

University of Groningen

Polystyrene-Poly(sodium methacrylate) Amphiphilic Block Copolymers by ATRP

Raffa, Patrizio; Brandenburg, Piter; Wever, Diego A. Z.; Broekhuis, Antonius; Picchioni, Francesco

Published in:
Macromolecules

DOI:
[10.1021/ma401453j](https://doi.org/10.1021/ma401453j)

IMPORTANT NOTE: You are advised to consult the publisher's version (publisher's PDF) if you wish to cite from it. Please check the document version below.

Document Version
Publisher's PDF, also known as Version of record

Publication date:
2013

[Link to publication in University of Groningen/UMCG research database](#)

Citation for published version (APA):

Raffa, P., Brandenburg, P., Wever, D. A. Z., Broekhuis, A. A., & Picchioni, F. (2013). Polystyrene-Poly(sodium methacrylate) Amphiphilic Block Copolymers by ATRP: Effect of Structure, pH, and Ionic Strength on Rheology of Aqueous Solutions. *Macromolecules*, 46(17), 7106-7111. DOI: 10.1021/ma401453j

Copyright

Other than for strictly personal use, it is not permitted to download or to forward/distribute the text or part of it without the consent of the author(s) and/or copyright holder(s), unless the work is under an open content license (like Creative Commons).

Take-down policy

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

Downloaded from the University of Groningen/UMCG research database (Pure): <http://www.rug.nl/research/portal>. For technical reasons the number of authors shown on this cover page is limited to 10 maximum.

Polystyrene-Poly(sodium methacrylate) amphiphilic block copolymers by ATRP: effect of structure, pH and ionic strength on rheology of aqueous solutions

Supporting Information

The synthesis of mono- di- and tetrafunctional PS-Br macroinitiators were performed in bulk at 100°C, using CuBr as catalyst and TMEDA as ligand. Results are summarized in Table S1.

Table S1. Synthesis of Polystyrene macroinitiators

	M/C/L/I ^(a)	Time (h)	Conv %	Theoretical M _n ^(b)	M _n (GPC)	PDI
PS1	200/1/1/1	20	24.0	5006	4325	1.26
PS2	120/2/2/1	15	81.5	10188	11250	1.34
PS4	120/4/4/1	5	77.7	9720	9491	1.16

(a) M = monomer, C = catalyst, L = ligand, I = initiator; (b) based on conversion

Chain extension with tBMA was performed in anisole at 90°C, using CuCl or CuBr as catalyst and Me₆TREN as ligands. Results are summarized in Table S2.

Table S2. Chain extension of PS-Br macroinitiator with tBMA

	M/C/L/I ^(a)	Time (h)	Conv %	PS – tBMA (NMR)	M _n (NMR)	M _n (GPC) ^(c)
PS1BMA	1000/2/2/1	1	39.8	49 - 555	83078	84970 (1.3)
PS2BMA	2000/2/2/1	5	26.5	108 - 625	100040	106360 (1.18)
PS4BMA	2000/2/2/1	5	58.9	91 - 1470	218269	129300 (1.09)

(a) M = monomer, C = catalyst, L = ligand, I = initiator; (c) DPI reported in parenthesis

The final copolymers were characterized by ¹H-NMR and GPC. The composition and molecular weight determined by conversion, NMR and GPC were in reasonable agreement in all cases (Table S2). The bigger discrepancies observed by GPC for PS4BMA can be ascribed to the deviation from linearity observed in the calibration procedure at high molecular weights.

The molar ratio between the styrenic and the acrylic moieties calculated by integration of ¹H-NMR signals of PS-*b*-PtBMA in CDCl₃ is consistent with the results found by GPC (see Table S2).

¹H-NMR of the hydrolyzed products in d₆-DMSO show complete disappearance of the tBu peaks (δ = 1.3 ppm), meaning that hydrolysis is complete (Figure S1).

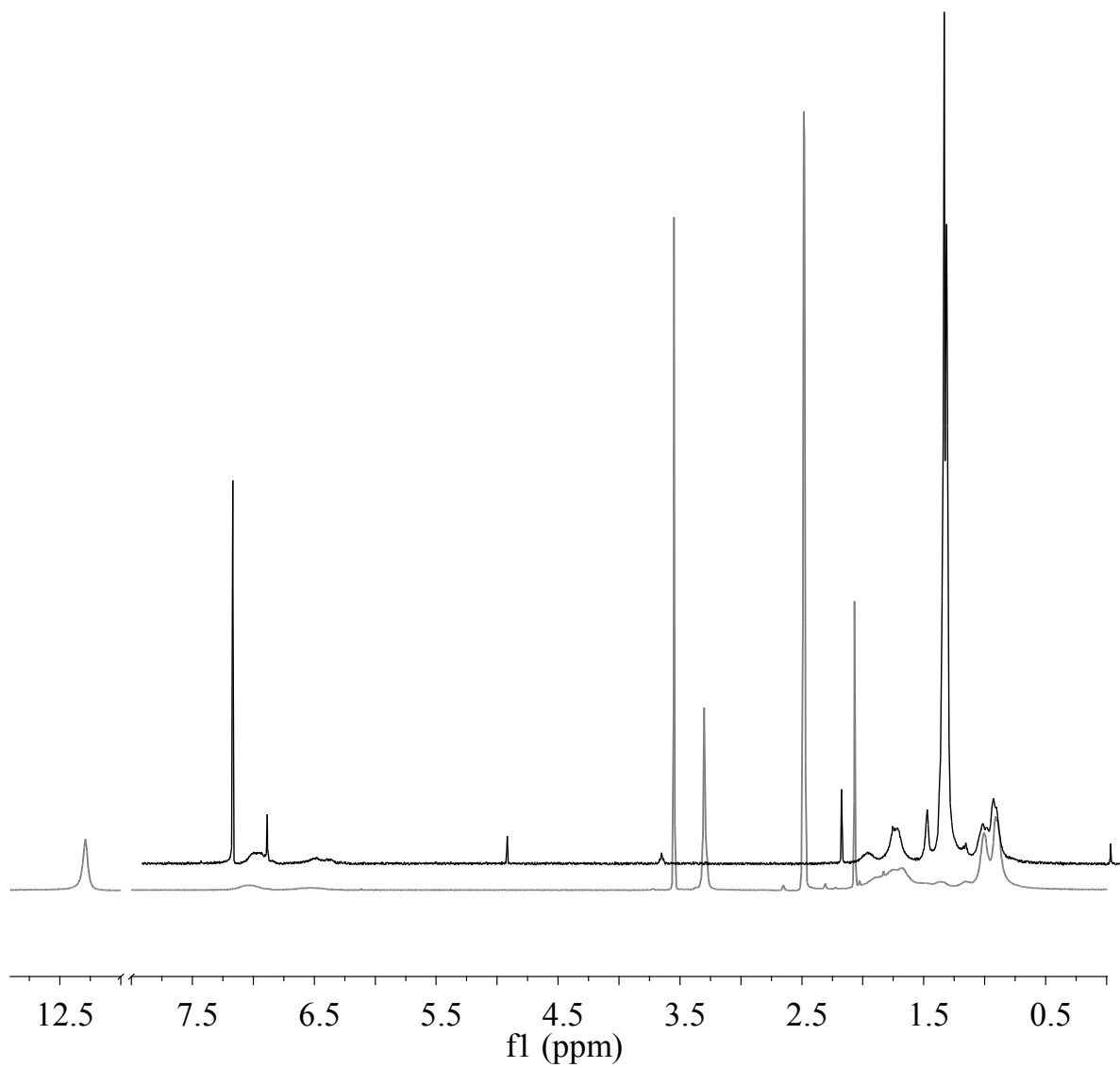


Figure S1. ¹H-NMR of PS2BMA in CDCl₃ (black) and PS2MA in d₆-DMSO (gray)