Electronic Supplementary Material (ESI) for Polymer Chemistry.
This journal is © The Royal Society of Chemistry 2020

Decagram Scale Production of Deuterated Mineral Oil and Polydecene as Solvents for Polymer Studies in Neutron Scattering

Supplementary Information

Mitchell A. Klenner,[1] Marina Cagnes,[1] Kathleen Wood,[2] Kazuki Mita,[3*] Mizuki Kishimoto[3] and Tamim Darwish[1*]

[1] National Deuteration Facility (NDF), Australian Nuclear Science and Technology Organisation (ANSTO), Lucas Heights, Sydney, Australia.

[2] Australian Centre for Neutron Scattering (ACNS), Australian Nuclear Science and Technology Organisation (ANSTO), Lucas Heights, Sydney, Australia.

[3] Process Technology Laboratory, Mitsui Chemicals, Inc., Sodegaura, Chiba, Japan.

Contents

NMR & IR spectroscopy of protonated and deuterated mineral oil 2

NMR spectroscopy & stability assessment of poly(1-decene) 6

GPC of protonated and deuterated mineral oil and poly(1-decene) 10

Determination of the critical overlap concentration 11

NMR & IR spectroscopy of protonated and deuterated mineral oil

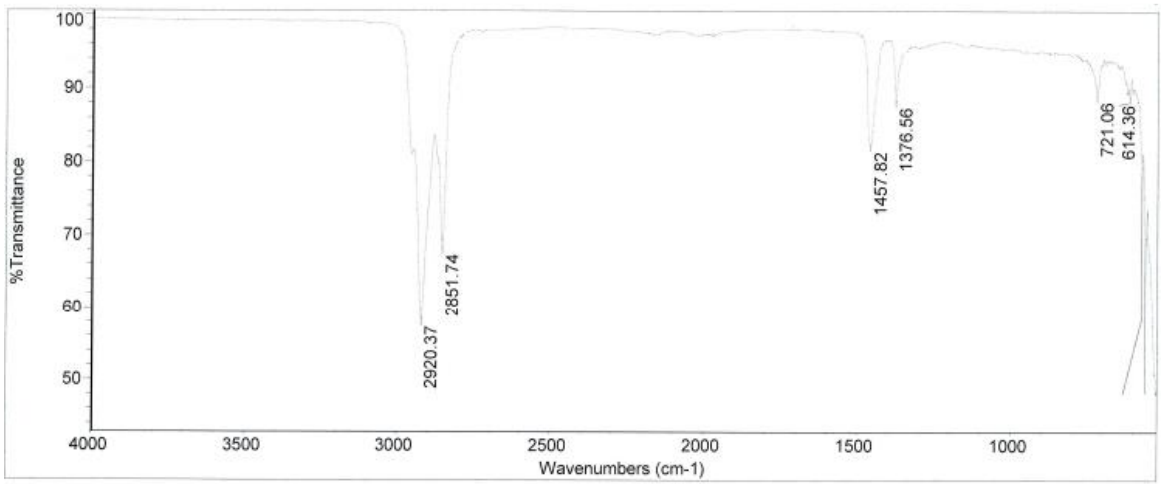

Figure S1. FTIR spectrum of protonated mineral oil.

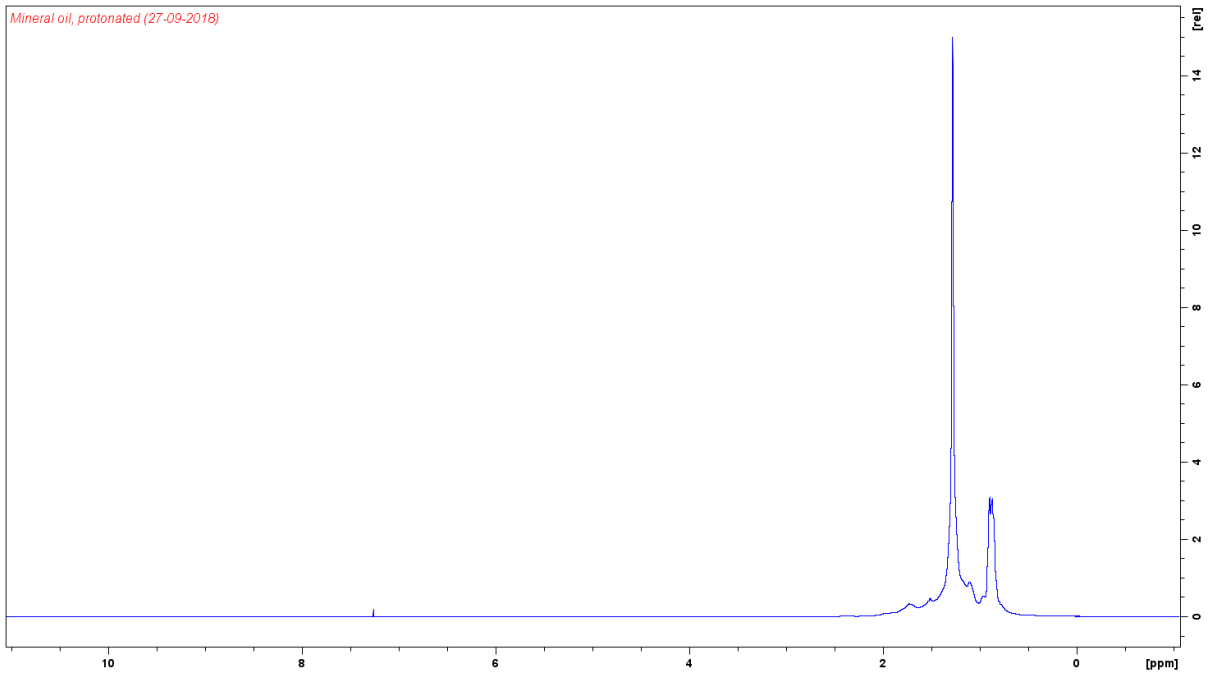

Figure S2. ^{1}H NMR (400 MHz, $CDCl_3$) of protonated mineral oil.

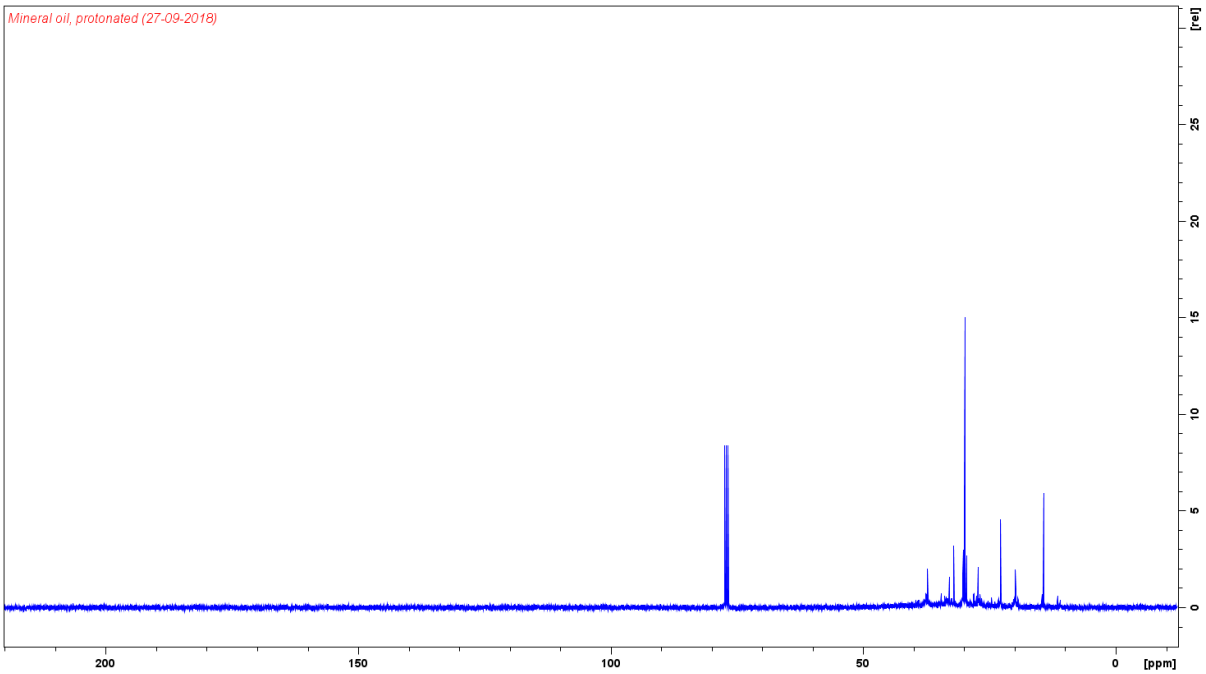

Figure S3. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of protonated mineral oil.

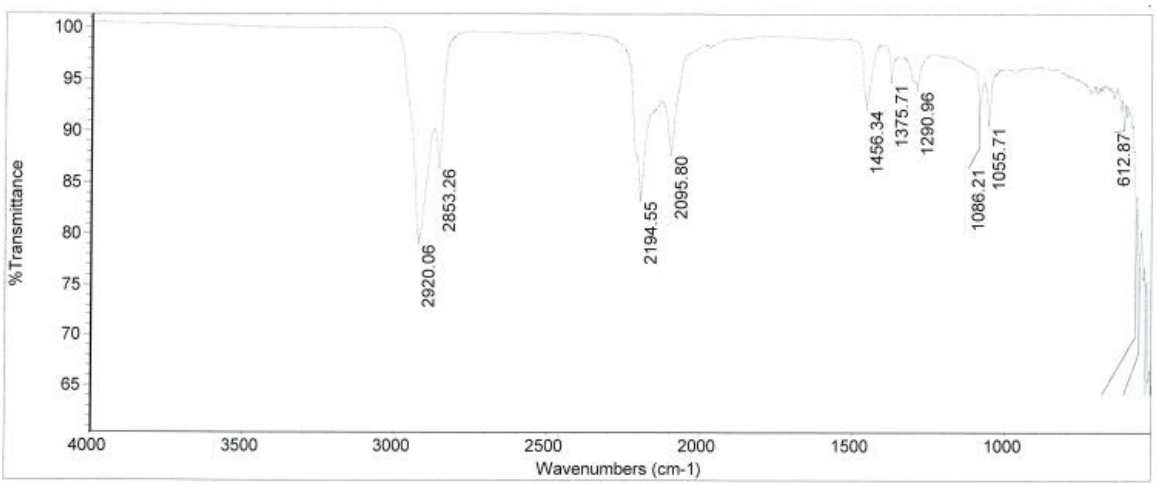

Figure S4. ^{1}H NMR (400 MHz, $CDCl_3$) of deuterated mineral oil.

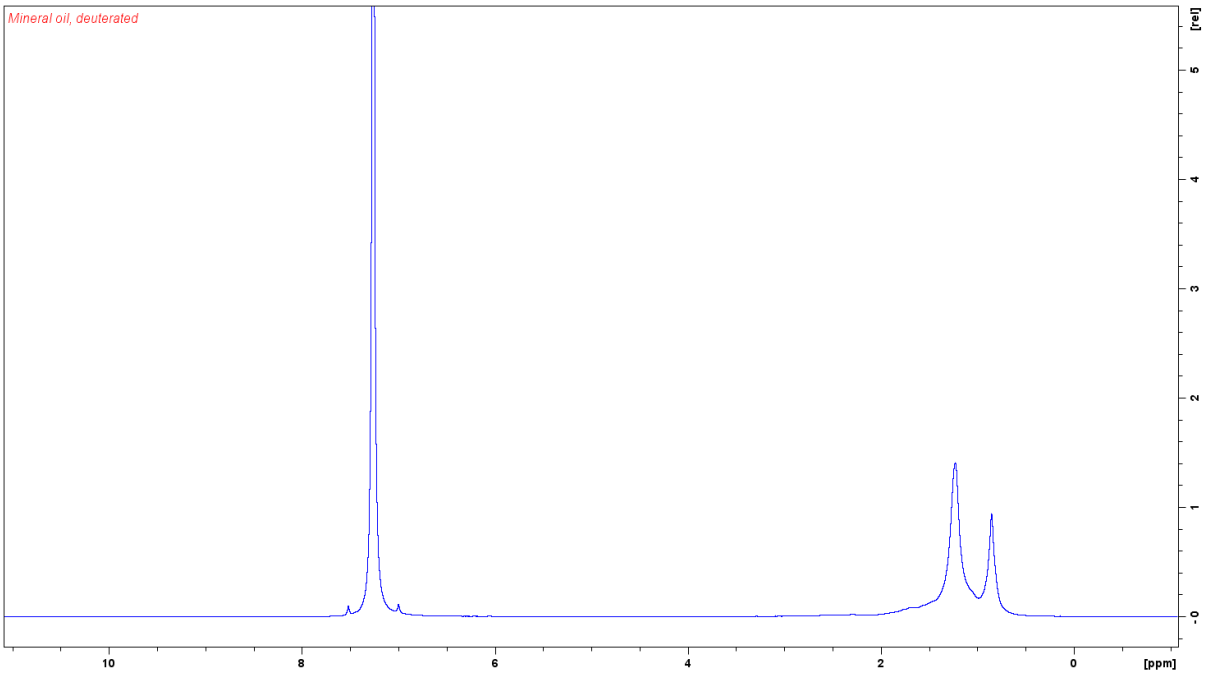

Figure S5. ^{2}H NMR (61.4 MHz, $CDCl_3$) of deuterated mineral oil.

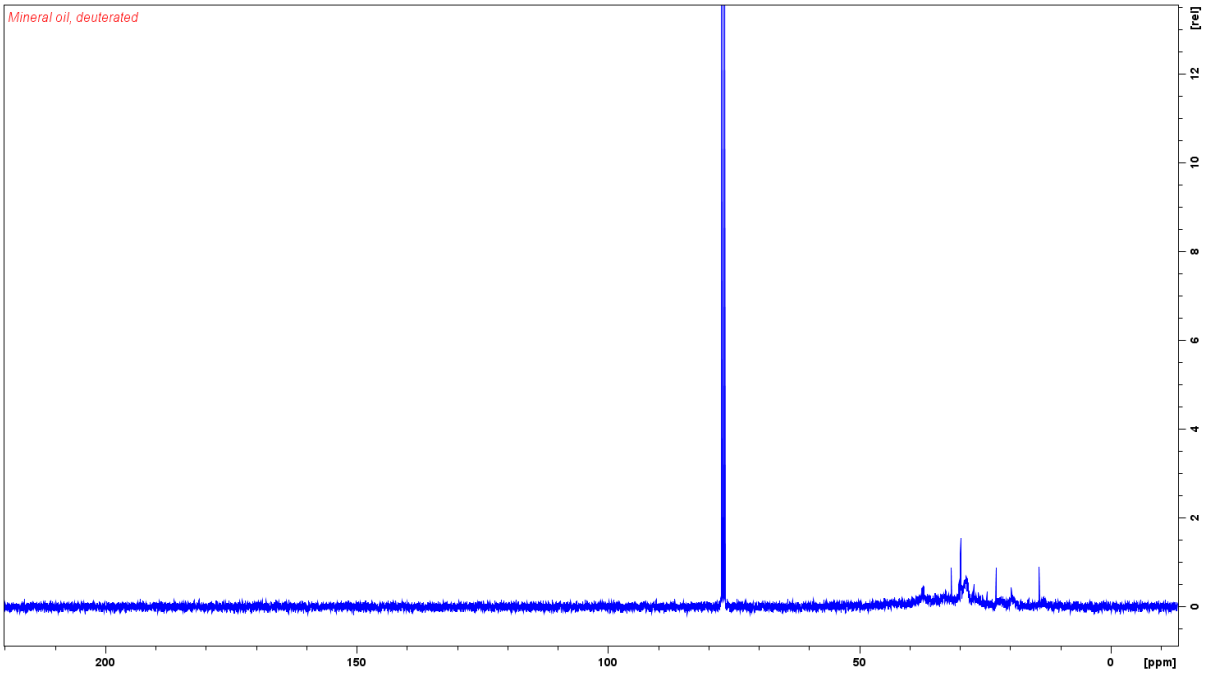

Figure S6. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of deuterated mineral oil.

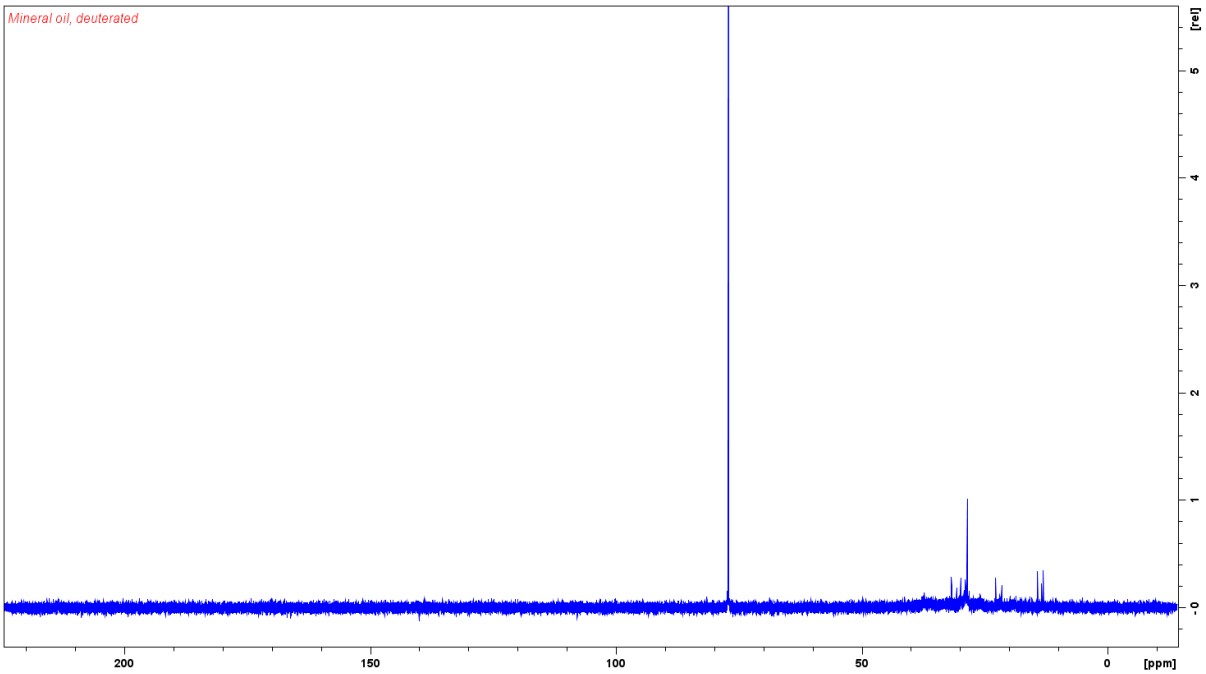

Figure S7. $^{13}C\{^{1}H, ^{2}H, d1 = 20 s\}$ NMR (101 MHz, $CDCl_3$) of deuterated mineral oil.

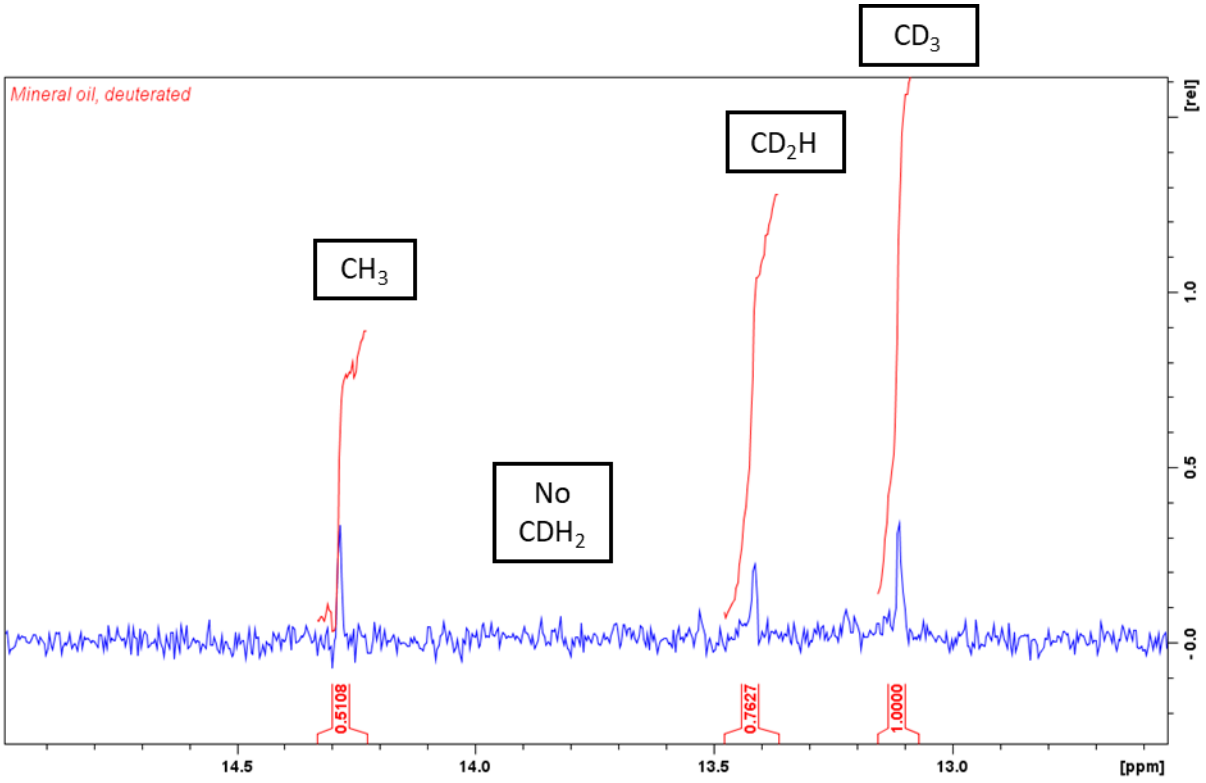

Figure S8. Sum of the integrals for the methyl carbon-13 resonance signals (CH_3, CDH_2, CD_2H and CD_3) of mineral oil. Sum of peak areas containing D / Sum of peaks areas containing D and H = (3 x 1.000 + 2 x 0.7627) / (3 x 1.000 + 3 x 0.7627 + 3 x 0.5108) = 66% D

NMR spectroscopy & stability assessment of 1-(polydecene)

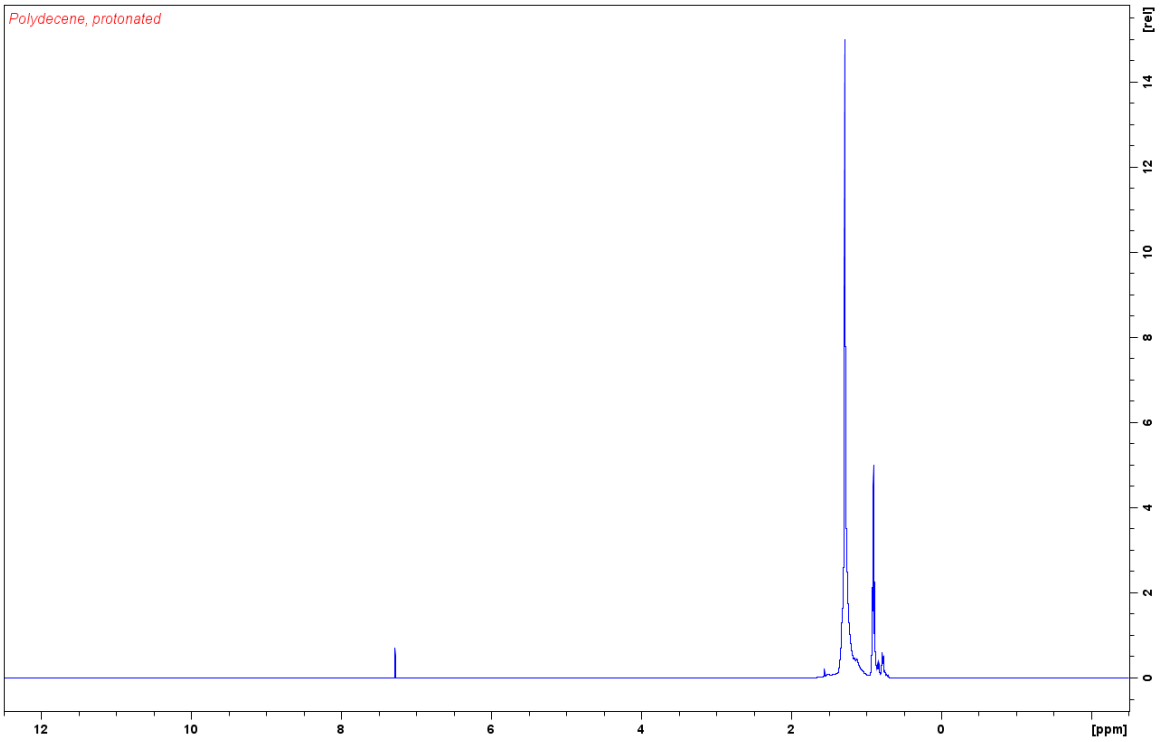

Figure S9. ^{1}H NMR (400 MHz, $CDCl_3$) of protonated poly(1-decene).

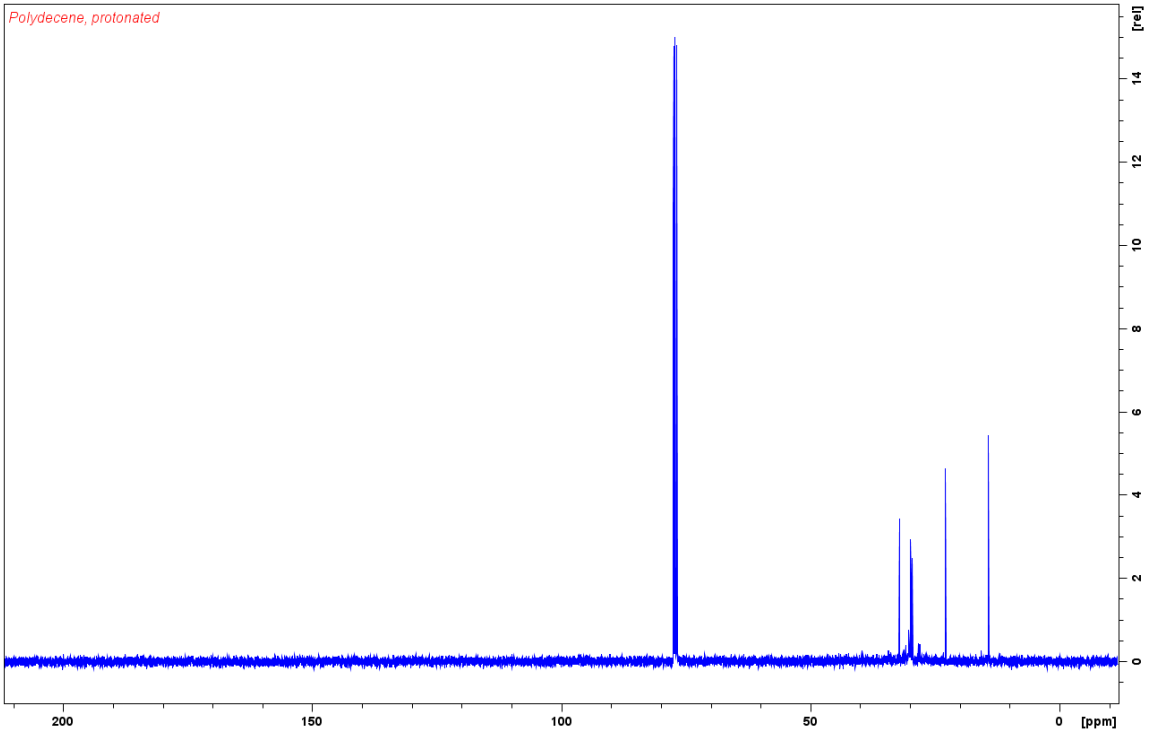

Figure S10. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of protonated poly(1-decene).

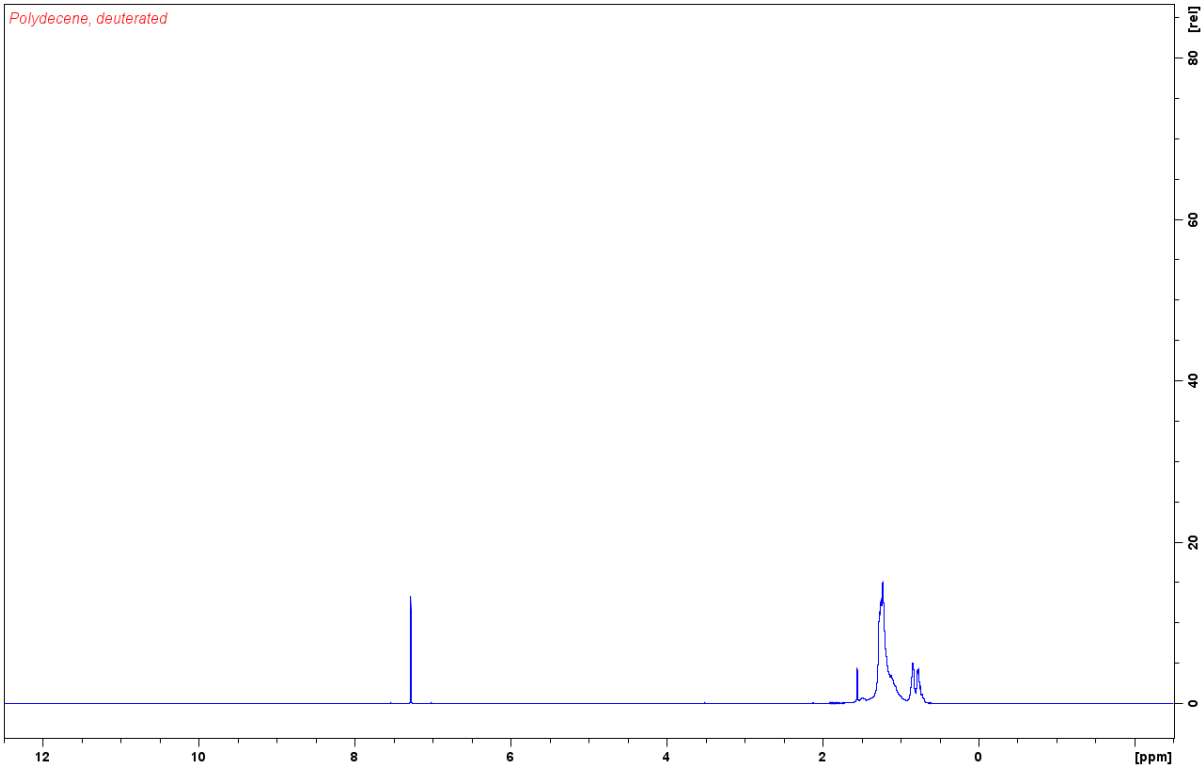

Figure S11. ^{1}H NMR (400 MHz, $CDCl_3$) of deuterated poly(1-decene).

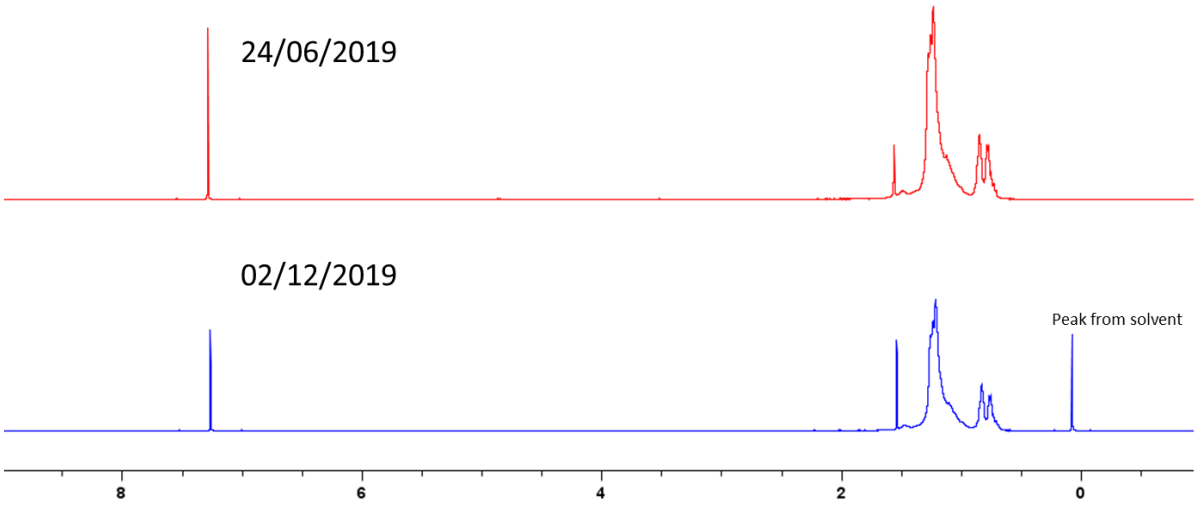

Figure S12. ^{1}H NMR (400 MHz, $CDCl_3$) of deuterated poly(1-decene), 5 month stability assessment.

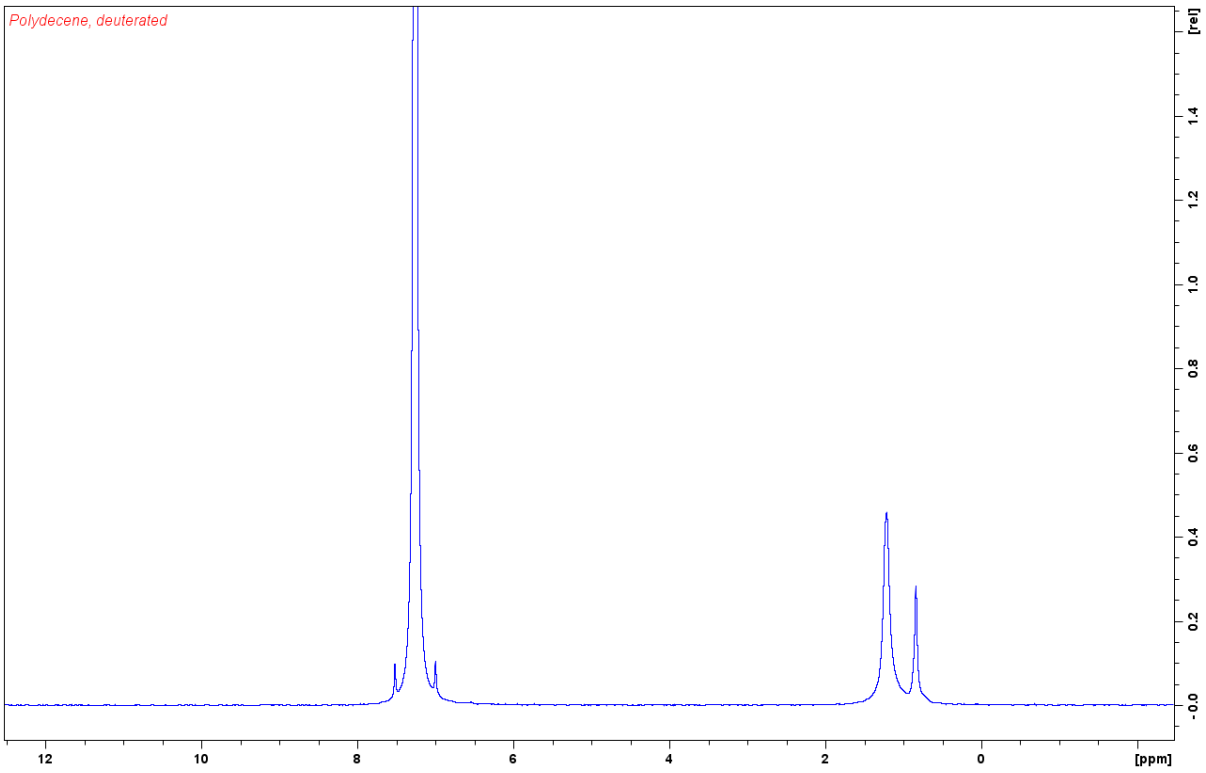

Figure S13. ^{2}H NMR (61.4 MHz, $CDCl_3$) of deuterated poly(1-decene).

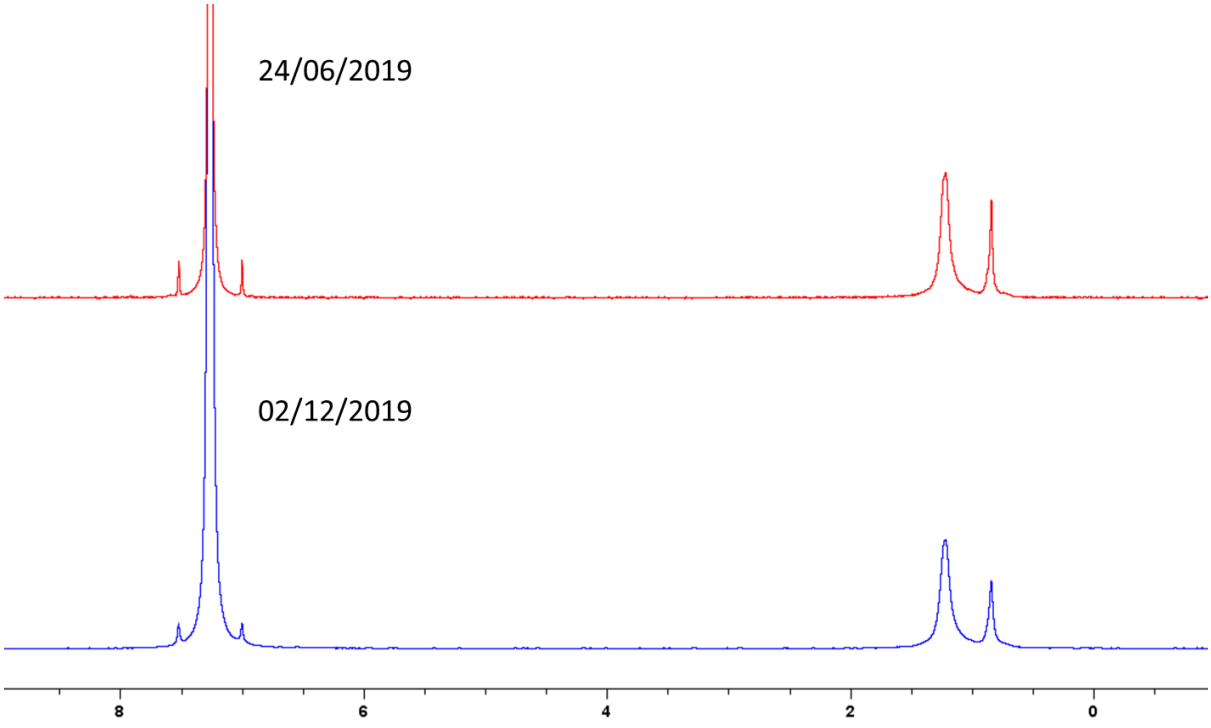

Figure S14. ^{2}H NMR (61.4 MHz, $CDCl_3$) of deuterated poly(1-decene), 5 month stability assessment.

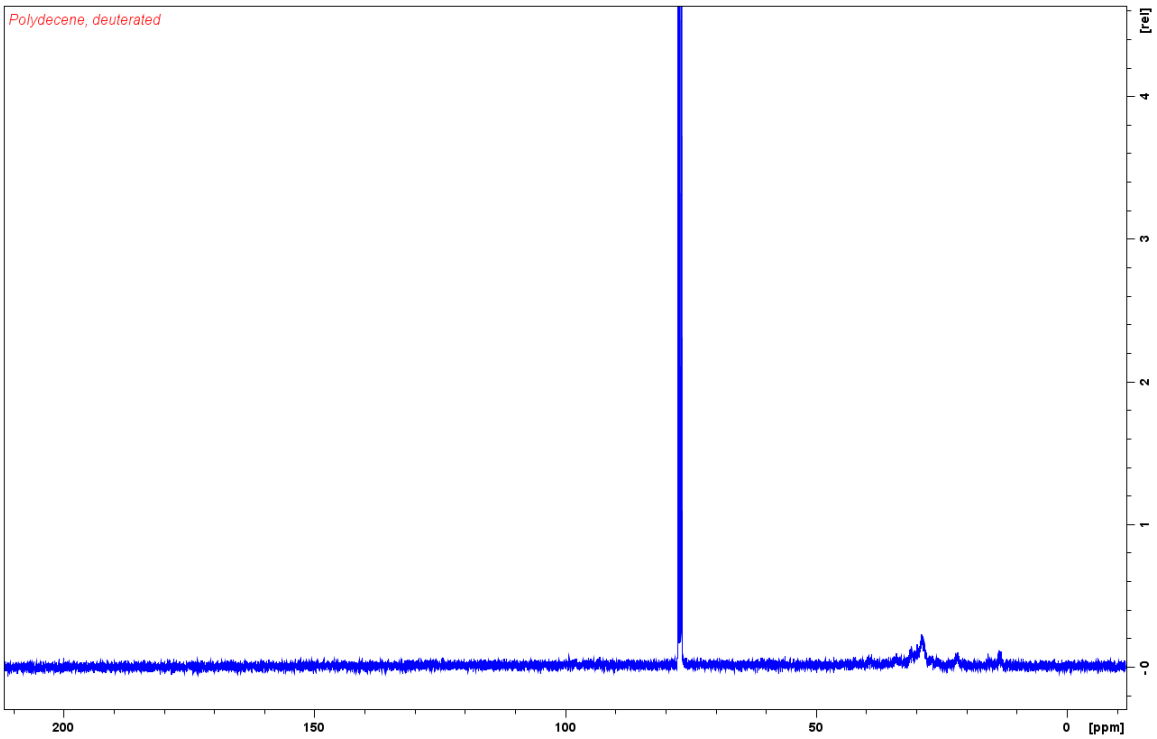

Figure S15. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of deuterated poly(1-decene).

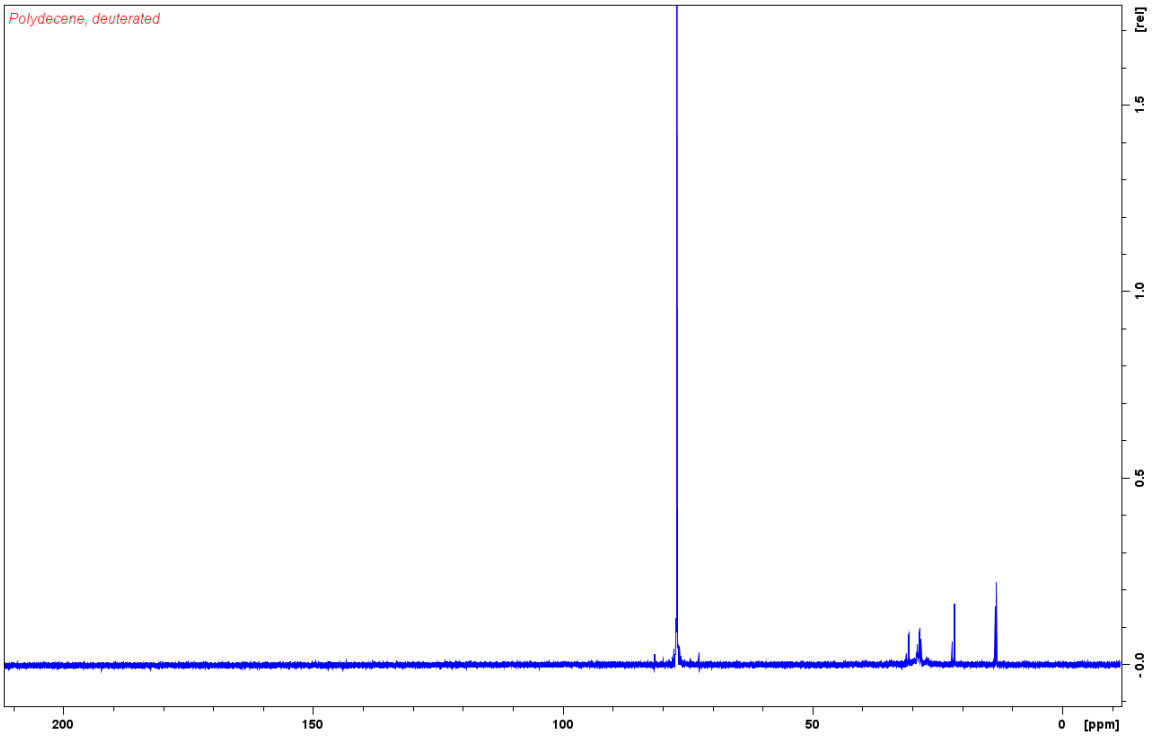

Figure S16. $^{13}C\{^{1}H, ^{2}H, d1 = 20 s\}$ NMR (101 MHz, $CDCl_3$) of deuterated poly(1-decene).

GPC of protonated and deuterated mineral oil and poly(1-decene)

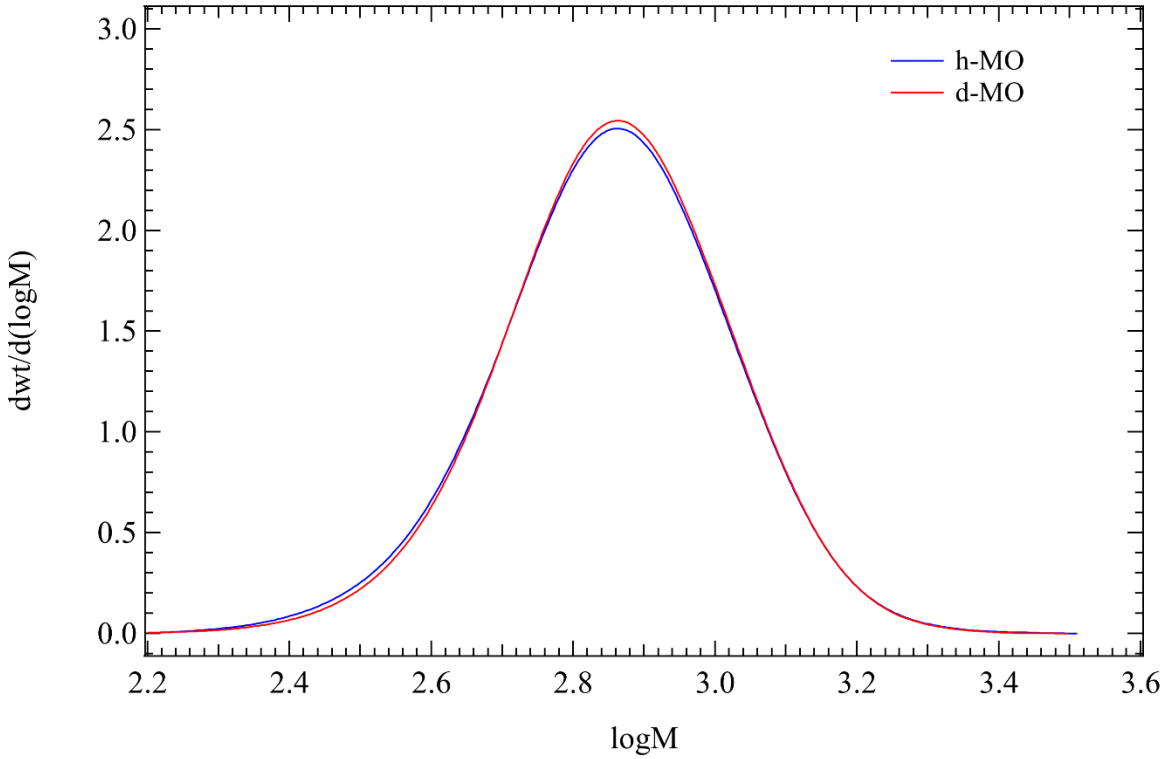

Figure S17. GPC curves of protonated and deuterated mineral oil.

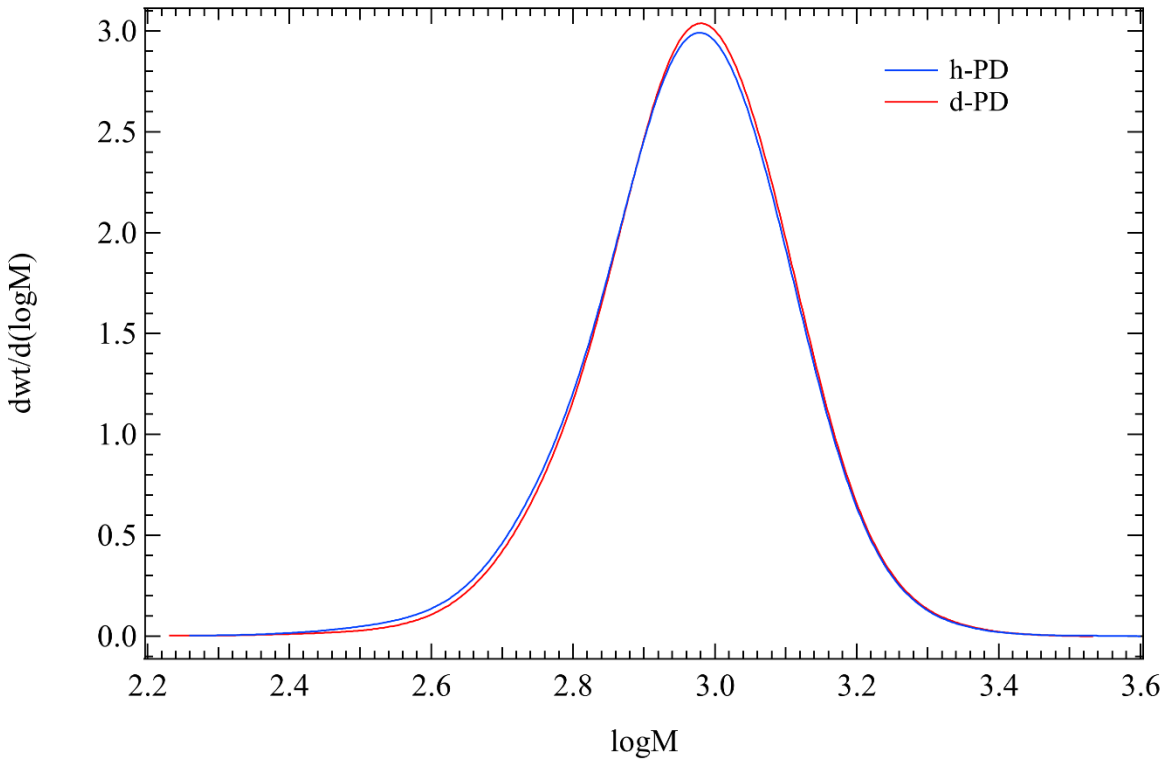

Figure S18. GPC curves of protonated and deuterated poly(1-decene).

Determination of the Critical Overlap Concentration

The critical overlap concentration (ϕ^*) was calculated was calculated using equation S1, whereupon M_p is the molecular weight of the polymer and N_A refers to Avogadro's number. The radius of gyration, R_g, was calculated using equation S2 whereupon b is the bond length, C_∞ is the characteristic ratio and n is the number of bonds along the polymer backbone. ϕ^* was converted to the critical overlap concentration in weight, C*, by adopting densities of the polymer (0.85 $g.cm^{-3}$) and the solvent (0.83 $g.cm^{-1}$).

$$\phi^* = \left(\frac{M_p}{N_A \rho_p}\right)\left(\frac{4}{3}\pi R_g^3\right)^{-1} \quad \text{(S1)}$$

$$R_g = \frac{b\sqrt{C_\infty n}}{\sqrt{6}} \quad \text{(S2)}$$