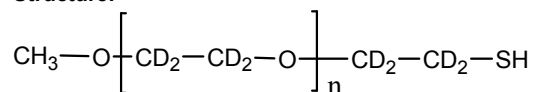


Sample Name:

Thiol Terminated Deuterated (d4) Poly(ethylene glycol)methyl ether

Sample #: **P5381A-dPEOCH3SH**

Structure:

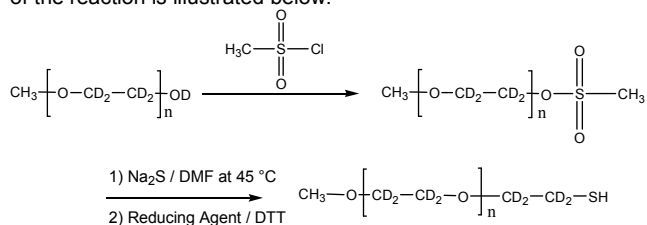


Composition:

Mn x 10 ³	PDI	SH functionality
2.0	1.10	>99%

Synthesis Procedure:

Thiol terminated deuterated Poly(ethylene glycol methyl ether) was prepared by mesylation of OH terminated deuterated PEG reacting it with NaSH.H₂O in polar solvent. The product was stabilized with DTT to avoid the formation of disulfide. The scheme of the reaction is illustrated below.



Characterization:

The molecular weight and polydispersity index of this polymer were determined by size exclusion chromatography (SEC) using a Varian liquid chromatograph equipped with a UV and refractive index detector. Polymer functionality was verified by oxidation of the thiol to disulfide.

Solubility:

Polymer is soluble in water, methanol and ethanol, THF, CHCl₃. It is precipitated out from cold hexane and ether.

Purification of the obtained polymer:

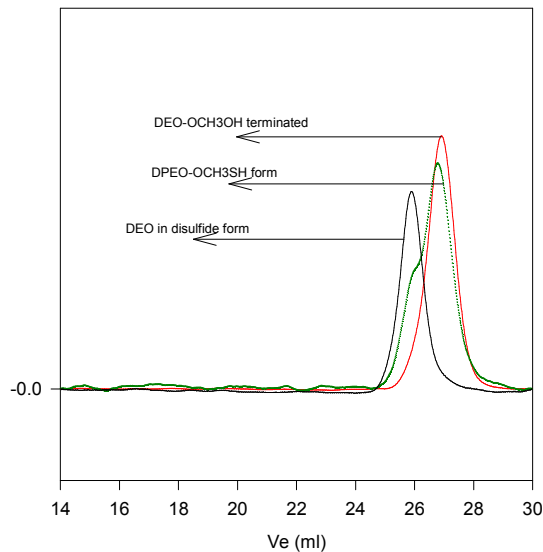
Obtained polymer after the reaction show the formation of disulfide. The Disulfide formation was close to 100%. It was reduced by tributylphosphine under well purified solvent and argon atmosphere in the presence of catalytic amount of DTT.

Purification of the obtained polymer was carried out rigorously as follows to ensure the removal of the any side product:

1. Dissolved the polymer in de-ionized distilled water to remove the any insoluble organic catalyst side product.
2. Polymer extracted from water with dichloromethane.
3. Polymer solution in dichloromethane was dried over anhydrous sodium sulfate.
4. Solution filtered and then passed through a column packed with basic Al₂O₃.
5. Solution concentrated on rota-evaporator
6. Solution precipitated in cold diethyl ether.
7. Dried under vacuum for 12h at 30°C.

SEC of Sample:

P5381A-dPEOCH3SH



Size exclusion chromatography of α -methoxy- ω -thiol poly(ethylene glycol):

---M_n=2000, M_w=2200, PI=1.10 (Methoxy mesylate form)

_____ After SH -formation (presence of a minor fraction disulfide)

This may be due to the formation of disulfide presence of Oxygen in THF
After oxidation with iodine - showing quantitative functionality