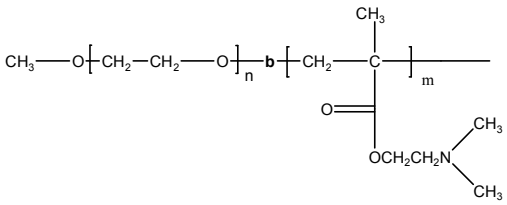
Sample Name:

Poly(ethylene oxide-b-N,N-dimethylaminoethylmethacrylate))

Sample #: **P4860- EODMAEMA**

Structure:



Composition:

Mn x 10^3 PEO-b-PDMAEMA	PDI
5.0-2.5	1.20

Synthesis Procedure:

Poly(ethylene oxide –b- 2-(dimethylamino)ethyl methacrylate) is prepared by living anionic polymerization of sequential addition of ethylene oxide and 2-(dimethylamino)ethyl methacrylate.

Characterization:

An aliquot of the first anionic block was analyzed by size exclusion chromatography (SEC) to obtain the molecular weight and polydispersity index (PDI) before addition of the second block. The final block copolymer composition and molecular weight are calculated from ^{1}H-NMR spectroscopy by comparing the peak area of the ethylene oxide protons at about 3.6 ppm with the 2-(dimethylamino)ethyl methacrylate protons at about 0.8-5 ppm that deducts the contribution of poly(ethylene oxide). For the diblock polymer with a shorter block of PDMEMA can be eluted from THF as eluent. However with longer block of PDMEMA block can not be eluted from THF as eluent. This may be due to strong interaction between the polymer and the column packing material.

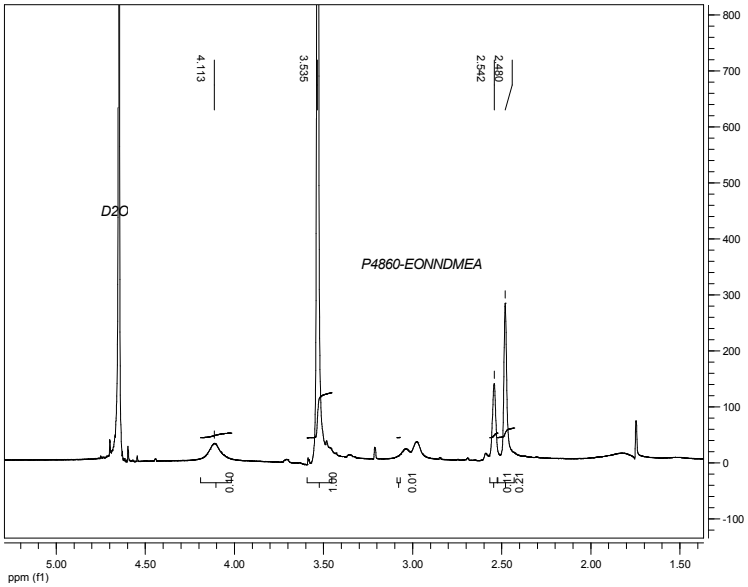
Purification of the polymer and removal of any un-reacted homopolyethylene oxide from the diblock copolymer:

Polymer dissolved in water and the pH of the medium increased to about 13 by adding NaOH. Now the solution warmed to 80 oC and the polymer precipitated out. This procedure was repeated 2 time to removed any homo PEO. Now the polymer dissolved in methanol and pH was adjusted to about 8 by adding HCL The polymer solution was filtered and the solvent was removed by rota-evaporator. The highly viscous solution was precipitated in cold hexane/ether mixture. The polymer was dried under vacuum at 40^{o}C.

Solubility:

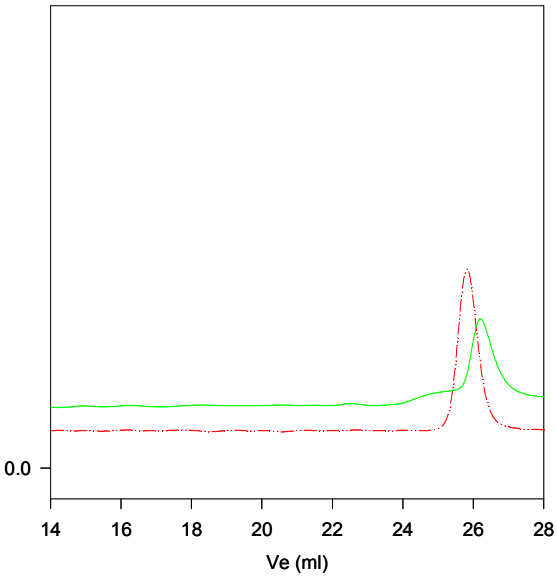
The polymer is soluble in water and precipitate out from hexane, ether.

^{1}H-NMR Spectrum of the block copolymer:



SEC of the Polymer:

P4860-EODMAEMA



Size exclusion chromatography of
poly ethylene oxide-b-N'N'-dimethylethylmethacrylate):

hydroxy terminated Polyethylene oxide M_n=5000, PI=1.05

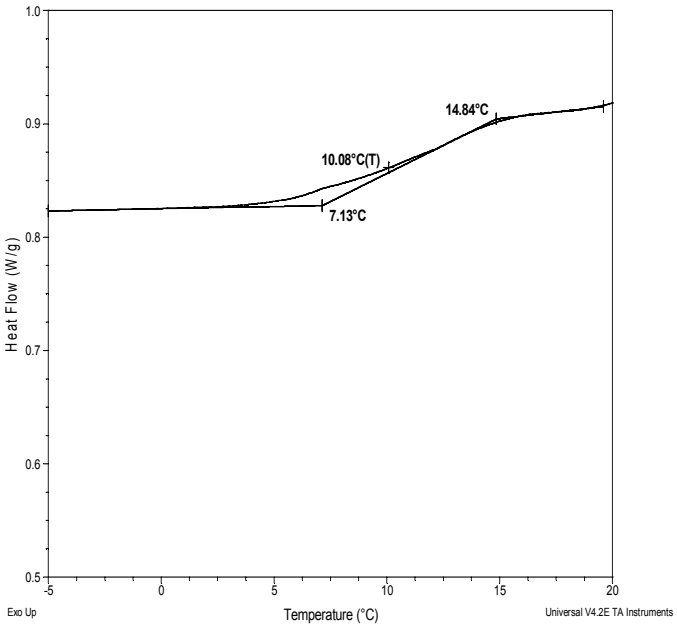
Block Copolymer PEO-NNDMAEMA
Mn:5000-b-2600, PI=1.2
The elution count for the diblock copolymer retarded, it may be due to interaction with the packing material of the SEc columns.
(Composition from H NMR)

Thermal analysis of # P4860-EODMAEMA

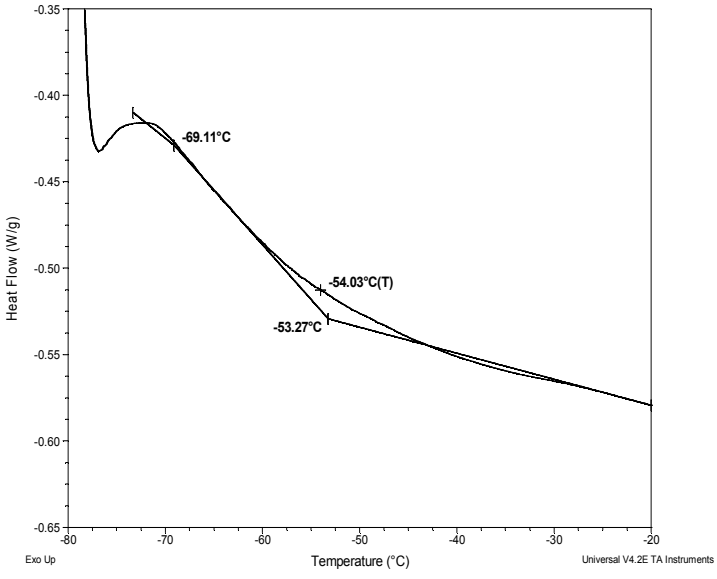
Thermal analysis of the samples was carried out on a TA Q100 differential scanning calorimeter at a heating rate of 15°C/min.The midpoint of the slope change of the heat flow plot of the second **heating scan** was considered as the glass transition temperature (T_g).

Thermograms for the sample

For DMAEMA block



For PEO block



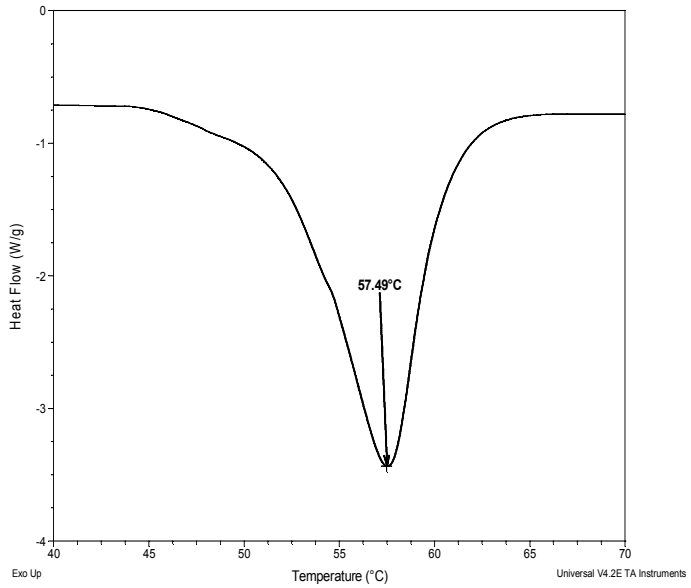
Thermal analysis results at a glance

For DMAEMA block		
T_g: 10°C	T_m: -	T_c: -
For PEO block		
T_g: -54°C	T_m: 57°C	T_c: 40°C

Melting and crystallization curve for the sample

The melting temperature (T_m) was taken as the maximum of the endothermic peak where as the crystallization temperature (T_c) was considered as the minimum of the exothermic peak. The T_c was calculated during **cooling ramp**.

Melting curve for PEO block



Crystallization curve for PEO block