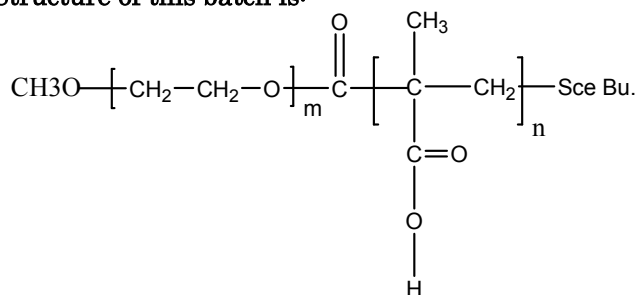


Sample Name: Poly(ethylene oxide -b- methacrylic acid)

Sample #: P18037-EOMAA

Structure of this batch is:

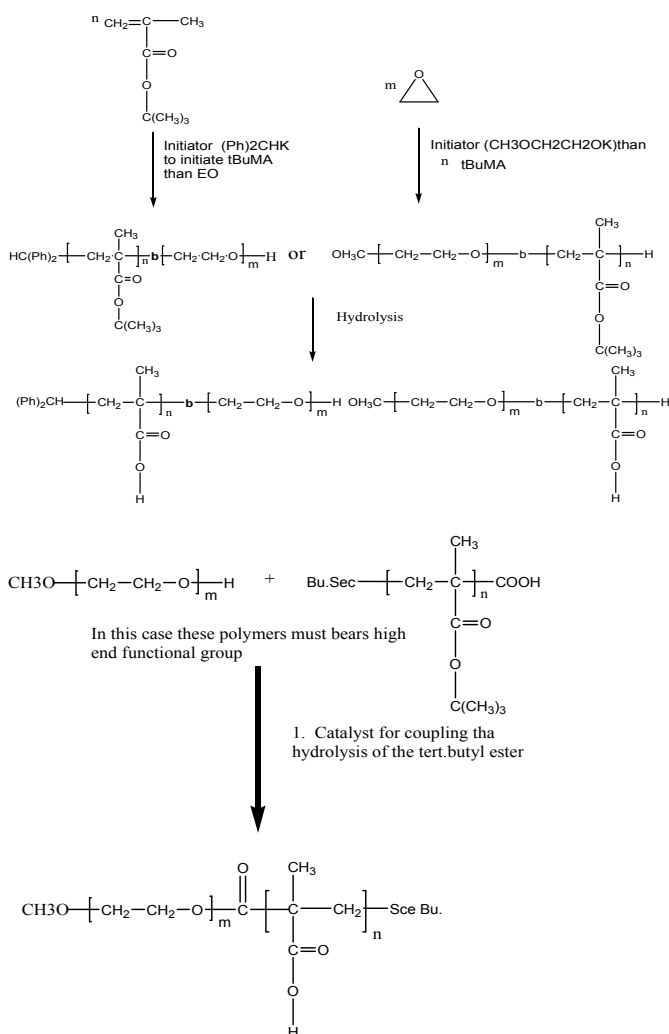


Composition:

Mn x 10 ³ PEO-b-PMAA	PDI
5.0-b-1.2	1.08

Synthesis Procedure:

Poly(ethylene oxide -b- methylacrylic acid) is prepared by 2 different routes: A. By living anionic polymerization of sequential addition of EO and tBuMA (ethylene oxide or t-butyl methacrylate) followed by hydrolysis of the t-butyl group¹ or B. by chemical coupling reaction of the corresponding functionalized polymer. The scheme of the reaction is illustrated below:



Characterization:

An aliquot of the first anionic block was terminated before addition of the second block and analyzed by size exclusion chromatography (SEC) to obtain the molecular weight and polydispersity index (PDI). The final block copolymer composition was calculated from ¹H-NMR spectroscopy by comparing the peak area of the ethylene oxide protons at about 3.6 ppm with the tert.butyl protons at about 1.4 ppm.

Hydrolysis:

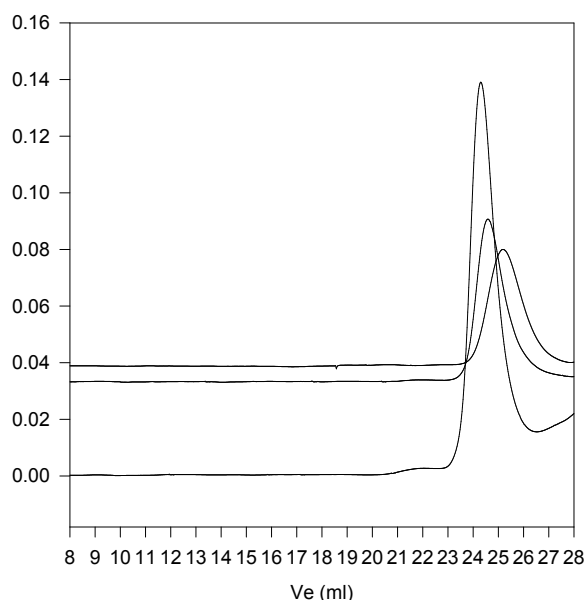
To cleave the tert.butyl ester moiety the hydrolysis was carried out in dioxane using acid catalyst. The degree of hydrolysis was checked by FTIR the disappearance of characteristics at 1362cm⁻¹.

Solubility:

Poly(ethylene oxide -b- methacrylic acid) is soluble in water, THF, methanol, ethanol and precipitate out in hexane, ether.

SEC of the block copolymer:

P18037-EOMAA



Size exclusion chromatography of poly(t.butyl methacrylate-b-ethylene oxide)

- End functionalized Poly(ethylene glycol methyl ether)
M_n=5000, M_w=5250, PI=1.05
- End functionalized Poly(tert.butyl methacrylate)
M_n=2000, M_w=2500, PI=1.18
- BlockAfter coupling reaction:
Block copolymer PEO(5000)-b-PtBMA(2000) Mw/Mn: 1.08
from Composition also from ¹H NMR
After Hydrolysis of ester functional group:
Mn: PEO-b-MAA 5000-b-1,200 Mw/Mn 1.08

References:

J. Wang, S. K. Varshney, J. Jerome and Ph. Teyssie "Synthesis of AB (BA) ABA and BAB Block copolymers of tert-butylmethacrylate (A) and ethylene oxide (B) "CA Vol 117, 16, 151478, J. Polym. Sci., Part-A: Polym. Chem. Ed., 1992, 30, 2251-2261.

Thermal analysis of the P18037-EOMAA

Thermal analysis of the samples was carried out on a TA Q100 differential scanning calorimeter at a heating rate of 10°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).

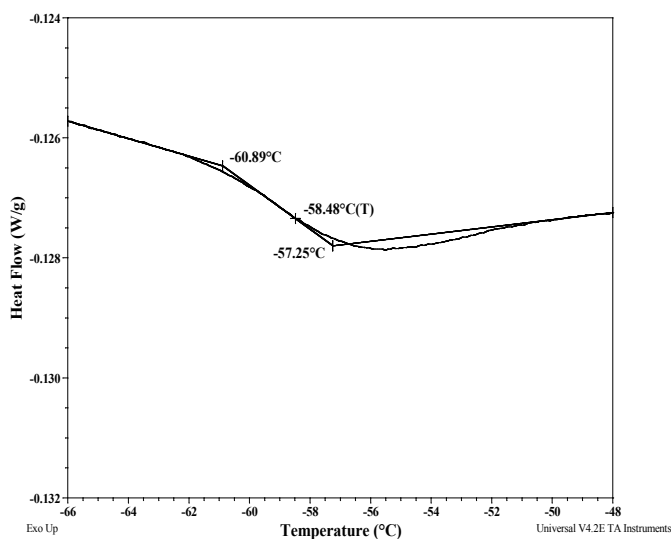
Melting and crystallization curve for the sample

The melting temperature (T_m) was taken as the maximum of the endothermic peak whereas the crystallization temperature (T_c) was considered as the minimum of the exothermic peak.

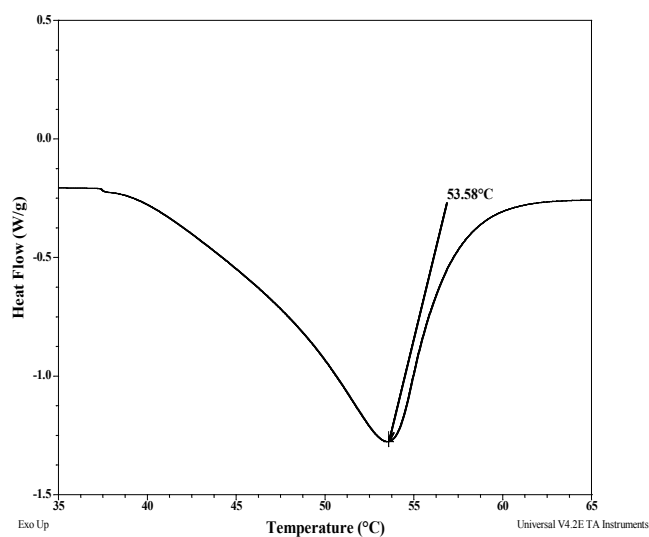
Thermal analysis results at a glance

Sample	T_m (°C)	T_c (°C)	T_g (°C)
EO Block	49	-	-58
AA block	-	-	Not distinct

Thermogram for the EO block:



Melting curve for the polymer:



DSC thermogram for MAA block:

