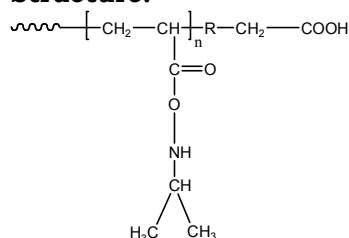


Sample Name: **Carboxy Terminated Poly(N-isopropyl acrylamide)**

Sample #: **P6849F3-NIPAMCOOH**

Structure:

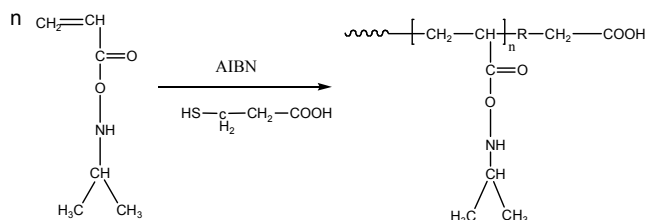


Composition:

| | |
|----------------------|------|
| Mn x 10 ³ | PDI |
| 6.3 | 1.62 |
| T _g (°C) | |

Synthesis Procedure:

Carboxy Terminated Poly(N-isopropyl acrylamide) was prepared by free-radical polymerization of N-isopropyl acrylamide with a carboxyl containing chain-transfer agent. The final polymer was purified by fractionation and the scheme of the reaction is illustrated below:



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 reprecipitated in cold ether.

Characterization:

Size exclusion chromatography (SEC) was carried out on a Varian liquid chromatograph equipped with a refractive index detector. A Shodex 806L GPC columns from Supelco was used with DMF(0.01M LiBr) as the eluent and also in THF following the procedure as out lined in **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 and the dn/dc was calculated and found : 0.104mL/g in THF at 35 oC The columns were calibrated with monodisperse polystyrene standards. The polydispersity index was calculated.

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 $[\eta] = 2.99 \times 10^{-2} M^{0.64}$ (Makromolecular Chem. V180, P969, 1979), the viscosity average molecular weight was calculated accordingly.

It is important that the values of molecular weights determined in DMF and in THF were found quite different (data are with respect to polystyrene as reference material) : see Table 1.

| In DMF Mn (Mw/Mn) | In THF Mn (Mw/Mn) | Molecular weight by titration | Mv by Viscosity |
|-------------------------|-------------------------|-------------------------------------|--------------------|
| 65,000(2.8) | 1500 (3.2) | 12300 | 14000 |

It is therefore, the titration values were found comparable with the values obtained by viscosity.

Molecular weight of the product is reported by end group titration by acid base process.

Solubility:

The polymer is soluble in methanol, cold water, THF, CHCl₃.

