## Supplementary information

## Facile, efficient synthesis of a phosphinated hydroxyl diamine, and properties of its high-performance poly(hydroxyl imides) and polyimide/SiO<sub>2</sub> hybrids

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Polyimide	GPC Data <sup>a</sup>		Solubility data <sup>c</sup>					
	$M_n^{b}(\times 10^4)$	$M_{w}^{c}$ (×10 <sup>4</sup> )	NMP	DMAc	DMSO	m-cresol	THF	$CH_2Cl_2$
2a	-	-	±	±	±	±		_
2b	-	-	+h	±	±	+h	_	_
2c	-	-	±	±	_	±	_	_
2d	-	-	+h	±	±	+h	_	_
2e	6.3	10.4	+	+	+	+	+	±

Table S1. GPC and solubility data of poly (hydroxyl imides) 2

<sup>a</sup> Relative to polystyrene standard, using THF as eluent. <sup>b</sup>M<sub>n</sub>: number-average molecular weight, M<sub>w</sub>: weight-average molecular weight. <sup>c</sup>Solubility was tested with a 5 mg sample in 0.5 g of solvent at room temperature. +, soluble; +h, soluble on heating;  $\pm$ , partially soluble on heating; -, insoluble on heating.



Figure S1. DSC thermograms of as-prepared, and DMAc or methanol recrystallized product.



Figure S2. <sup>1</sup>H-<sup>1</sup>H COSY NMR spectrum of (1).



Figure S3. <sup>1</sup>H-<sup>13</sup>C HETCOR NMR spectrum of (1).



Figure S4. IR spectra of solution-polymerization 2e after thermal treatment at 200 °C (1h) and 300

<sup>o</sup>C (1h).



Figure S5. <sup>1</sup>H NMR spectrum of 2e.



Figure S6. DMA thermograms of 2c and 2c'.



Figure S7. Pictures of 2e and 2e16.



Figure S8. Solid-state <sup>29</sup>Si NMR spectrum of **2e16**.