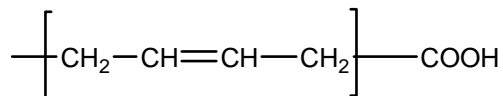


Sample Name: Carboxy-terminated
Polybutadiene (1,4-rich microstructure)

Sample #: P2950-BdCOOH

Structure:

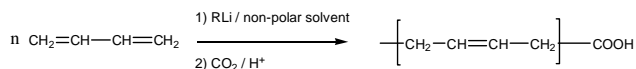


Composition:

| | |
|----------------------|-------|
| Mn x 10 ³ | PDI |
| 2.5 | 1.04 |
| COOH functionality: | >95 % |
| PBd 1,4-addition: | 87 % |

Synthesis Procedure:

1,4-addition carboxy-terminated polybutadiene was prepared by anionic living polymerization of butadiene in non-polar media (or in presence of 10% diethyl ether), followed by termination of the polymerization with dried CO₂. The scheme of the reaction is presented below:



Characterization:

The molecular weight and polydispersity index (PDI) are obtained by size exclusion chromatography (SEC) in THF. SEC analysis was performed on a Varian liquid chromatograph equipped with refractive and UV light scattering detectors. Three SEC columns from Supelco (G6000-4000-2000 HXL) were used with triple detectors from Viscotek Co.

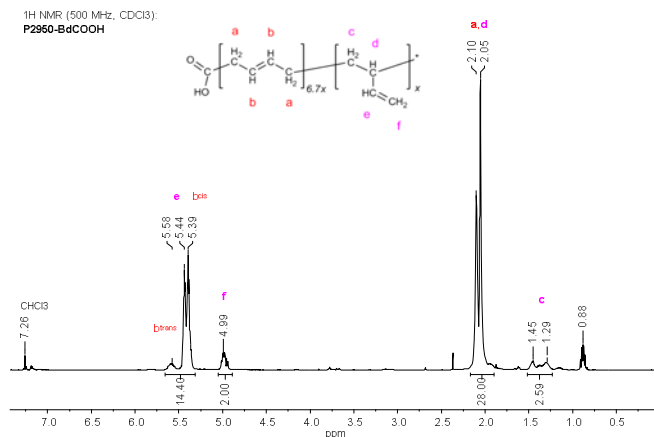
Functionality: The degree of polymer functionality was determined by acid-base titration.

Microstructure: The ratio between 1,4- and 1,2-addition was calculated by ¹H NMR spectroscopy.

Solubility:

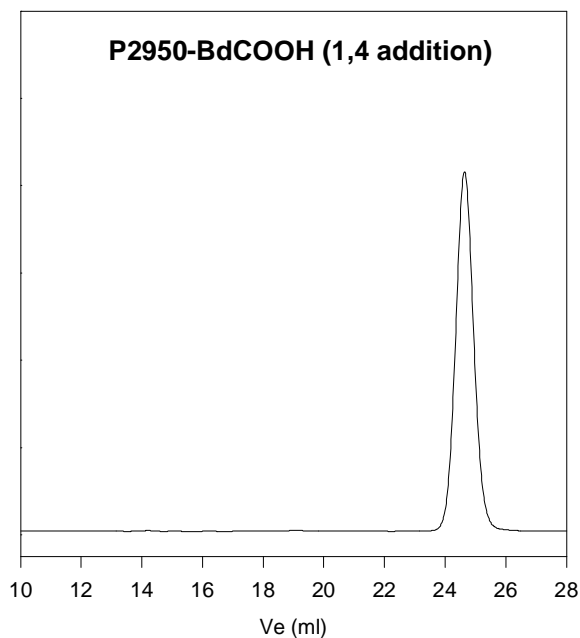
COOH terminated polybutadiene is soluble in DMF, THF, toluene, hexane, cyclohexane and CHCl₃. It precipitates from methanol, ethanol and water.

¹H NMR spectrum:



Ratio 1,4- to 1,2-addition = 0.87 : 0.13

SEC elugram:



Size exclusion chromatography of polybutadiene before termination with CO₂.

M_n=2500 M_w=2600, PI=1.04, functionality=>0.95%