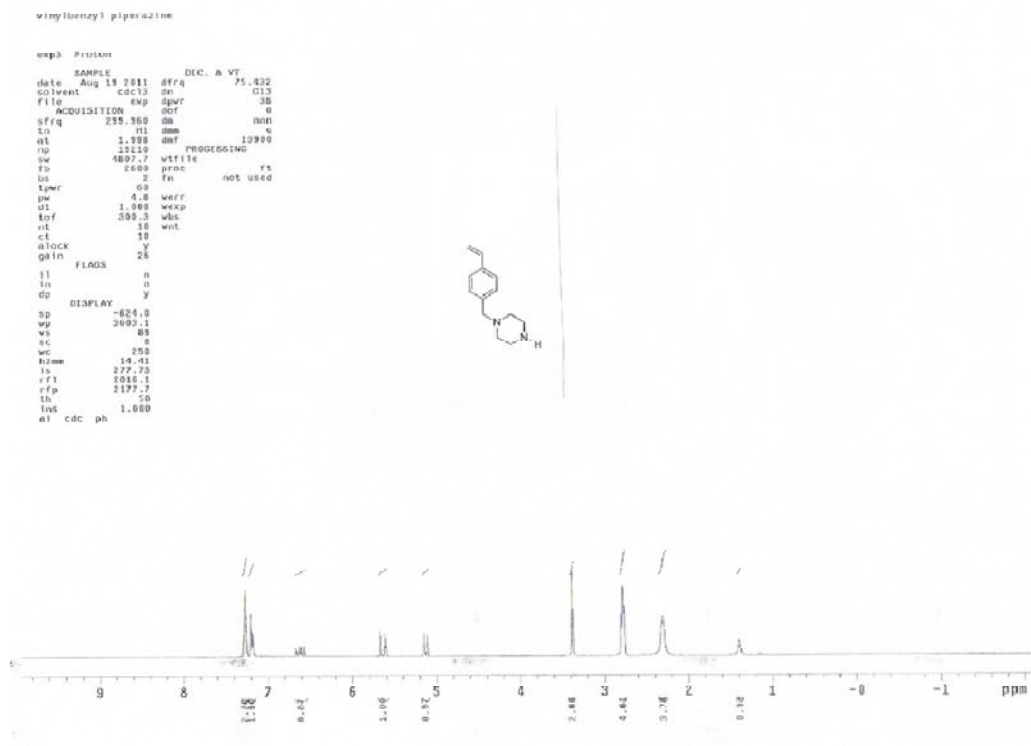
¹H NMR of 1-carboethoxy-4-vinylbenzylpiperazine.



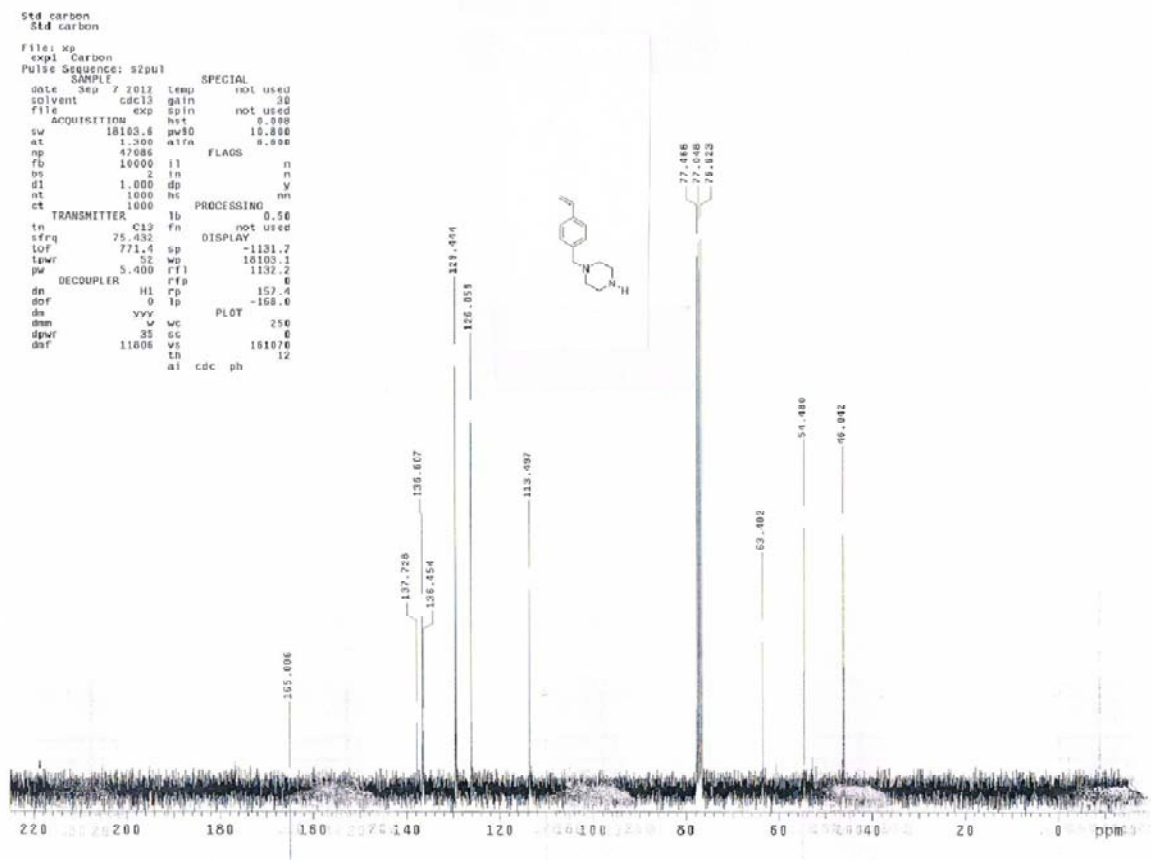
¹³C NMR of 1-carboethoxy-4-(4-vinylbenzyl)piperazine.



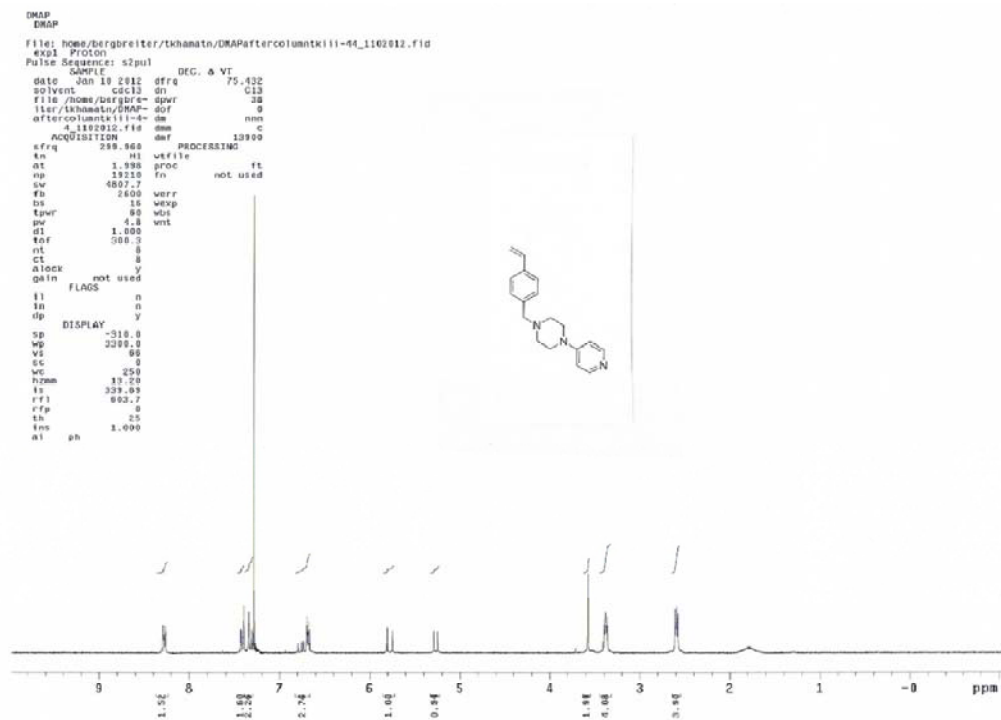
¹H NMR of 1-(4-vinylbenzyl)piperazine.



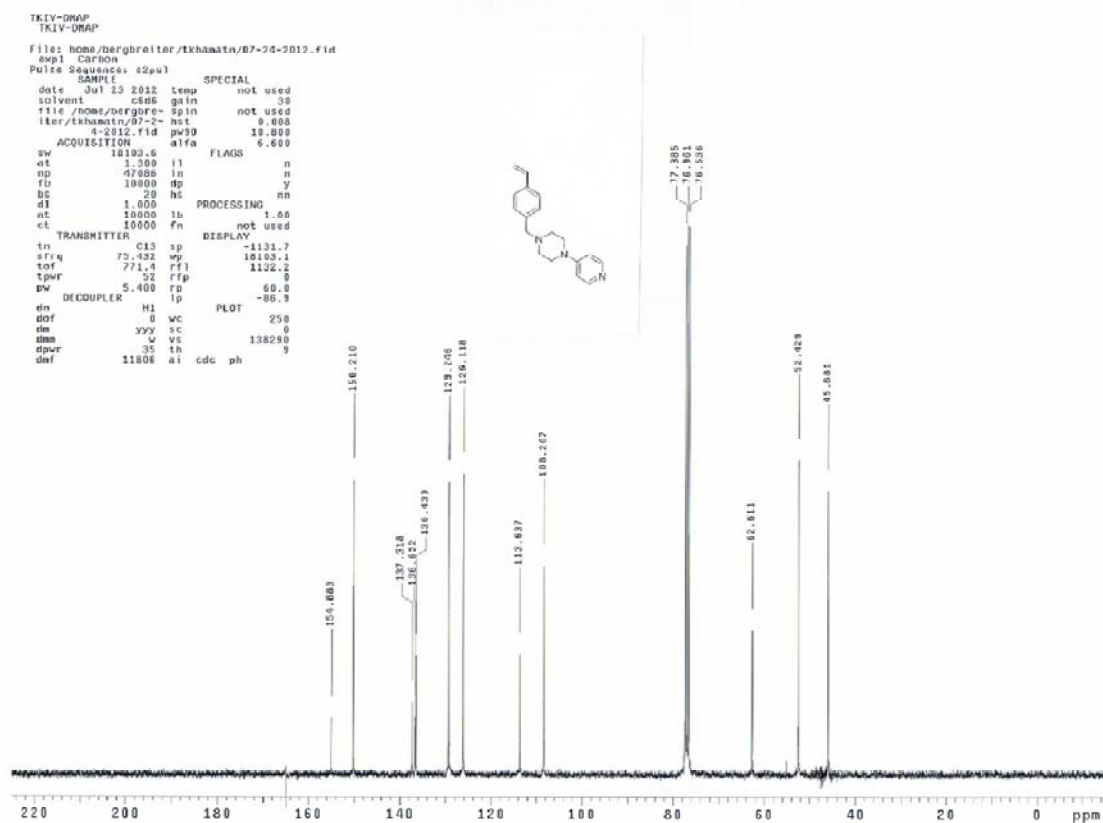
¹³C NMR of 1-(4-vinylbenzyl)piperazine.



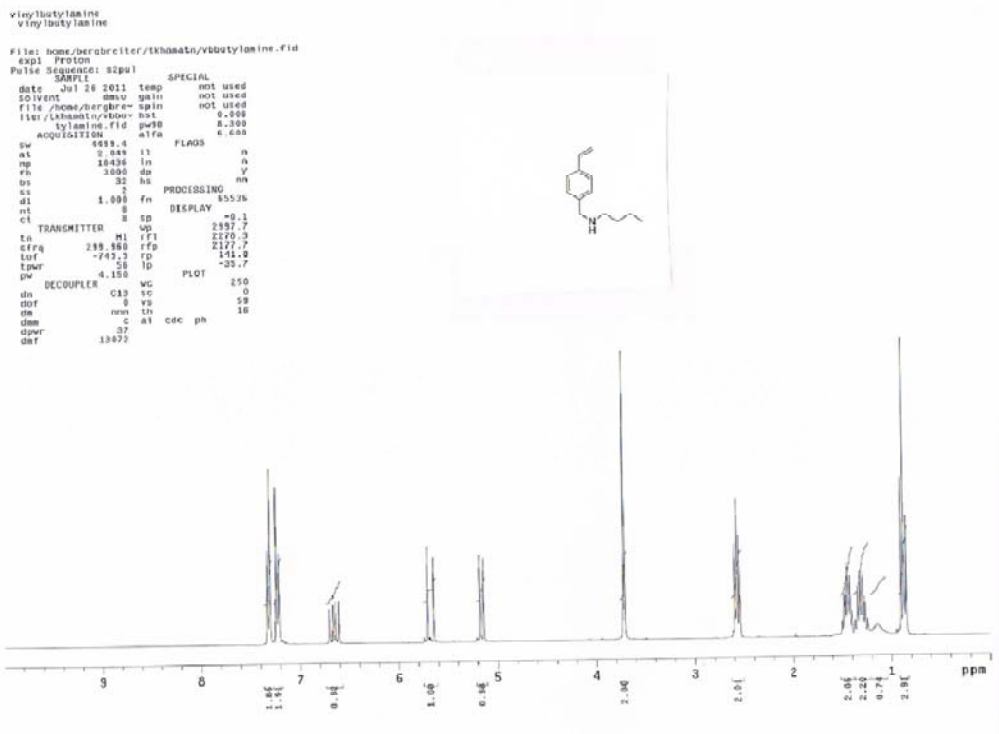
¹H NMR of 1-(4-N-vinylbenzyl)-4-N-(4-pyridyl)piperazine 7.



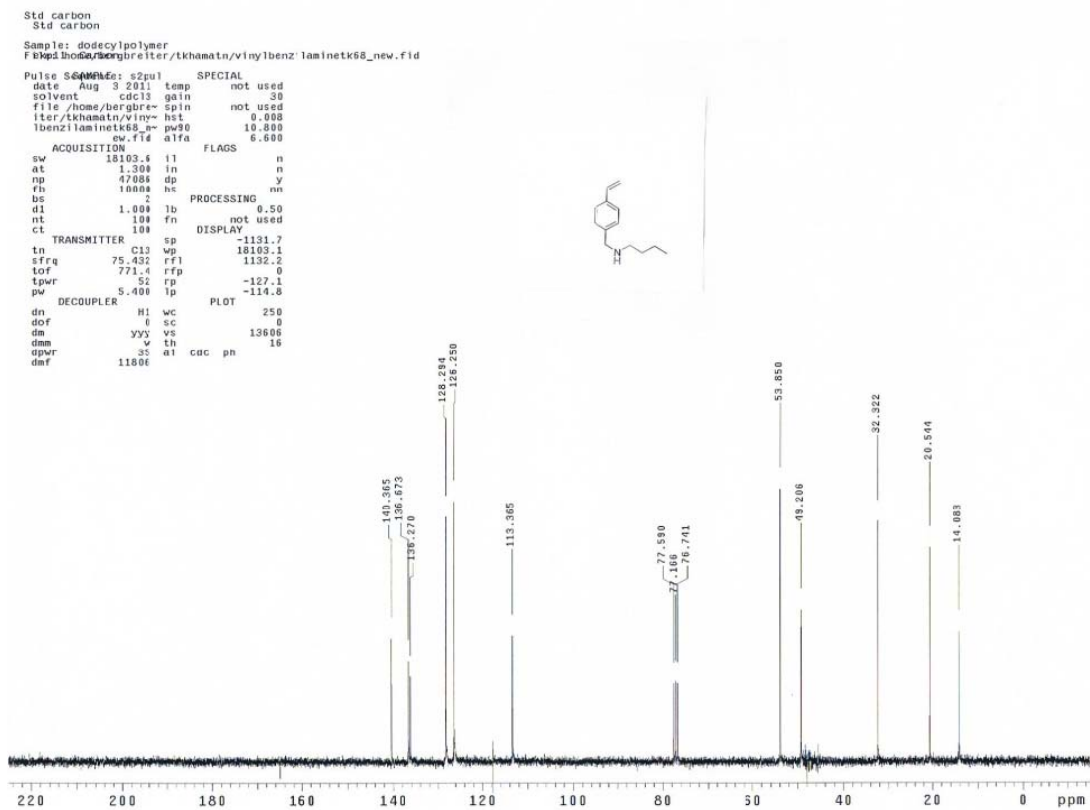
¹³C NMR of 1-(4-N-vinylbenzyl)-4-N-(4-pyridyl)piperazine 7.



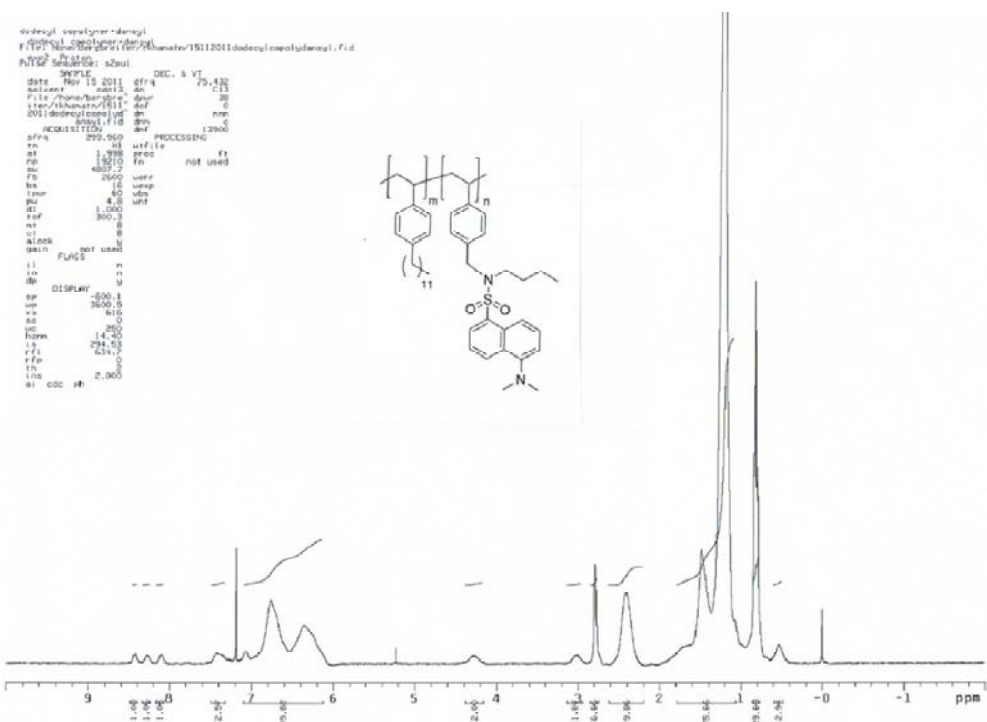
¹H NMR of 4-vinylbenzylbutylamine 8.



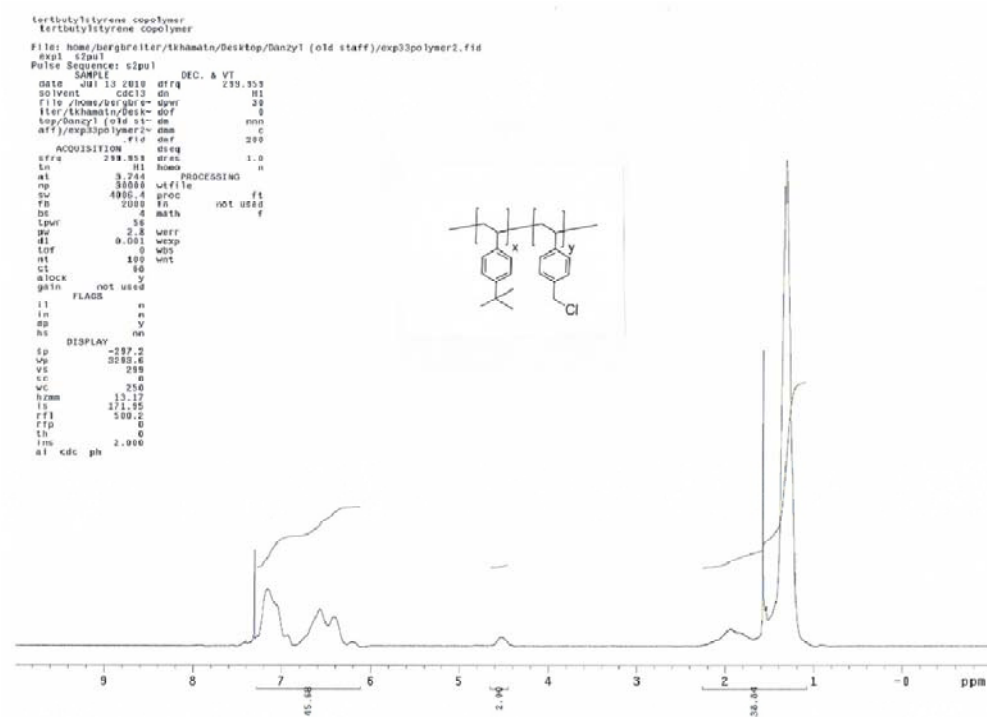
¹³C NMR of 4-vinylbenzylbutylamine 8.



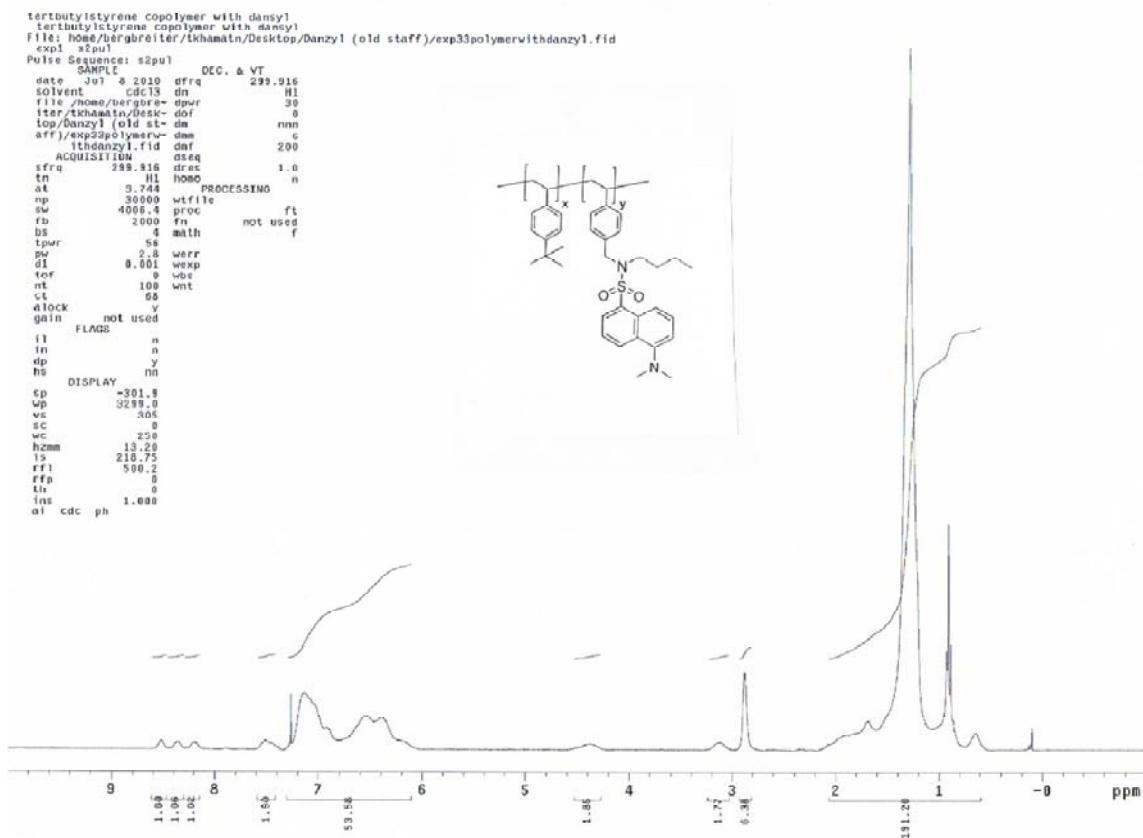
^1H NMR of Poly((4-dodecylstyrene)-*c*-(4-(*N*-dansylaminomethyl)styrene) 5.



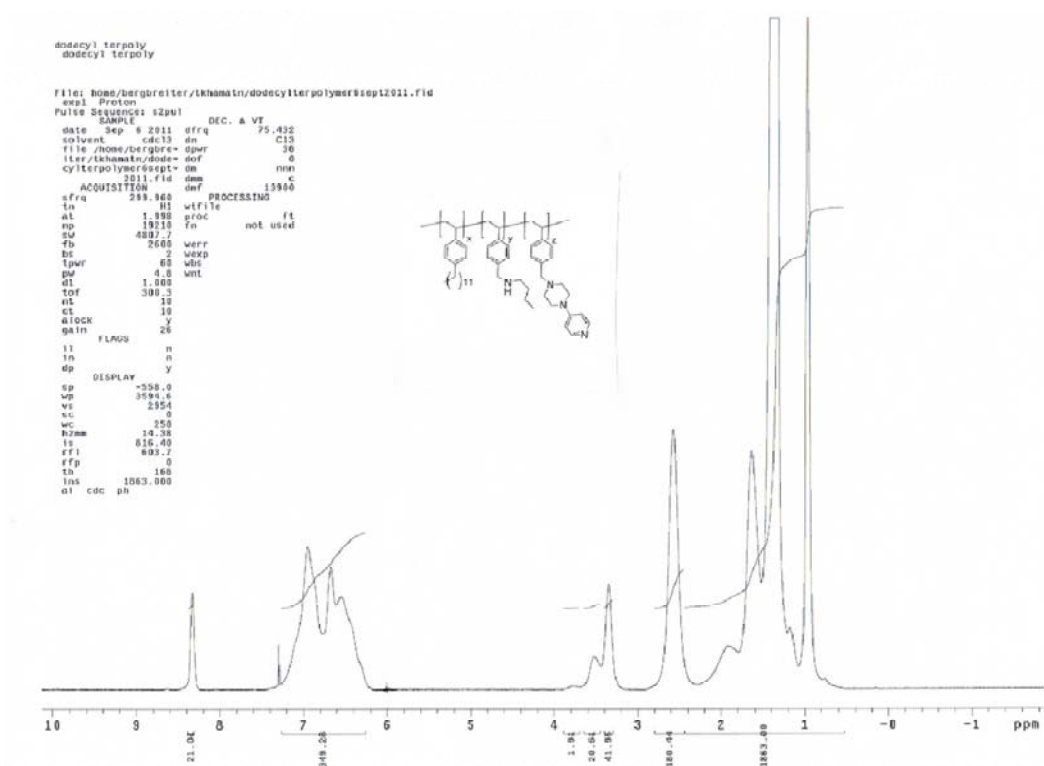
¹H NMR of poly((4-*tert*-butylstyrene)-*c*-(4-chloromethylstyrene)) 4.



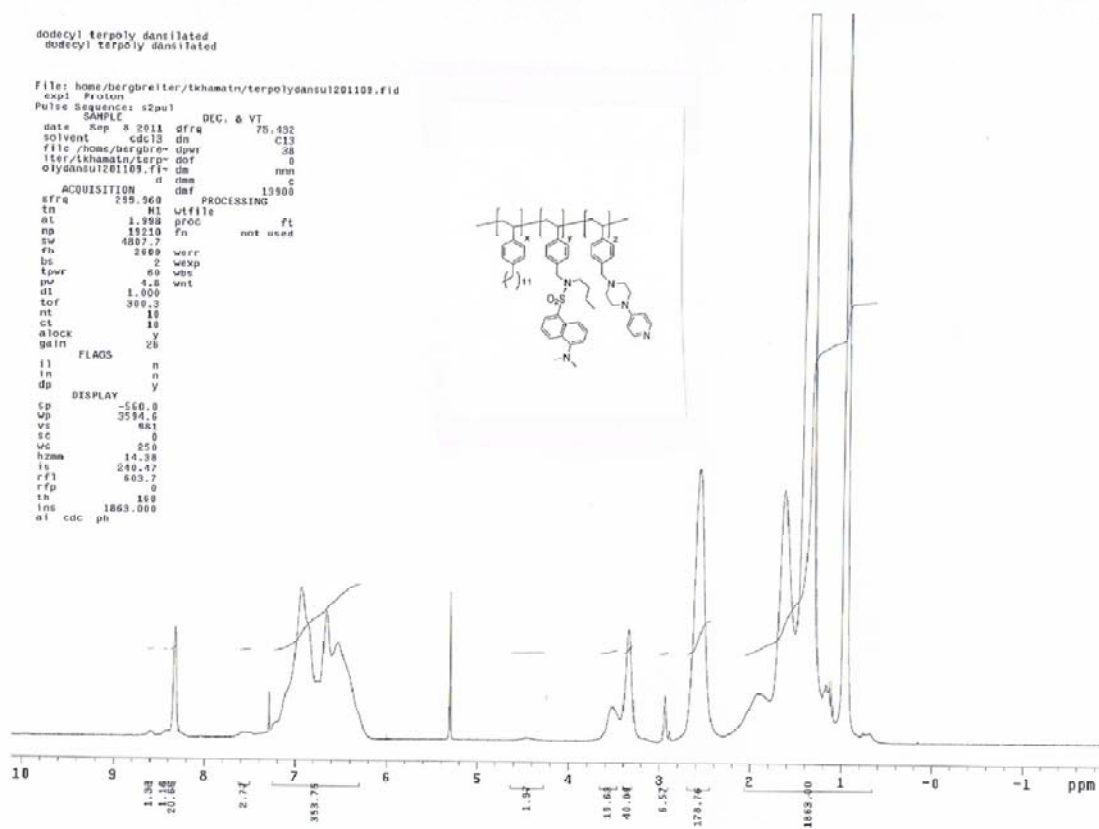
¹H NMR of poly((4-*tert*-butylstyrene)-*c*-(4-(*N*-dansylaminomethyl)styrene) 6.



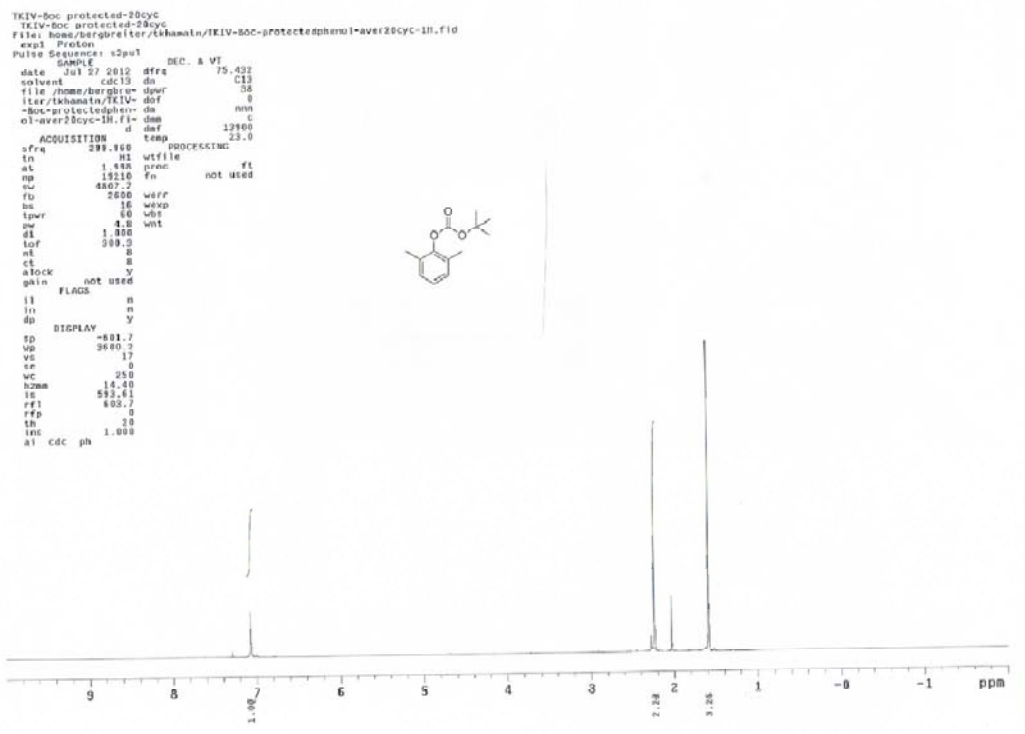
¹H NMR of terpolymer 9 containing dodecyl-, DMAP- and N-butyl-aminomethyl-substituted styrene groups



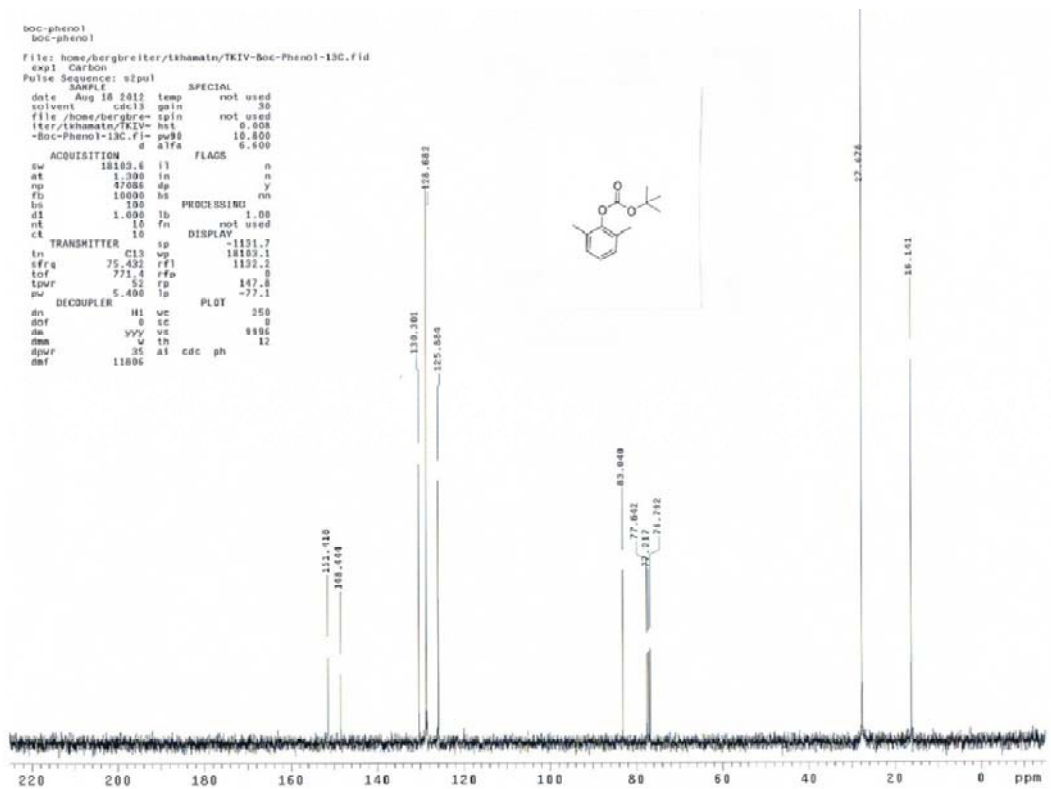
¹H NMR of Terpolymer 10 containing dodecyl-, DMAP- (0.44 mmol/g loading) and *N*-butyl-*N*-dansylaminomethyl- substituted styrene groups (0.07 mmol/g loading).



¹H NMR of Boc protected 2,6-dimethylphenol 11.

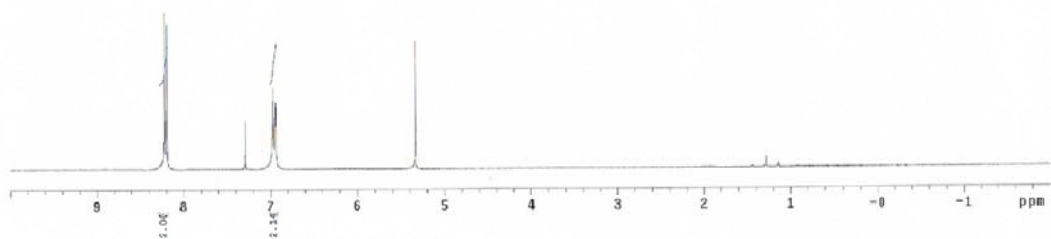


¹³C NMR of Boc protected 2,6-dimethylphenol 11.

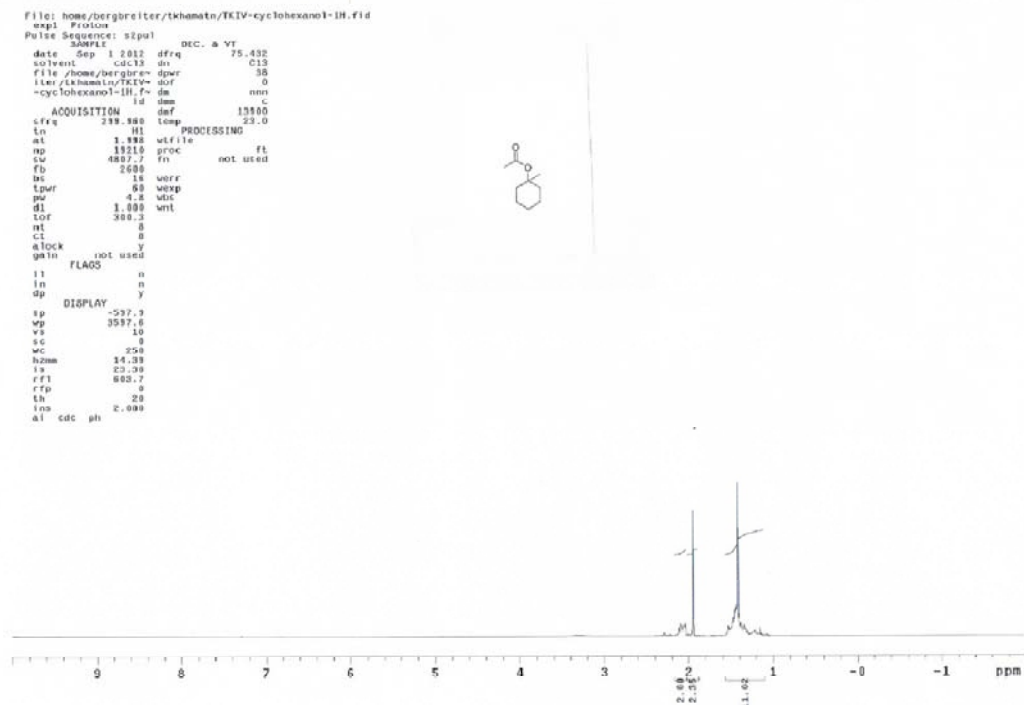


¹H NMR of 4-nitrophenol 12.

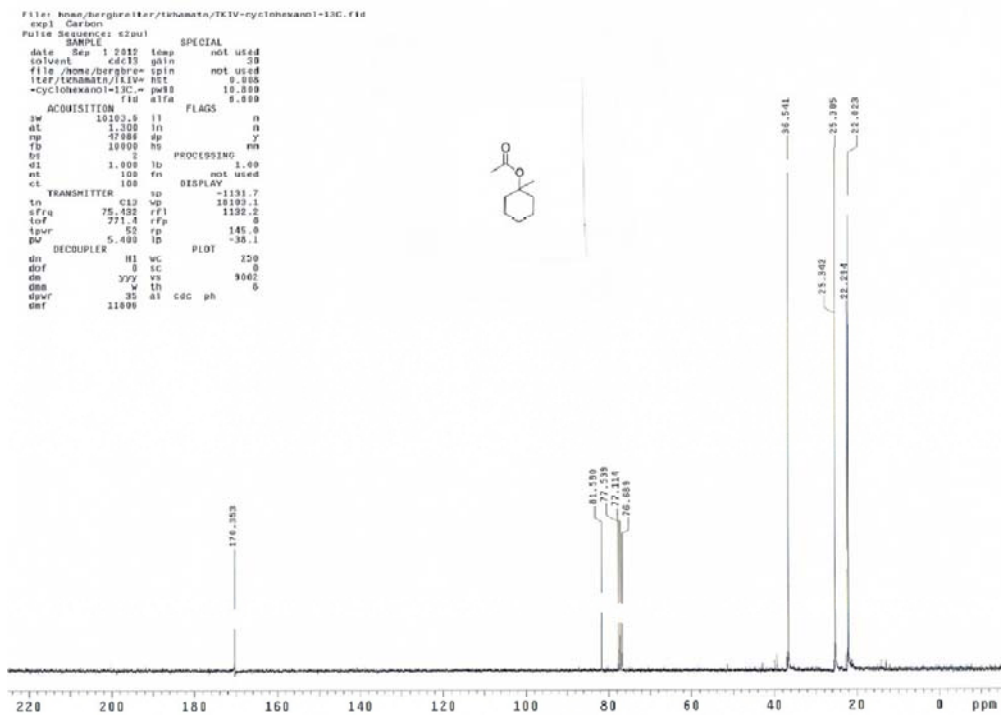
```
File: home/berghreit/...hamata/TKIV-nitrophenol-H.fid
===== Proton
Pulse Sequence: s2pul
=====
date Sep  4 2012  dfrs 75.432
solvent cdcl3  dn  C13
file /home/berghreit/...hamata/TKIV-nitrophenol-H.fid
iter/cxhamata/TKIV-...hamata/TKIV-nitrophenol-H.fid
-nitrophenol-H.fid  dn  non
acquisition  dn  C
=====
sfrq 239.960  def 13980
ln  M1  Temp 22.0
at 1.998  PROCESSING
np 19210  wfile
su 4897.7  proc
fb 2500  Fn  not used
bs 16
spwr 6.0  veff
pw 4.6  vexp
d1 1.500  vho
tor 300.3  wnt
nt 8
ct 6
elock not used
seln FLAGG
il n
in n
dp v
=====
DISPLAY
sp +597.9
vp 3597.0
vc 41
sc 0
uc 950
hzmm 14.38
ic 107.94
rfi 693.7
rfe a
lh 20
lvo 5.688
al cdc ph
```



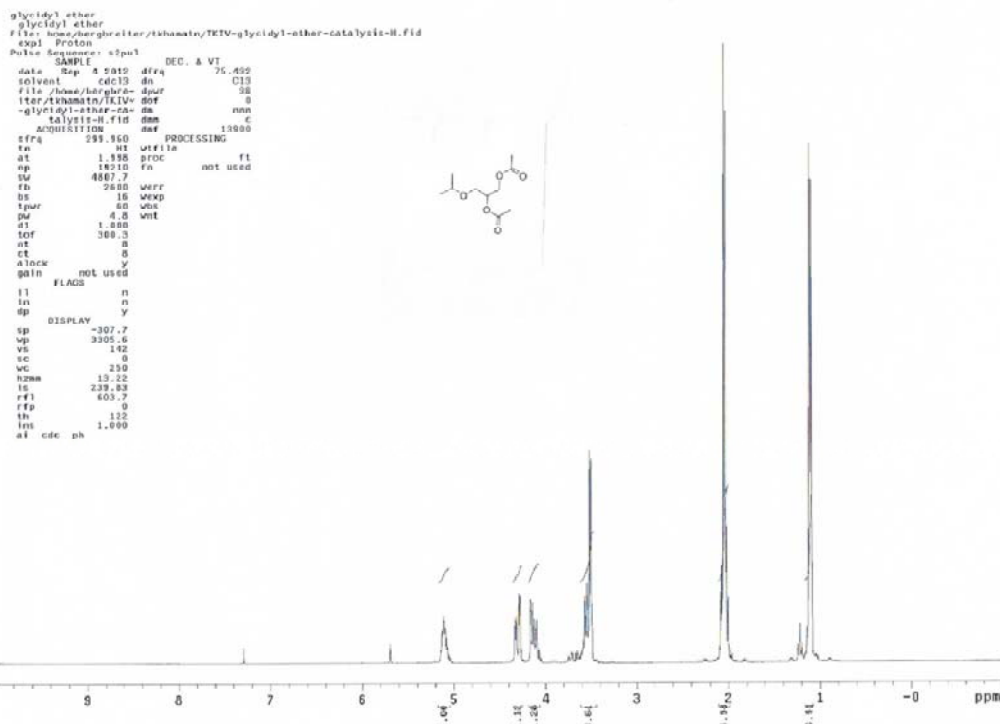
¹H NMR of acylated 1-methylcyclohexanol 13.



¹³C NMR of acylated 1-methylcyclohexanol 13.



¹H NMR of acylated glycidyl isopropyl ether 14.



¹³C NMR of acylated glycidyl isopropyl ether 14.

