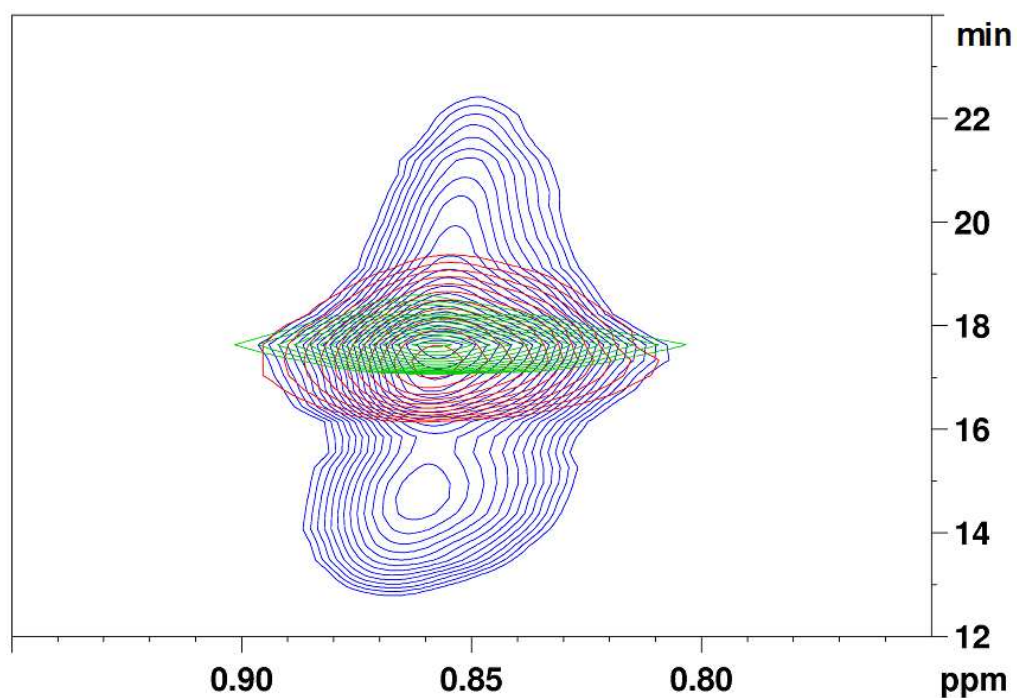


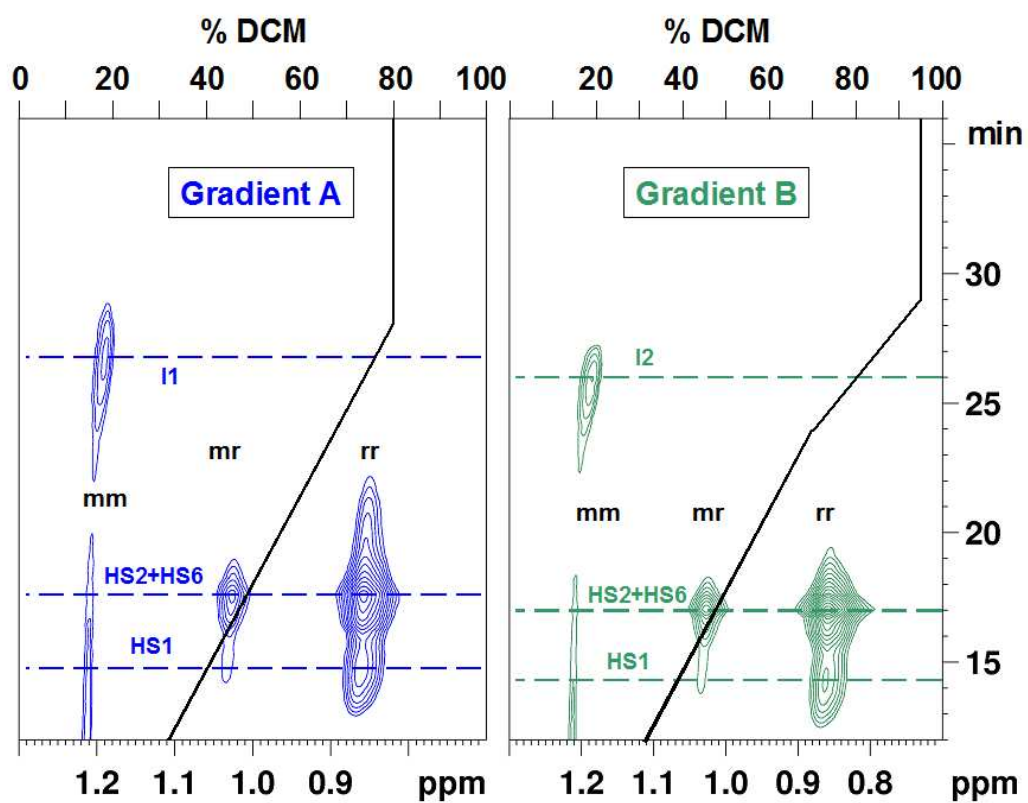
## Supporting Information

### Online HPLC-NMR: An efficient method for the analysis of PMMA with respect to tacticity

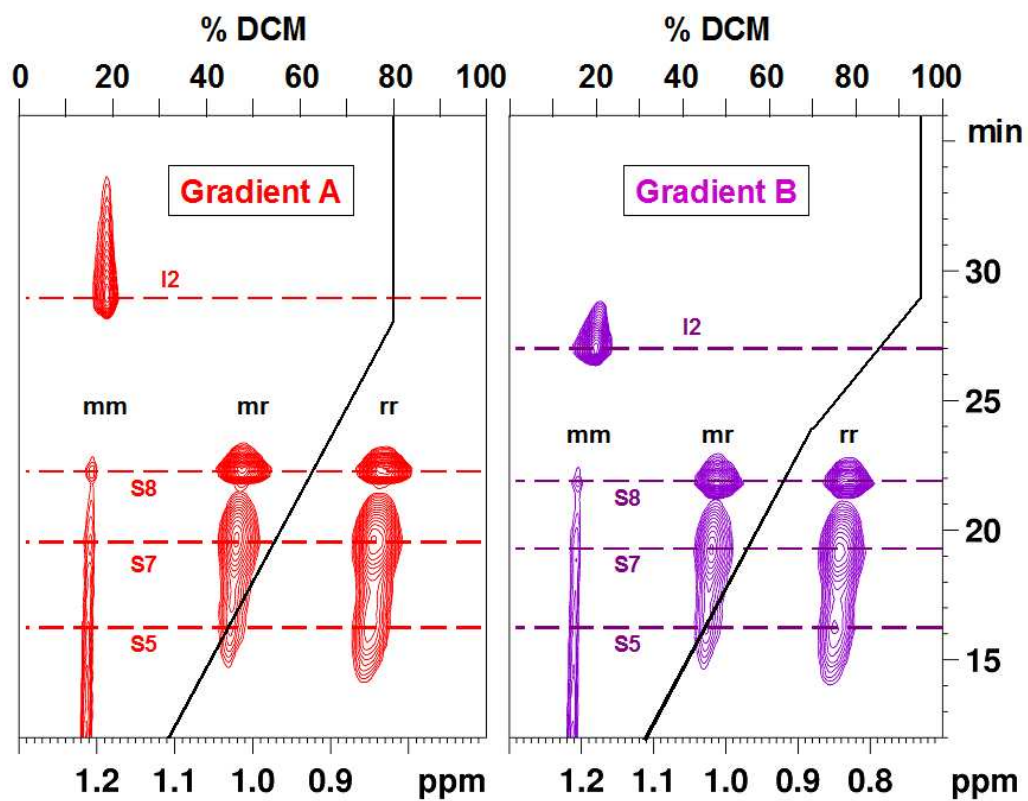
Mathias Hehn, Khumo Maiko, Harald Pasch, Wolf Hiller



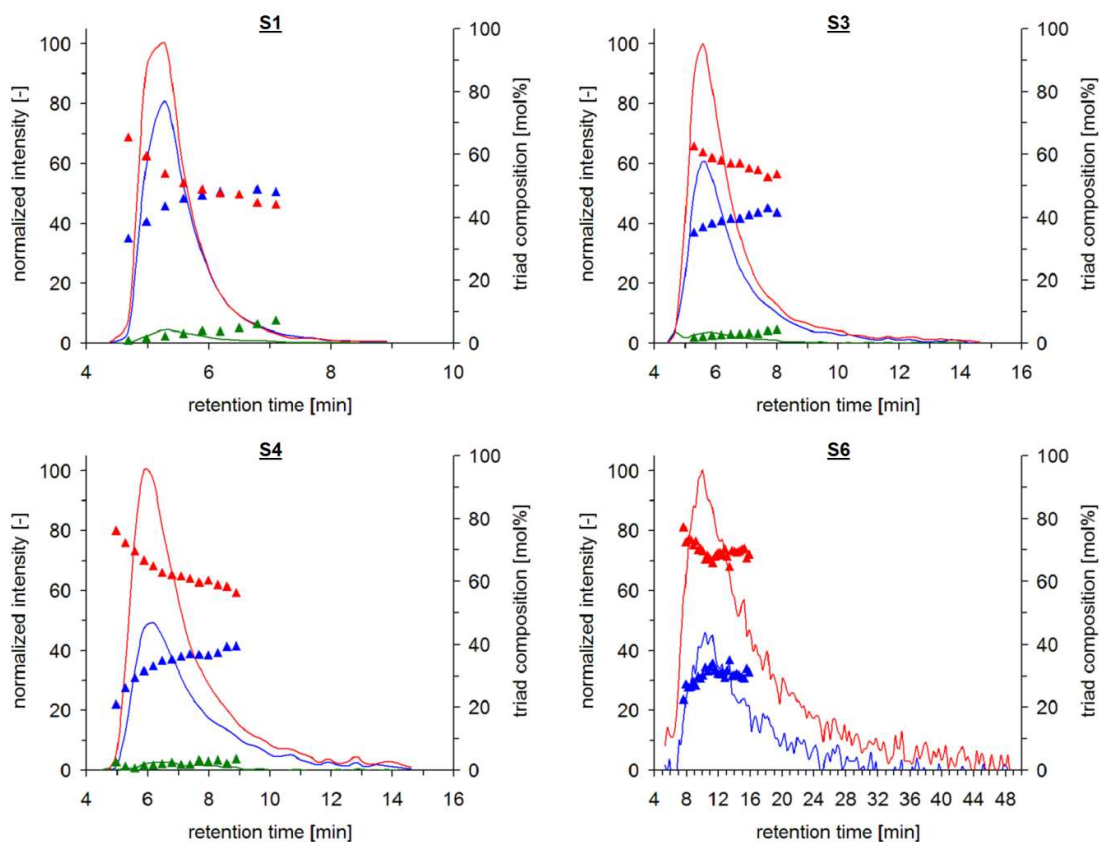
**Figure S1.** Overlaid HPLC-NMR contour plots of the syndiotactic  $\alpha$ -CH<sub>3</sub> signals of blend 1 (blue), sample HS2 (red) and sample HS6 (green), measured with gradient A.



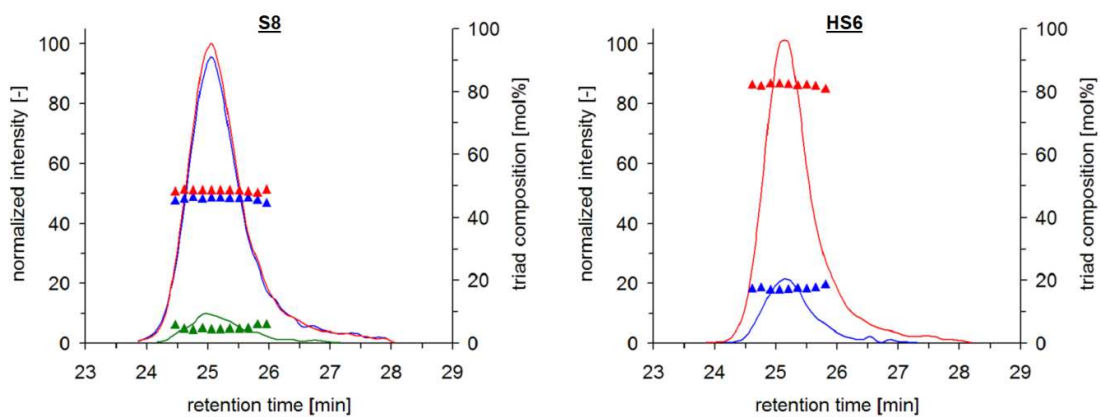
**Figure S2.** HPLC-NMR measurements of blend 1 performed with gradients A (left) and B (right); black solid lines: DCM content with respect to the effective gradient.



**Figure S3.** HPLC-NMR measurements of blend 2 performed with gradients A (left) and B (right); black solid lines: DCM content with respect to the effective gradient.



**Figure S4.** Isocratic HPLC-NMR chromatograms of samples S1, S3, S4 and S6 at a solvent composition of 60% acetone and 40% DCM; solid lines: chromatograms of  $\alpha$ -CH<sub>3</sub> signals, triangles = change in triad composition; red = rr triad, blue = mr triad, green = mm triad (mm not observable in S6 because of too low signal-to-noise-levels).



**Figure S5.** SEC-NMR chromatograms of samples S8 and HS6; solid lines: chromatograms of  $\alpha$ -CH<sub>3</sub> signals, triangles = change in triad composition; red = rr triad, blue = mr triad, green = mm triad (mm in HS6 is not evaluated because of too low signal-to-noise-level).

**Table S1.** Average microstructures of the samples measured by SEC-NMR (calculated over the total elution times).

Sample	Tacticity [mol%]			Sample	Tacticity [mol%]		
	mm	mr	rr		mm	mr	rr
S5	5.0	37.6	57.4	HS2	2.4	21.3	76.3
S7	2.8	36.6	60.6	HS6	0.7	17.1	82.2
S8	5.0	46.6	48.4	I1	93.6	-	6.4
HS1	1.5	25.5	73.0	I2	98.3	-	1.7