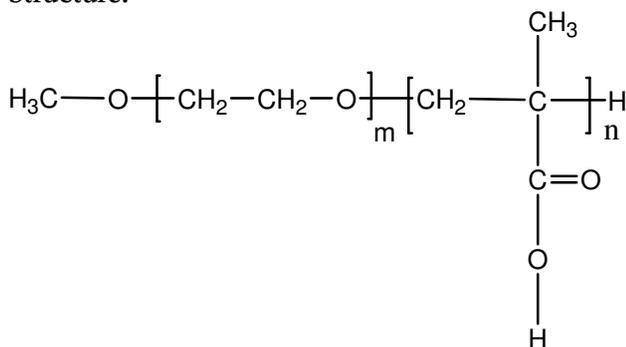


Sample Name: Poly(ethylene oxide -b- methacrylic acid)

Sample #: P20178-EOMAA

**Structure:**



**Composition:**

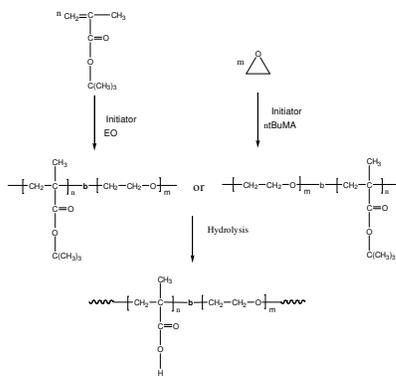
|                                 |      |
|---------------------------------|------|
| $M_n \times 10^3$<br>PEO-b-PMAA | PDI  |
| 7.5-b-15.0                      | 1.35 |

**Synthesis Procedure:**

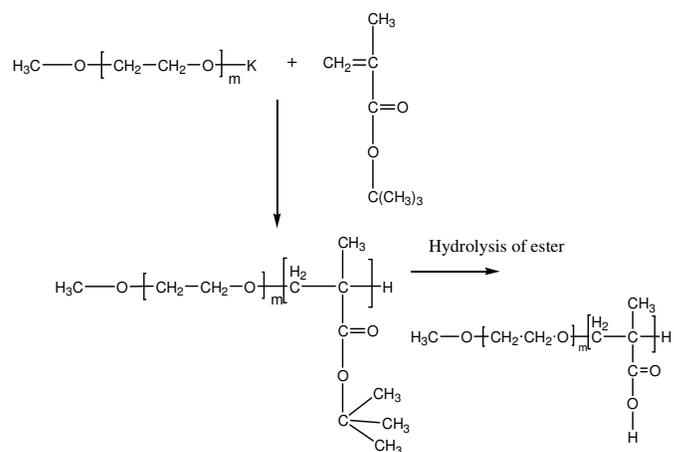
Poly(ethylene oxide -b- methacrylic acid) is prepared by 2 different routes:

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 group or

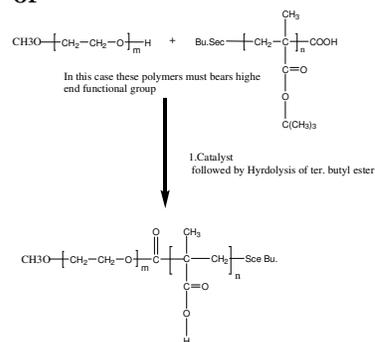
B. by chemical coupling reaction of the corresponding functionalized polymer. The scheme of the reaction is illustrated below:



or



or



**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  $^1\text{H-NMR}$  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.

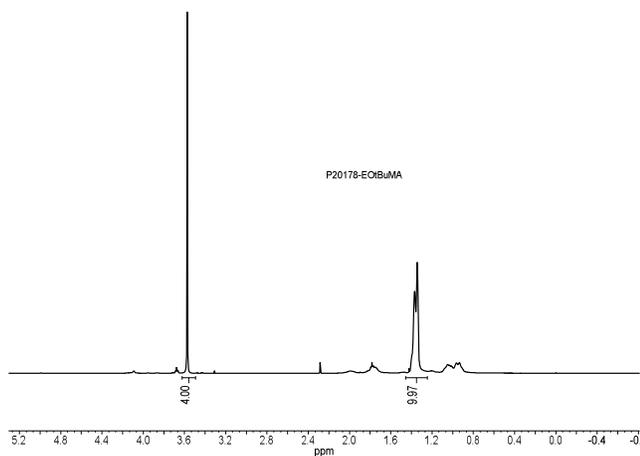
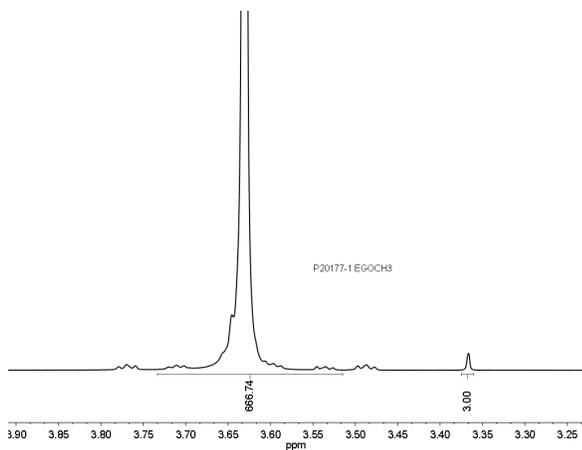
**Hydrolysis:**

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  $1362\text{cm}^{-1}$ .

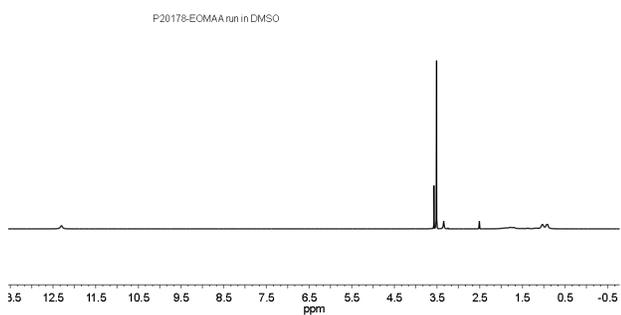
**Solubility:**

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

### $^1\text{H}$ NMR of PEG



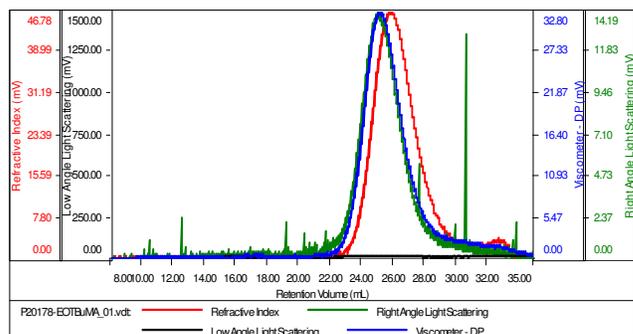
### $^1\text{H}$ NMR of EOMAA in Methanol



### SEC:

Sample ID: P20178-EOTBuMA

|                       |                           |
|-----------------------|---------------------------|
| Concentration (mg/mL) | 4.9919                    |
| Sample concn (mL/g)   | 0.0800                    |
| Method File           | PS80K-Jar22-2015-0003.vcm |
| Column Set            | 3xPL 1113-6300            |
| Solvent               | THF                       |



| Sample                | MW Number Average (Da) | MW Weight Average (Da) | MW at Peak (Da) | Polydispersity | Intrinsic Viscosity (dL/g) |
|-----------------------|------------------------|------------------------|-----------------|----------------|----------------------------|
| P20178-EOTBuMA_01.vct | 33,473                 | 45,128                 | 40,610          | 1.348          | 0.3940                     |

### 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.*