

## Supporting Information

# Semi-fluorinated di and triblock copolymer nano-objects prepared via RAFT alcoholic dispersion polymerization (PISA)

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Table S1: Summary of assemblies focusing on HCFDDMA

N°	Composition	Target DP	Conversion %	Theo. DP, x	Size DLS (nm)	Size TEM (nm)
1	PDMA <sub>58</sub> -PBzMA <sub>x</sub>	200	99	198	70	46
2	PDMA <sub>58</sub> -PBzMA <sub>198</sub> -PHCFDDMA <sub>x</sub>	20	95	19	172	60
3	PDMA <sub>58</sub> -PBzMA <sub>198</sub> -PHCFDDMA <sub>x</sub>	50	NA	NA	NA	NA

Table S2: Summary of the different copolymers synthesized and their dimensions

N°	Composition	Target DP	Conversion %	Theo. DP, x	Size DLS (nm)	Size TEM (nm)	Morphology
1	PDMA <sub>58</sub> -PBzMA <sub>x</sub>	70	99	69	40	23	Sphere
2		200	97	194	75	46	Sphere
3	PDMA <sub>58</sub> -PBzMA <sub>69</sub> -PTDFOA <sub>x</sub>	50	95	48	58	26	Sphere
4		100	91	91	95	49	Sphere
5		150	96	144	160	54	Sphere
6	PDMA <sub>58</sub> -PBzMA <sub>194</sub> -PTDFOA <sub>x</sub>	50	88	44	70	52	Sphere
7		100	83	83	90	61	Sphere
8		150	79	120	170	69	Sphere

Table S3: Summary of selected copolymers molecular weights and dipersities obtained from THF SEC calibrated with PS standards.

N°	Composition	<i>M<sub>n</sub></i> (g/mol)	<i>M<sub>w</sub></i> (g/mol)	Đ
1	PDMA <sub>39</sub> -PBzMA <sub>198</sub> -PHFBMA <sub>96</sub>	37000	45000	1.22
2	PDMA <sub>39</sub> -PBzMA <sub>198</sub> -PHFBMA <sub>144</sub>	59000	85000	1.45
3	PDMA <sub>39</sub> -PHFBMA <sub>99</sub> -PBzMA <sub>198</sub>	98000	126000	1.28
4	PDMA <sub>39</sub> -PHFBMA <sub>149</sub> -PBzMA <sub>198</sub>	100000	123000	1.23
5	PDMA <sub>58</sub> -PBzMA <sub>194</sub> -PTDFOA <sub>83</sub>	64000	79000	1.23
6	PDMA <sub>58</sub> -PBzMA <sub>194</sub> -PTDFOA <sub>120</sub>	42000	52000	1.25

**Figure S1.**  $^1\text{H}$  NMR spectra of unpurified (a) and purified (b) PDMA macro-CTA. For more details please refer to entry 1 in Table 1.

