

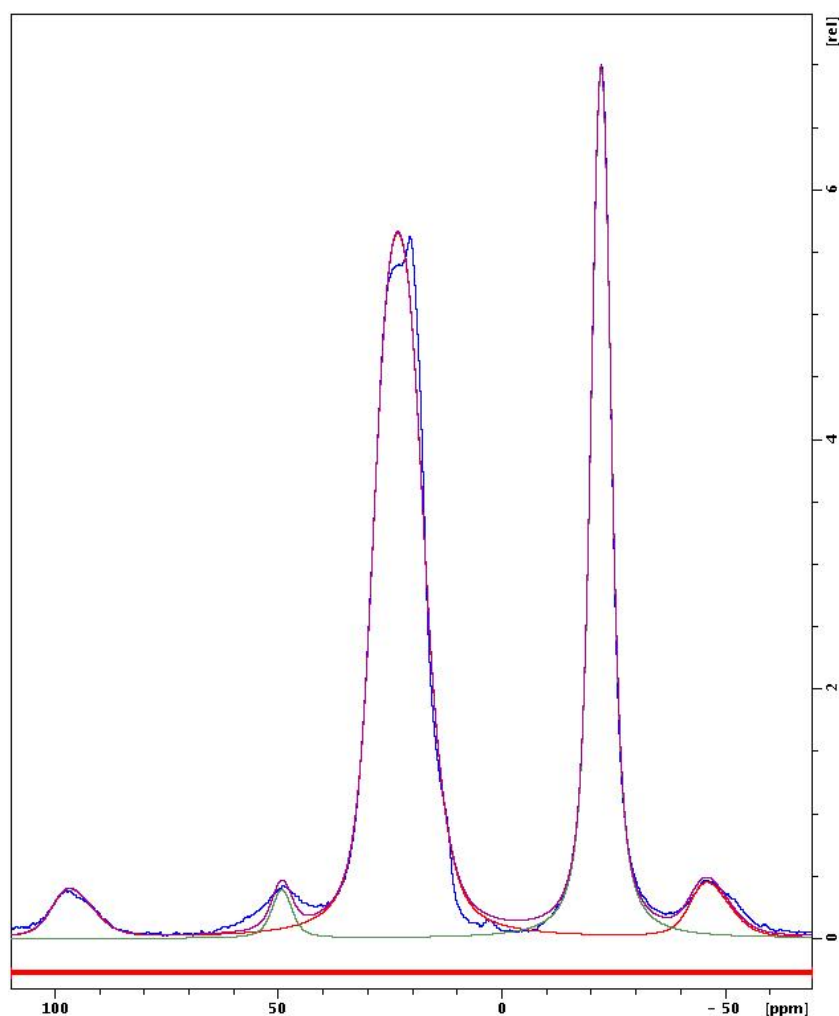


Fig. S1. Example output from Bruker TopSpin programme after lineshape analysis, for the spectrum shown in Fig. 3, 90 °C. Blue curve is experimental spectrum, red curve is fitted envelope of 2 peaks + side-bands. Parameters for the broader peak are compared below to those reported by Gervais *et al.* (Ref 10: *Chem. Mater.* **13** 1700, 2001) for a BN₃ resonance in polyborazilene.

Parameter	Present study	BN ₃ , Gervais <i>et al.</i>
δ_{iso} /ppm	30	27
δ_{CSA} /ppm	78	-
η_{CSA}	0.75	0.1
η_{quad}	0.03	
CQ_{quad} /MHz	2.62	2.7
Lorentz broadening /Hz	620	1800
Gauss broadening /Hz	866	

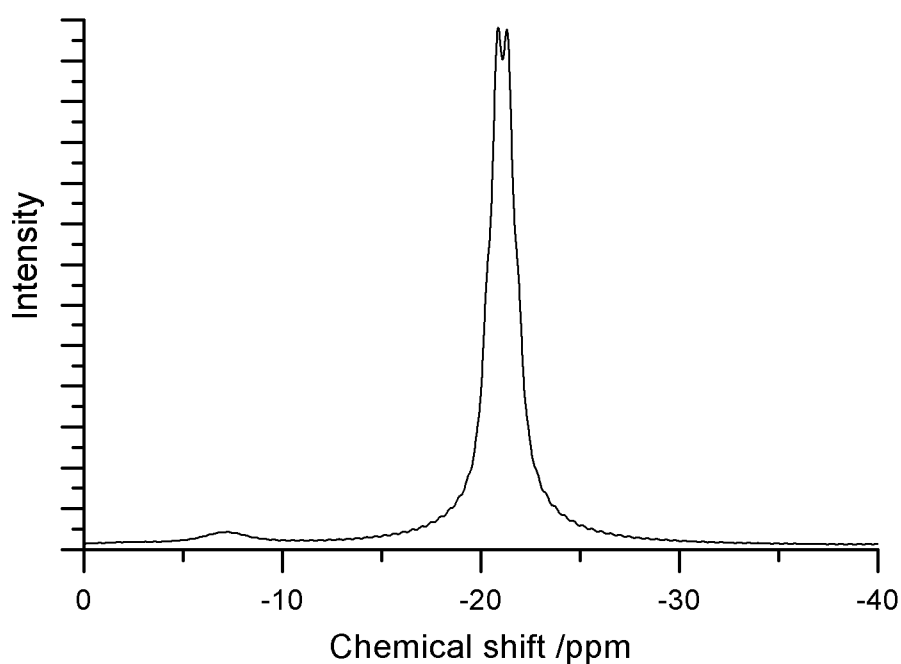


Fig. S2. NMR spectrum of hybrid material after a small amount of hydrogen loss, exhibiting weak resonance at -7ppm. This spectrum is from the same sample shown (before hydrogen loss) in Fig. 1 (A).

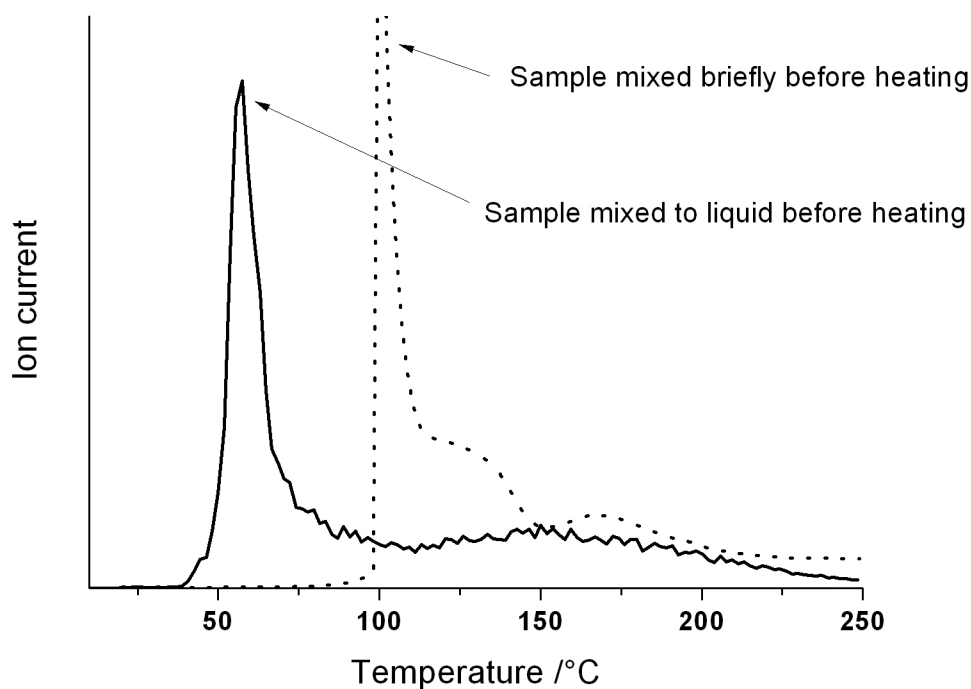


Fig. S3. Comparison of hydrogen release profiles for sample mixed to form liquid phase before heating (solid line) and sample mixed for a shorter time and consequently not forming the liquid phase (dotted line).