## **Electronic supporting information**

## Formation of gold nanoparticles in polymeric nanowires by lowtemperature thermolysis of gold mesitylene

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Figure S1. First and second differential scanning calorimetry heating runs of bulk PS-*b*-isoPMMA (Netzsch DCS 404 C; heating and cooling rates 10 K/min).



Figure S2: Mass spectrum of the degradation product of gold mesitylene recorded with a Varian MAT 711 mass spectrometer. The degradation product was isolated as a colourless crystalline solid at the cold end of a Schlenk tube within which the gold mesitylene contained in gold mesitylene/PS-*b*-isoPMMA nanowires located in AAO was thermolized. The peaks can be assigned to 2,2',4,4',6,6'-hexamethylbiphenyl according to the literature <sup>1,2</sup> (EI-MS: m/z 238 [M]<sup>+</sup>, 223 [M-CH<sub>3</sub>]<sup>+</sup>, 208 [M-2 CH<sub>3</sub>]<sup>+</sup>).



Figure S3: <sup>1</sup>H-NMR spectrum of the degradation product of gold mesitylene in deuterated benzene recorded with a JEOL 400 MHz NMR spectrometer. The degradation product was isolated as a colourless crystalline solid at the cold end of a Schlenk tube within which the gold mesitylene contained in gold mesitylene/PS-*b*-isoPMMA nanowires located in AAO was thermolized. The signals can be assigned to 2,2',4,4',6,6'-hexamethylbiphenyl according to the literature.<sup>1,2</sup> <sup>1</sup>H-NMR (C<sub>6</sub>D<sub>6</sub>, 400 MHz):  $\delta = 6,90$  ppm (*s*, 4 H, aryl-H); 2,21 ppm (*s*, 6 H, *p*-CH<sub>3</sub>); 1,93 ppm (*s*, 12 H, *o*-CH<sub>3</sub>).



Figure S4. Raman spectra of gold mesitylene powder (black curve) as well as of NW(1/1.1)-120 located in AAO (green curve), NW(1/1.1)-150 located in AAO (blue curve) and gold mesitylene/PS-*b*-isoPMMA nanowires without any heat treatment located in AAO (red curve) obtained from the same gold mesitylene/PS-*b*-isoPMMA solution. The asymmetric deformation vibration of the methyl groups at the phenyl rings of the mesitylene ligands at 1571 cm<sup>-1</sup> appears in the spectra of gold mesitylene powder, of gold mesitylene/PS-*b*isoPMMA nanowires without any heat treatment and of NW(1/1.1)-120 but not in the spectrum of NW(1/1.1)-150. The Raman spectra were acquired as described in the experimental part of the main manuscript (excitation wavelength: 532 nm).



Figure S5. X-ray powder diffraction pattern of released, randomly oriented *NW*(1/1.9)-200 obtained with a STOE STADI P transmission powder diffractometer in theta/2theta geometry using Cu-K<sub> $\alpha$ </sub> radiation.

<sup>&</sup>lt;sup>1</sup> G. Cahiez, C. Chaboche, F. Mahuteau-Betzer and M. Ahr, Org. Lett. 2005, 7, 1943-1946.

<sup>&</sup>lt;sup>2</sup> Y. Miyake, M. Wu, M. J. Rahman, Y. Kuwatani and M. Iyoda, J. Org. Chem. 2006, 71, 6110-6117.