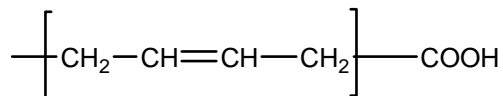


Sample Name: Carboxy-terminated Polybutadiene (1,4-rich microstructure)

Sample #: P8629-BdCOOH

Structure:

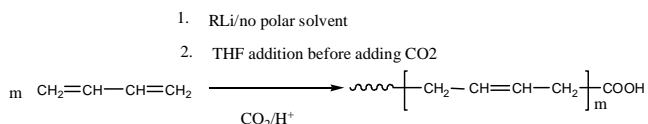


Composition:

Mn x 10 ³	PDI
5.2	1.10
COOH functionality:	> 80 %
PBd 1,4-addition:	92 %

Synthesis Procedure:

1,4-addition carboxy terminated polybutadiene was prepared by anionic living polymerization of butadiene in non-polar media, followed by end capping with a unit of diphenyl ethylene than the addition of THF followed by termination of the polymerization with dried CO₂. The scheme of the reaction is illustrated 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.

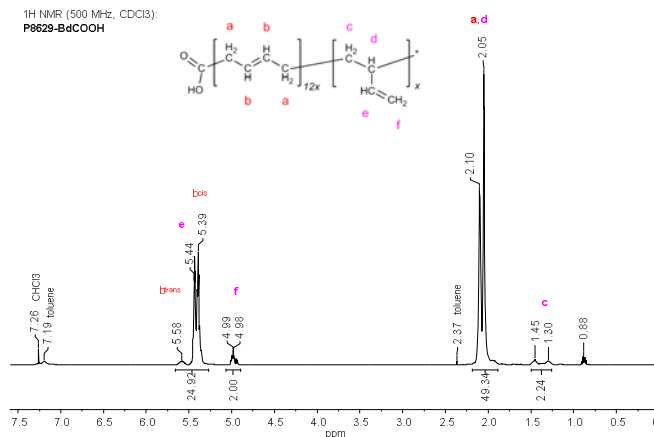
Functionality: The degree of polymer functionality was determined by acid-base titration.

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.

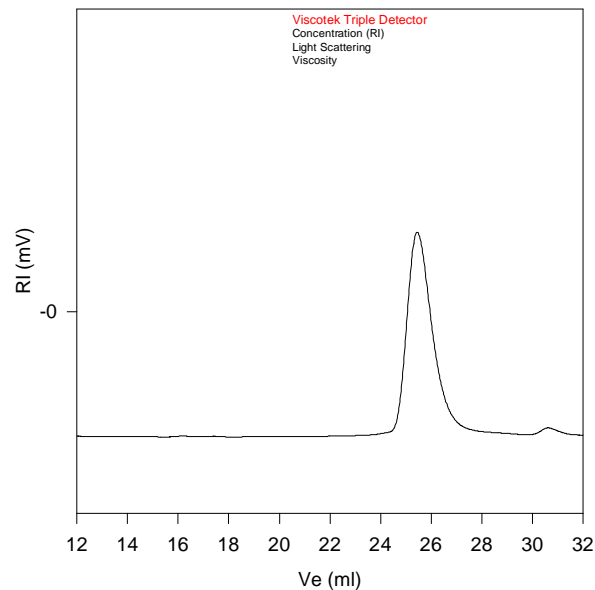
¹H NMR spectrum:



Ratio 1,4- to 1,2-addition = 0.92 : 0.08

SEC elugram:

P8629-BdCOOH



Size Exclusion Chromatography of polybutadiene end functionalized with COOH:

— M_n = 5200, M_w = 5800, M_w/M_n = 1.10

Functionality > 80%