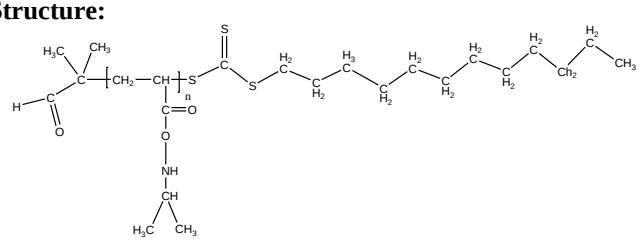
Sample Name: **COOH end terminated Poly(N-isopropyl acrylamide) of Narrow molecular weights distribution**
Sample #: **P14506-NIPAMCOOH**

Structure:



Composition:

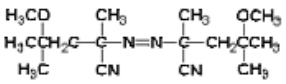
Mn x 10^3	PDI
25.0	1.4

Synthesis Procedure:

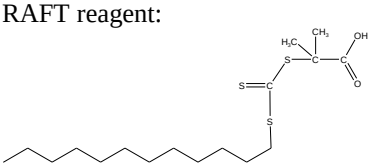
COOH end functionalized poly(N-isopropyl acrylamide) is obtained by RAFT polymerization process.

Following catalyst was used:

2,2'-Azobis(4-methoxy-2,4-dimethylvaleronitrile)



RAFT reagent:



2-dodecylsulfonyl-2-methylpropionic acid

Characterization: The molecular weight and polydispersity index (PDI) were obtained by size exclusion chromatography (SEC) in THF and in DMF. SEC analysis was performed on a Varian liquid chromatograph equipped with refractive and UV light scattering detectors. Two SEC columns from Supelco (G6000-4000 HXL) were used with triple detectors from Viscotek Co.

Sample Preparation: Polymer sample for the GPC were prepared as reported in the literature (**Macromolecules, 2000,33,6738).** To avoid the effect of concentration and the amount of water present in the sample, on line triple detectors were used.

Viscosity measurement was carried out in a Ubbelohde viscometer at 25°C. Four solutions in methanol of different concentrations were measured. The intrinsic viscosity was obtained by extrapolation to c=0. From viscosity-molecular weight relationship $[\eta]= 2.99\times10^{-2}\ M^{0.64}$ (Makromolecular Chem. V180, P969, 1979), the viscosity average molecular weight was calculated accordingly.

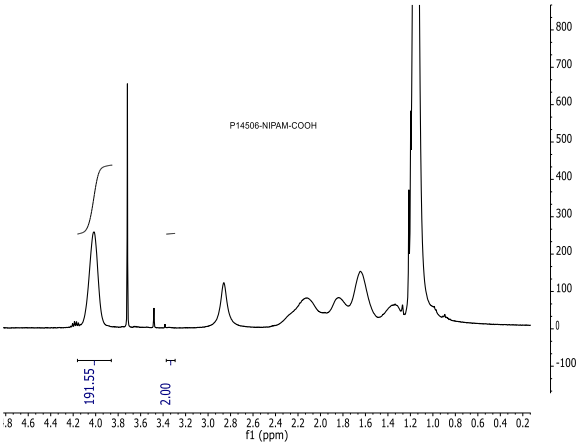
DMF containing LiBr as an additive was found efficient to avoid any adsorption effect of polymer. It was observed 0.05 to 0.1 M solution of LiBr in DMF was efficient to analyze polymer. In pure DMF without any additive the molecular weight was found higher and Mw/Mn was broad.

Purification of polymer:

Unreacted monomer was removed by dissolving the product in cold water than warming the solution. The polymer separated out. This procedure was applied 2 times to remove the unreacted monomer. The obtained polymer was dissolved in acetone and fractionated from Hexane. After several fractionation to remove the low molecular weight fraction < 10% and to obtain fairly narrow molecular weight fraction polymer. Polymer was finaly reprecipitated in cold hexane and dried at room temperature.

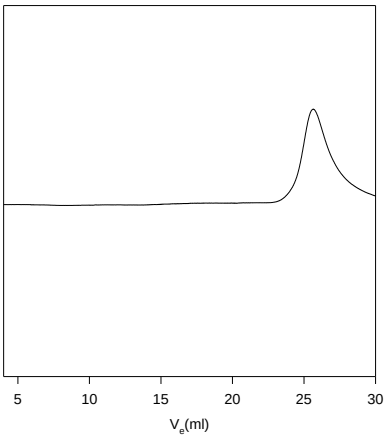
The molecular weights of end functionalized PNIPAM polymer eluted in DMF in presence of LiBr, the comparison with Polystyrene or poly ethyelene glycol as referece material are not effective for molecular weights lower then 25,000. The following is the table illustrate such results: Molecular weight for such End functionalized PolyNIPAM COOH can be determined more accurately by acid base titration in $CHCl_3$. The Mw/Mn was determined from SEC using PEG as reference material.

Reference Polymer	Mn of PNIPAMCOOH
Polystyrene	68,000
Poly(ethylene glycol)	30,600
By tiration	**25,000**



SEC of the Polymer carried out in DMF

P14506-NIPAMCOOH



Size exclusion chromatography of N-Isopropyl Acrylamide in DMF/LiBr(0.05M)
Molecular Weight Distribution with respect to Poly ethylene glycol Standards:

Mn: 25,000 Mw: 35,100 M_w/M_n= 1.4