Table S1. Amounts of surfactant used and the resulting ratio of metal [M] to surfactant [Sf], samples **1** to **10**; $Fe(OEt_3)$ (0.5 mmol, 95.9 mg) and $Pt(acac)_2$ (0.25 mmol, 100 mg) were used in dioctyl ether (17 mL), **11** to **32**; $Fe(OEt)_3$ (1 mmol, 192 mg) and $Pt(acac)_2$ (0.5 mmol, 201 mg) in dioctyl ether and dibenzyl ether (in bold) 4
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Experimental

Chemicals: Iron (III) ethoxide [$Fe(OEt)_3$, 99.6%] was purchased from Alfa Aesar, Johnson Matthey company, Pt (II) acetylacetonate [$Pt(acac)_2$, 97%], oleic acid, [OA, 99%), oleylamine [OLA, 70%), dioctyl ether (99%) and dibenzyl ether (≥ 98%) were purchased from Sigma Aldrich, UK LLC. Pt (II) acetylacetonate [$Pt(acac)_2$, 98%] was also purchased from ACROS organics. Dioctyl ether and dibenzyl ether were degassed by bubbling N_2 through them overnight, all other reagents were used without further purification or treatment.

Syntheses: The syntheses were performed under oxygen free conditions on a Schlenk line with flowing N_2. In a typical synthesis, the ratio of Fe to Pt precursor amounts used was 2: 1. In the presence of different amounts of OA and OLA (**Table S1**), the volume of solvent, (dioctyl ether or dibenzyl ether), was kept constant (17 mL). $Fe(OEt)_3$ was weighed into a 100 mL round bottomed flask in a glove box. On a Schlenk line, $Pt(acac)_2$ was added, and with stirring (500 rpm), OLA, OA, and dioctyl ether (or dibenzyl ether) were subsequently added to the vessel. The reaction was degassed three times by evacuation and purging with N_2. The reaction mixture was stirred at 500 rpm for 5 minutes before heating under N_2 to 287 °C (20 °C / min, samples **1-10**) or 260 °C (20 °C / min, samples **11-32**) where it was maintained at this temperature for 30 minutes with a temperature controller and an internal electronic probe. In the range 140-160 °C, white fumes were observed; a white precipitate subsequently appeared on the surface of the liquid and disappeared as the temperature approached 260 °C. The identification of the white residue is beyond the scope of this paper, however, it is thought to either be the acetylacetonate by-product of $Pt(acac)_2$, or impurities in the OLA[43]. After 30 minutes, the reaction was cooled to 50 °C, degassed (by bubbling with N_2) ethanol (20 mL) was injected into the vessel to precipitate products and dissolve any remaining $Fe(OEt)_3$. $Fe(OEt)_3$ does not dissolve in dioctyl ether at room temperature. Cleaning was performed three times by precipitation with ethanol, centrifugation, and redispersion in hexane.

The effect of increasing total surfactant amount (tSf) was first studied; then the effects of individual surfactant amounts (nOA and nOLA) were investigated in dioctyl ether and dibenzyl ether. The amounts of OA and OLA employed are detailed in supplementary information (**Table 1**). Samples **1** – **6** reflect a linear increase in tSf while OA: OLA is fixed at 1: 1. Samples **7** – **10**, tSf was maintained at 20 mmol while OA: OLA was changed through 1: 3, 1: 2, 2: 1 and 3: 1. Samples **11-14** and **20-23** reflect an increase in tSf whilst maintaining OA: OLA = 1: 6, in dioctyl ether and dibenzyl ether respectively. Samples **15-19** and **24-28**, tSf was maintained at 17.5 mmol while OA: OLA was changed through 2: 5, 3: 4, 4: 3, 5: 2, and 6: 1, in dioctyl ether and dibenzyl ether respectively. Samples **29-32**, OA: OLA is kept constant at 6: 1, while tSf was changed, in dioctyl ether and dibenzyl ether.

Table S1. Amounts of surfactant used and the resulting ratio of metal [M] to surfactant [Sf], samples **1** to **10**; $Fe(OEt_3)$ (0.5 mmol, 95.9 mg) and $Pt(acac)_2$ (0.25 mmol, 100 mg) were used in dioctyl ether (17 mL), **11** to **32**; $Fe(OEt)_3$ (1 mmol, 192 mg) and $Pt(acac)_2$ (0.5 mmol, 201 mg) in dioctyl ether and dibenzyl ether (in bold)

Sample	OA/ mmol	OLA/ mmol	[OA+OLA]/ mM	Ratio [M]: [Sf]
1	5	5	477	1: 13.3
2	6	6	552	1: 16
3	7	7	621	1: 18.7
4	8	8	686	1: 21.3
5	9	9	746	1: 24
6	10	10	803	1: 26.7
7	5	15	779	1: 26.7
8	6.7	13.3	787	1: 26.7
9	13.3	6.7	820	1: 26.7
10	15	5	828	1: 26.7
11 (**20**)	1.5	9	482	1: 10.3
12 (**21**)	2.5	15	701	1: 17.2
13 (**22**)	3.5	21	871	1: 24.1
14 (**23**)	4.5	27	1006	1: 31
15 (**24**)	5	12.5	710	1: 17.2
16 (**25**)	7.5	10	719	1: 17.2
17 (**26**)	10	7.5	728	1: 17.2
18 (**27**)	12.5	5	737	1: 17.2
19 (**28**)	15	2.5	747	1: 17.2
29 (**31**)	9	1.5	503	1: 10.3
30 (**32**)	27	4.5	1102	1: 31.1

Table S2. Size of discrete NPs and mcNPs components calculated from XRD using the Scherrer equation tool in the software 'X'Pert HighScore Plus'.

Sample	Size / Å	Sample	Size / Å	Sample	Size / Å
1	41	**12**	35 / 647	**23**	36
2	44	**13**	35 / 700	**24**	27
3	44	**14**	37	**25**	25
4	49	**15**	30	**26**	39
5	44	**16**	32	**27**	48
6	44	**17**	40	**28**	47
7	44	**18**	48	**29**	37
8	43	**19**	55	**30**	48
9	41	**20**	28	**31**	45
10	41	**21**	28	**32**	43
11	38 / 764	**22**	30		

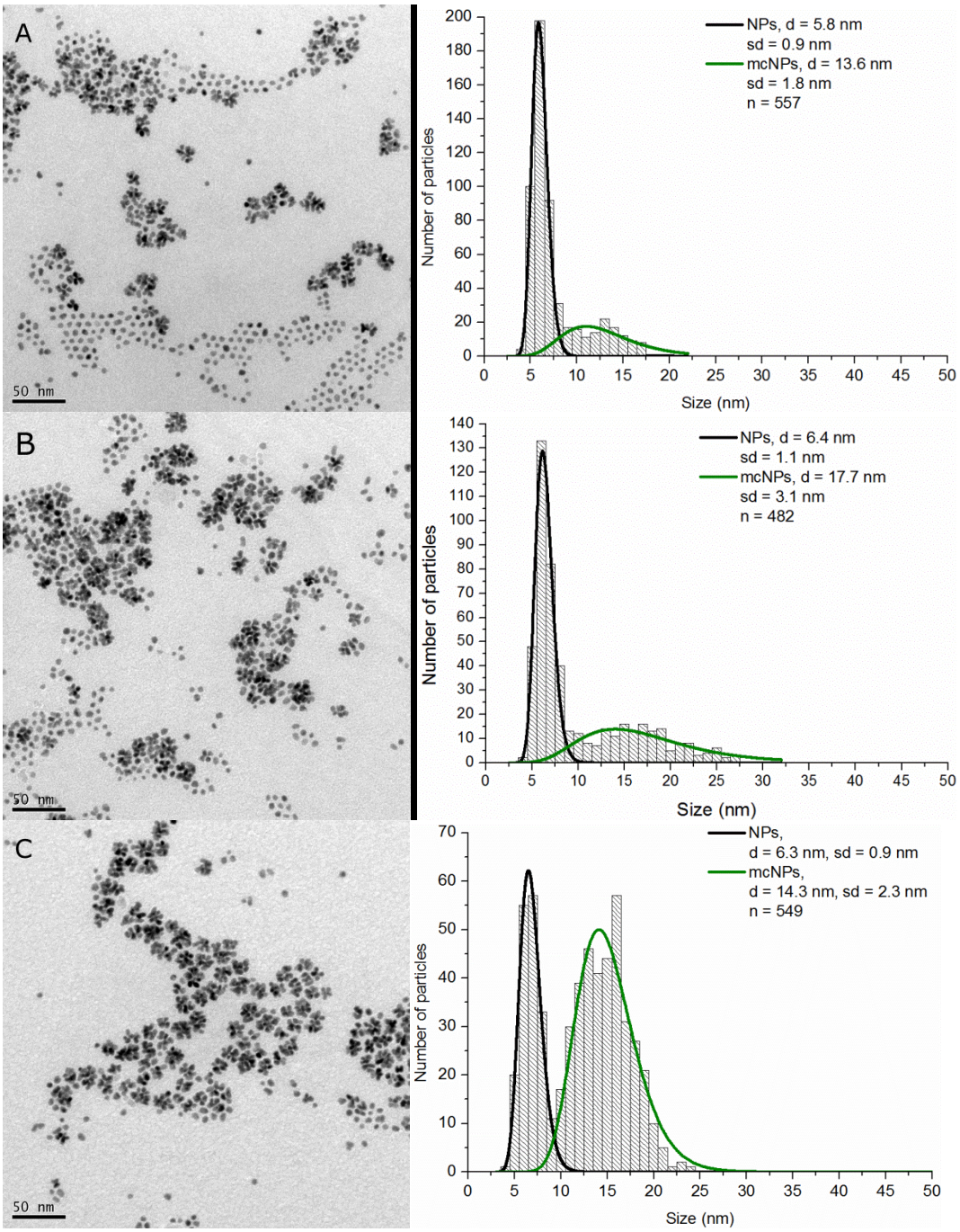
Table S3. XPS composition and peak position data of mcNPs and discrete NPs synthesized with varying surfactant amounts and in both dioctyl and dibenzyl ether.

	Morphology		Composition		Peak Position		
Sample	d NP / nm	d mcNP / nm	ICP Fe %	XPS Fe %	Charging	Pt position (eV)	Fe position (eV)

11	Aggregate		63	49	Yes	71.3	710.8
12	N.O.	41.0 ± 8.2	72	61	Yes	71.4	710.8
14	N.O.	44.3 ± 7.3	61	37	No	71.4	711
20	N.O.	45.3 ± 6.6	66	49	Minor	71.3	710.8
21	N.O.	47.6 ± 6.6	46	37	No	71.4	711
23	N.O.	22.8 ± 2.0	76	46	No	71.4	710.9
29	7.2 ± 0.8	N.O.	24	28	No	71.3	709.7
19	8.2 ± 1.3	N.O.	28	22	No	71.3	709.8
30	7.6 ± 0.8	N.O.	30	25	No	71.3	710.7
31	8.8 ± 0.8	N.O.	32	35	Yes	71.3	711.4
28	5.9 ± 1.2	N.O.	34	43	Yes	71.3	710.6
32	7.3 ± 0.6	N.O.	30	26	No	71.3	711

Table S4. Thermal gravimetric analyses, organic content of select samples and resulting effect on magnetic saturation properties.

Sample	Solvent	TGA determined organic matter / %	Magnetic saturation / emu.g^{-1}	Magnetic saturation minus organic matter / emu.g^{-1}
11	Dioctyl ether	18	20	24
12	Dioctyl ether	21	23	29
21	Dibenzyl ether	4	19	20
23	Dibenzyl ether	4	25	26



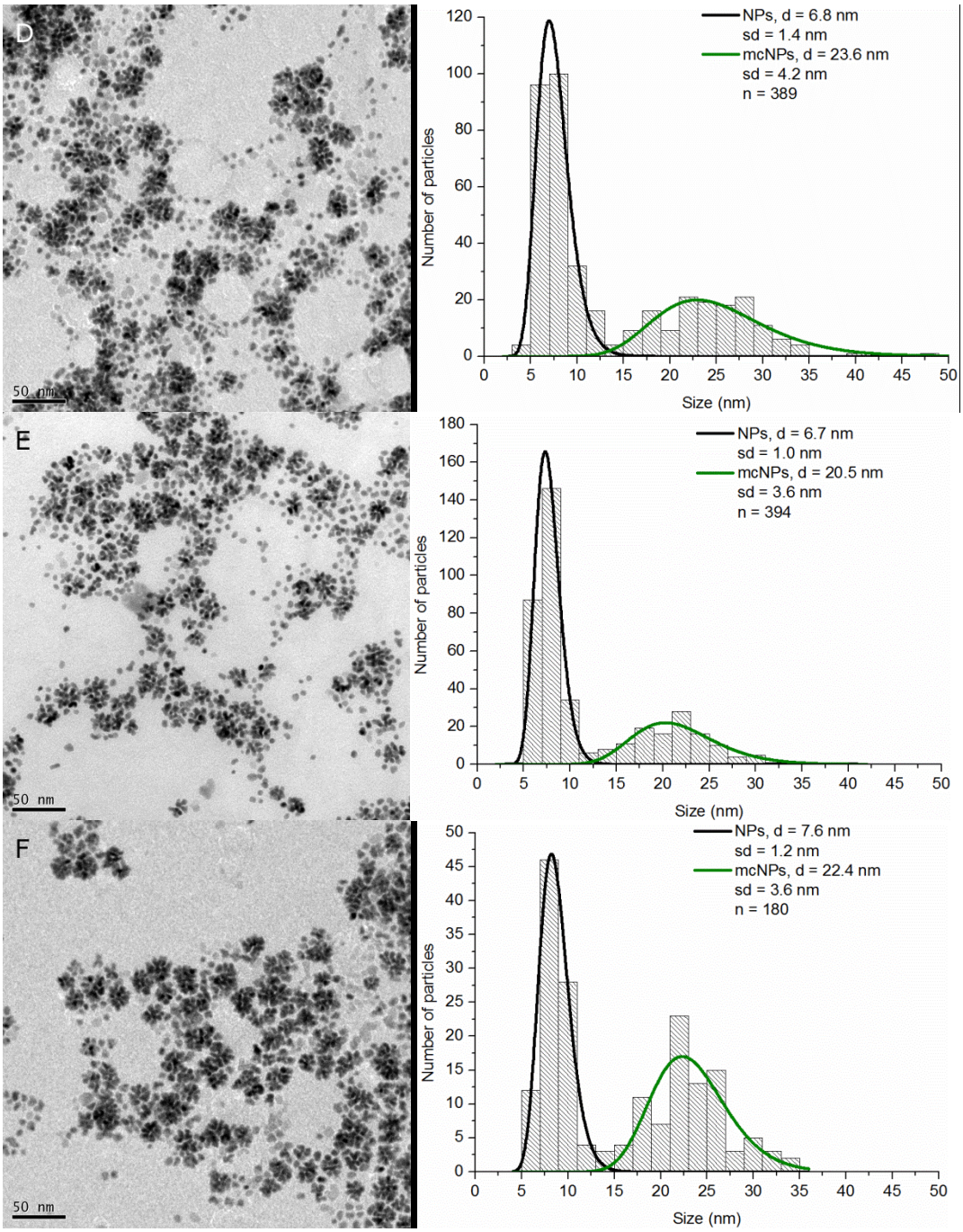


Figure S1. TEM images, samples **1 – 6** synthesized in dioctyl ether with increasing tSf; **A** 10 mmol; **B**, 12 mmol; **C**, 14 mmol; **D**, 16 mmol; **E**, 18 mmol; **F**, 20 mmol.

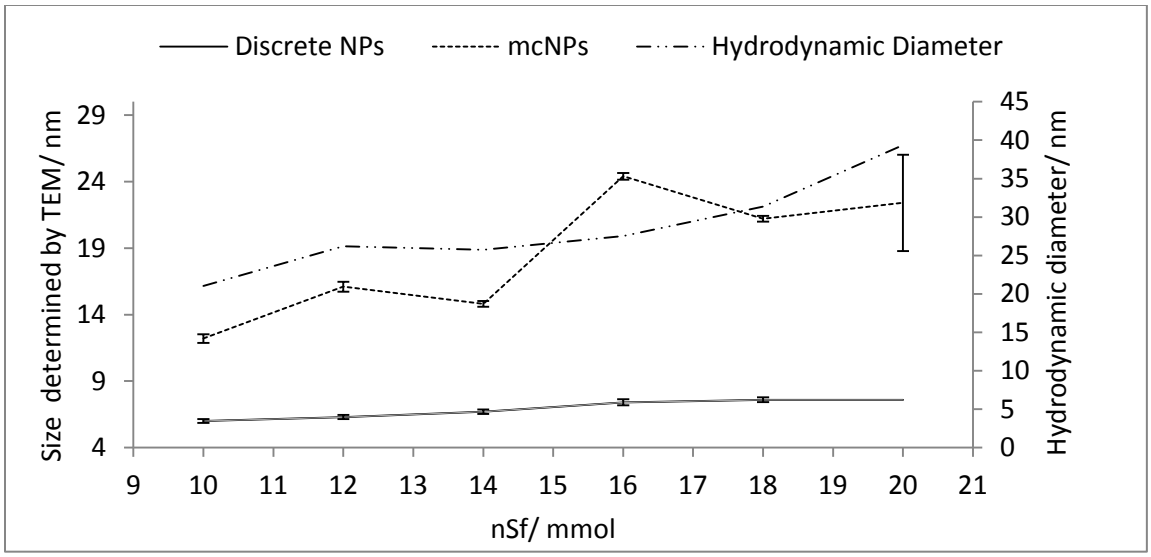


Figure S2. Samples **1** – **6**, synthesized with equal OA and OLA and increasing tSf, diameters of discrete NPs and mcNPs were measured with TEM (solid line, discrete NPs; dashed line, mcNPs); hydrodynamic diameters were measured with DLS (double dashed line) , vertical bars represent the standard deviation from mean size.

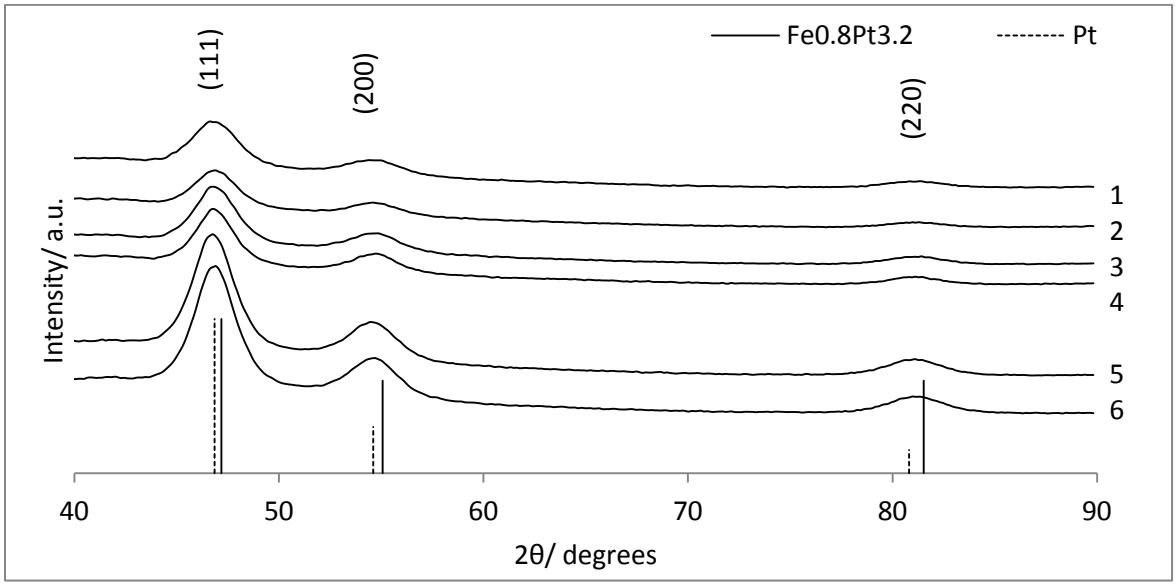
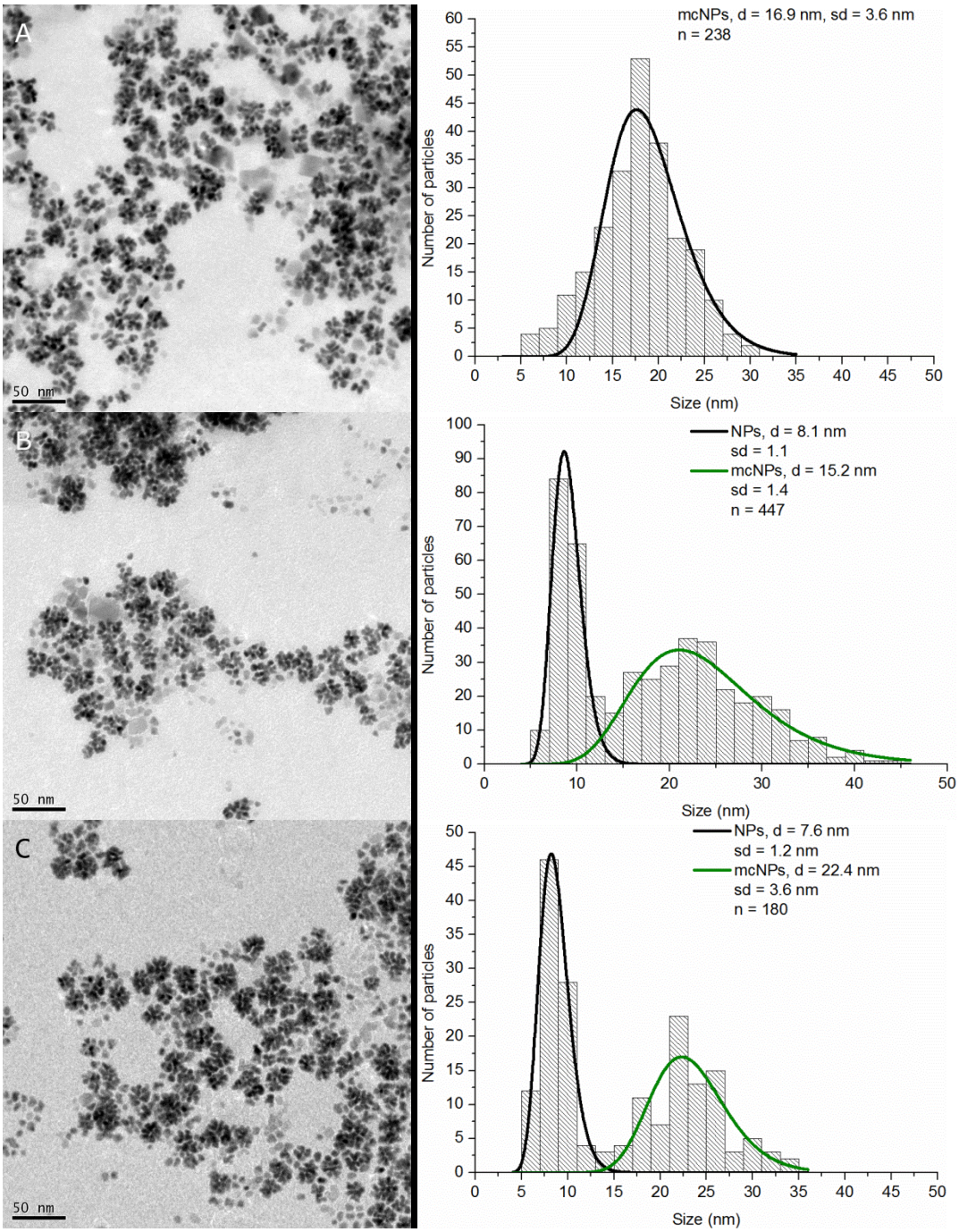


Figure S3. XRD patterns of mcNPs prepared with equal OA and OLA and increasing tSf; **1** 10 mmol; **2**, 12 mmol; **3**, 14 mmol; **4**, 16 mmol; **5**, 18 mmol; **6**, 20 mmol.



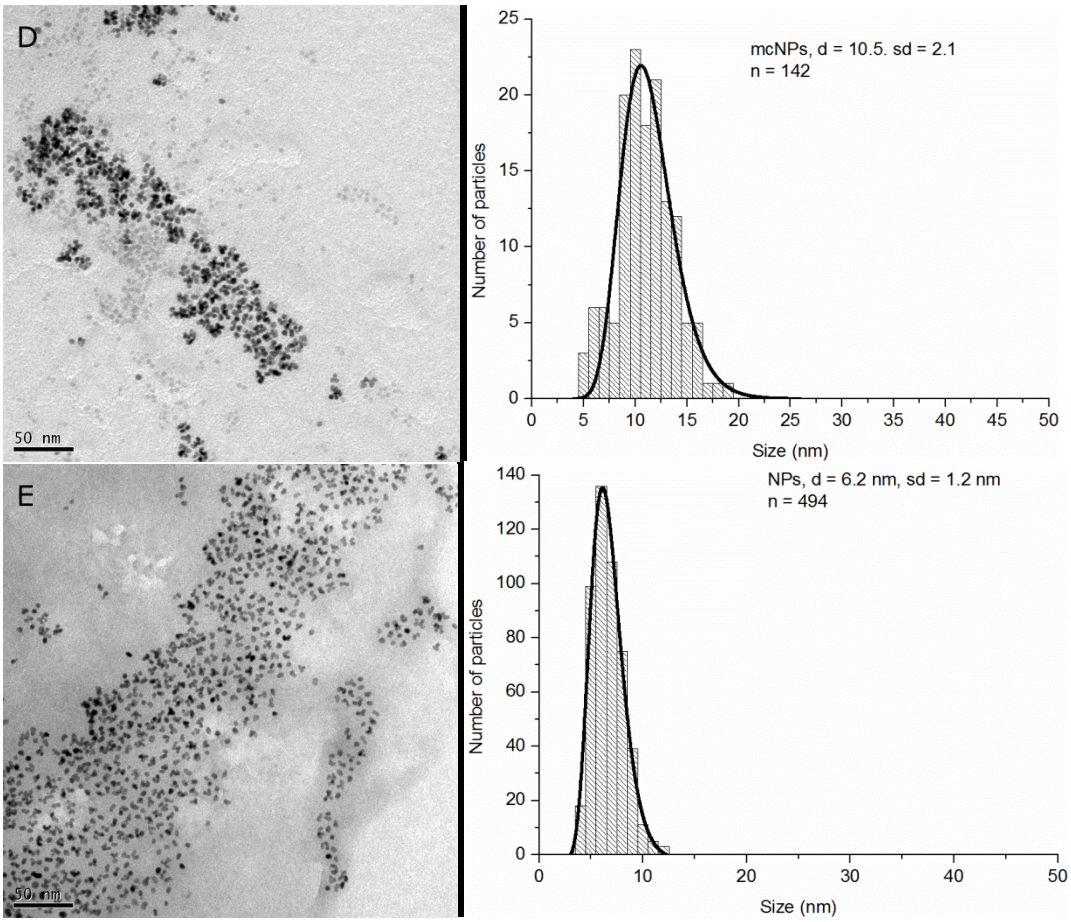


Figure S4. TEM images, samples **7**, **8**, **6**, **9**, and **10** synthesized with a constant total amount of surfactant equal to 20 mmol and increasing amount of OA; **A**, 5 mmol (25%); **B**, 6.7 mmol (33%); **C**, 10 mmol (50%); **D**, 13.3 mmol (67%); and **E**, 15 mmol (75%).

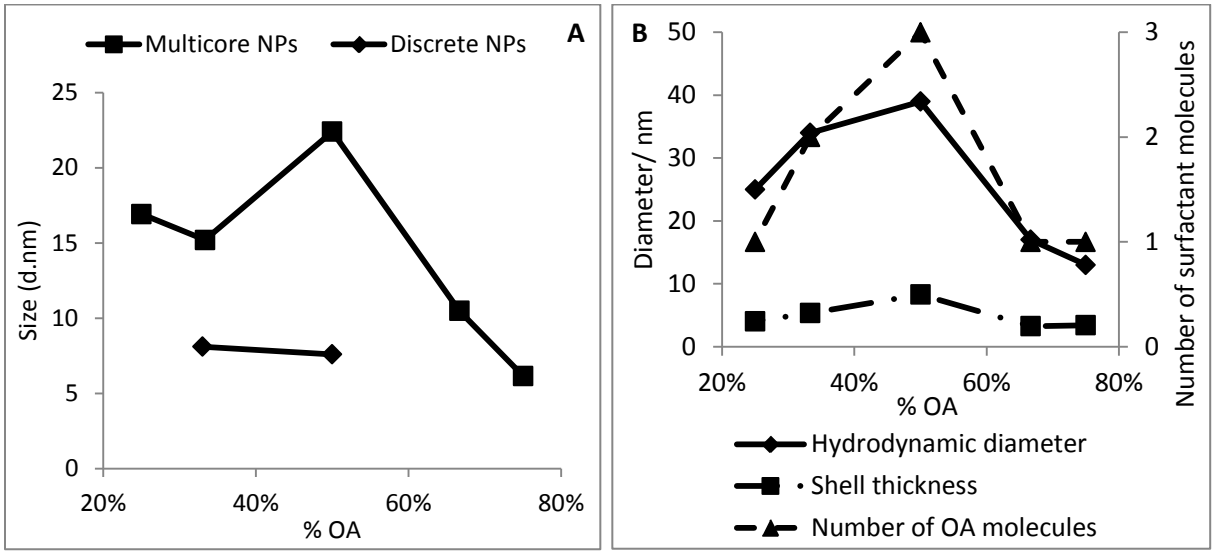


Figure S5. Trends in discrete NP and mcNP size as the amount of OA relative to OLA increases, with constant tSf set at 20 mmol, as shown by TEM, **A**; and DLS, **B**.

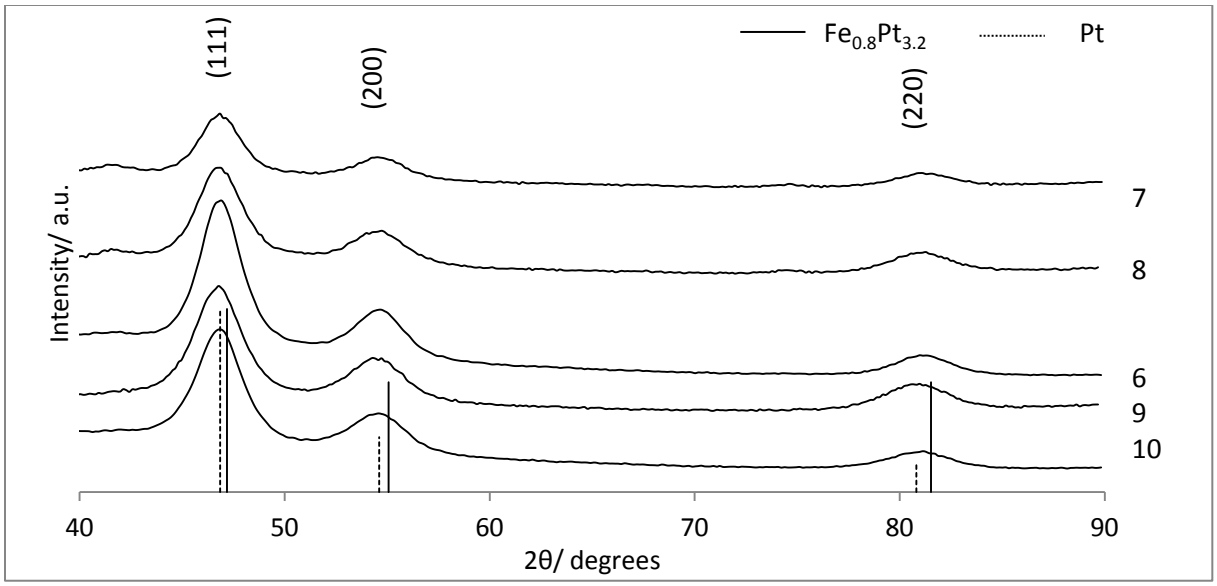
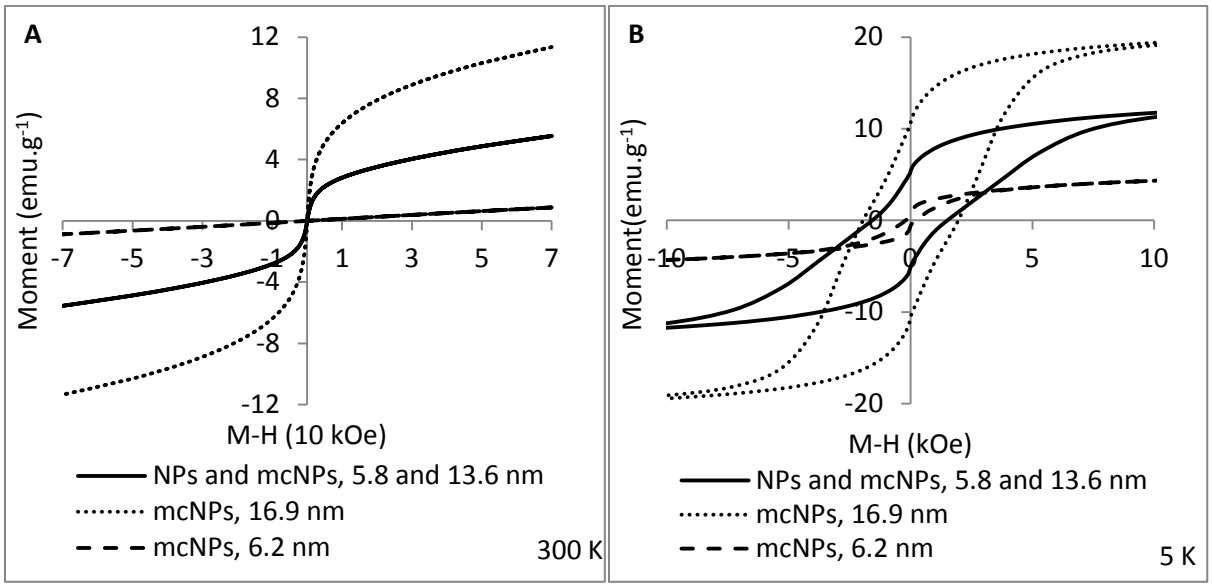


Figure S6. XRD pattern of mcNPs prepared with constant tSf equal to 20 mmol and increasing amount of OA; **7**, 5 mmol (25%); **8**, 6.7 mmol (33%; **6**, 10 mmol / (50%); **9**, 13.3 mmol (67%); and **10**, 15 mmol (75%).



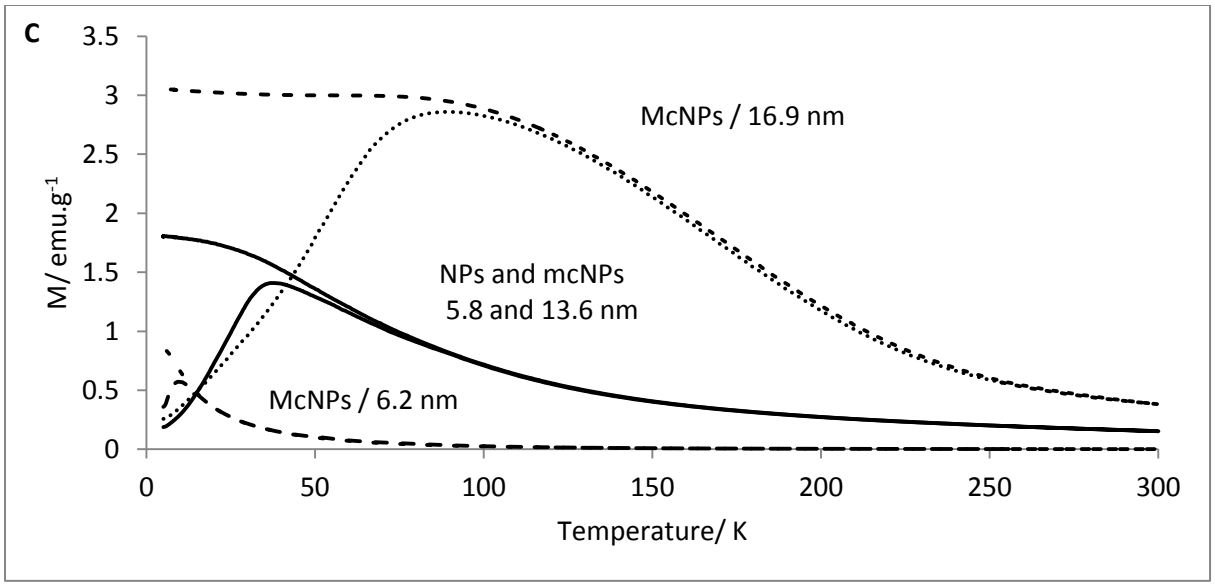
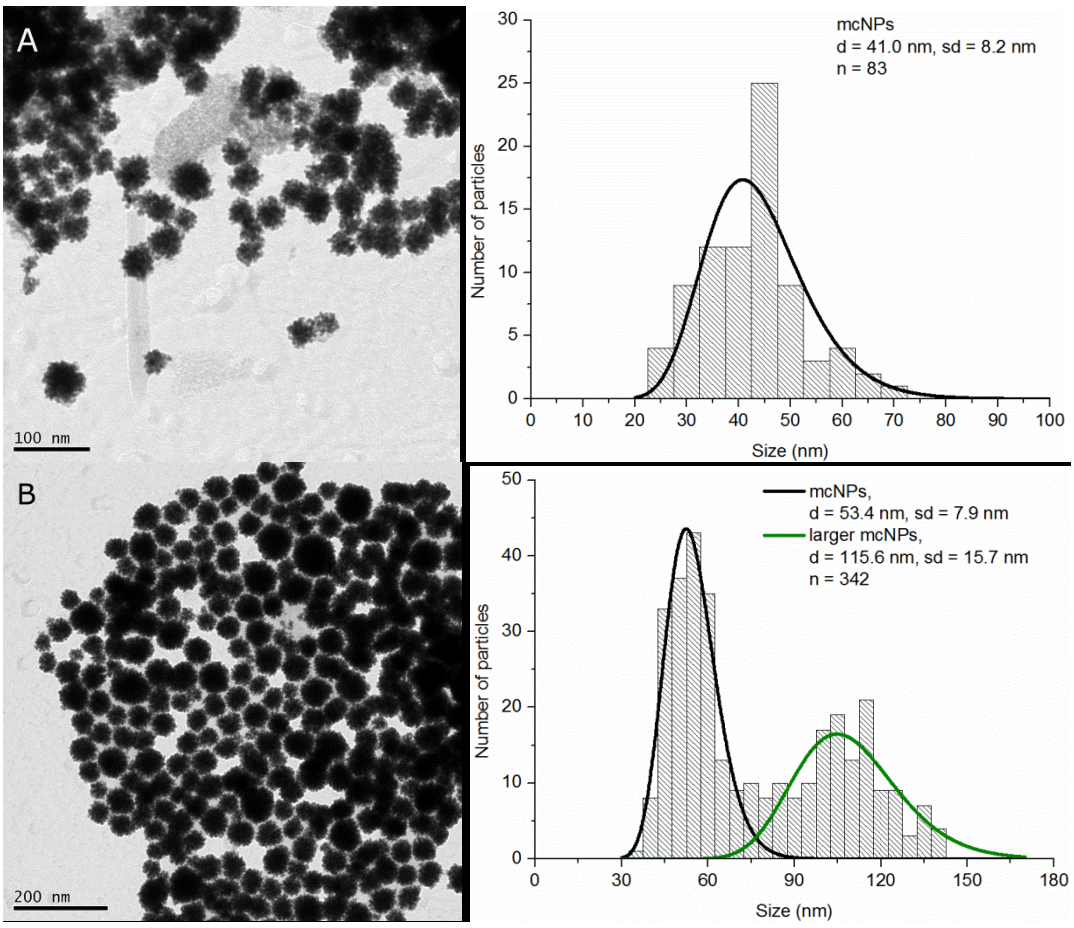


Figure S7. Hysteresis loops recorded at **A,** 300 K; **B,** 5 K; and **C,** ZFC/FC curves (100 Oe) of sample **7,** large mcNPs (d = 16.9 ± 3.6 nm) synthesized with 5 mmol (25%) OA; sample **1,** a mixture of discrete NPs and mcNPs (d = 7.6 ± 1.2 and 22.4 ± 3.6 nm) synthesized in 10 mmol (50%) OA; and sample **10,** small mcNPs (d = 6.2 ± 1.2 nm) 15 mmol (75%) of OA.



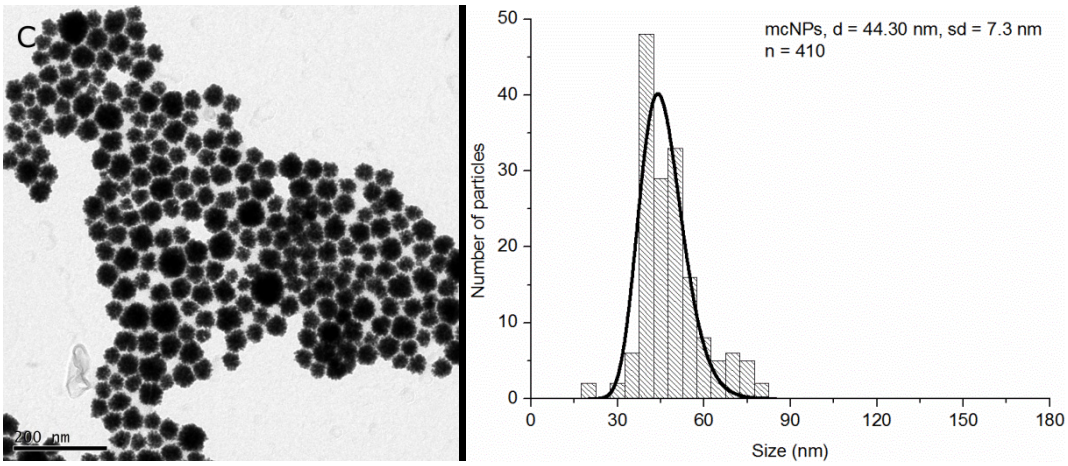


Figure S8. TEM images **A-C** of samples **12-14**, mcNPs synthesized in dioctyl ether with an amount ratio of OA: OLA equal to 1: 6 and increasing tSf: **A**, 17.5 mmol; **B**, 24.5 mmol; **C**, 31.5 mmol.

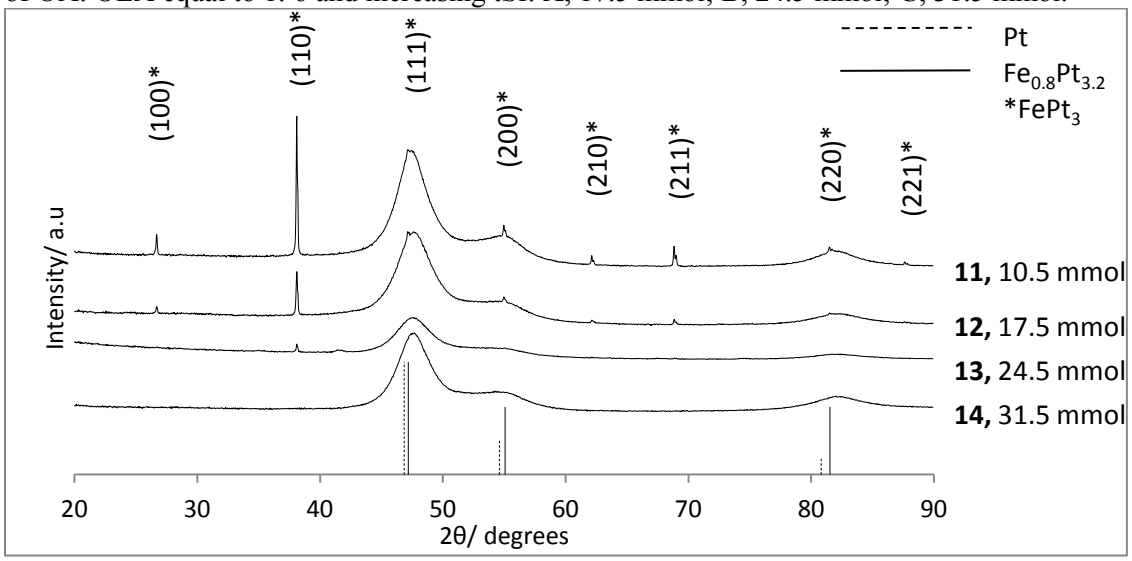


Figure S9. XRD patterns of samples prepared with an amount ratio of OA: OLA equal to 1: 6 and increasing tSf; **11**, 10.5 mmol; **12**, 17.5 mmol; **13**, 24.5 mmol; **14**, 31.5 mmol.

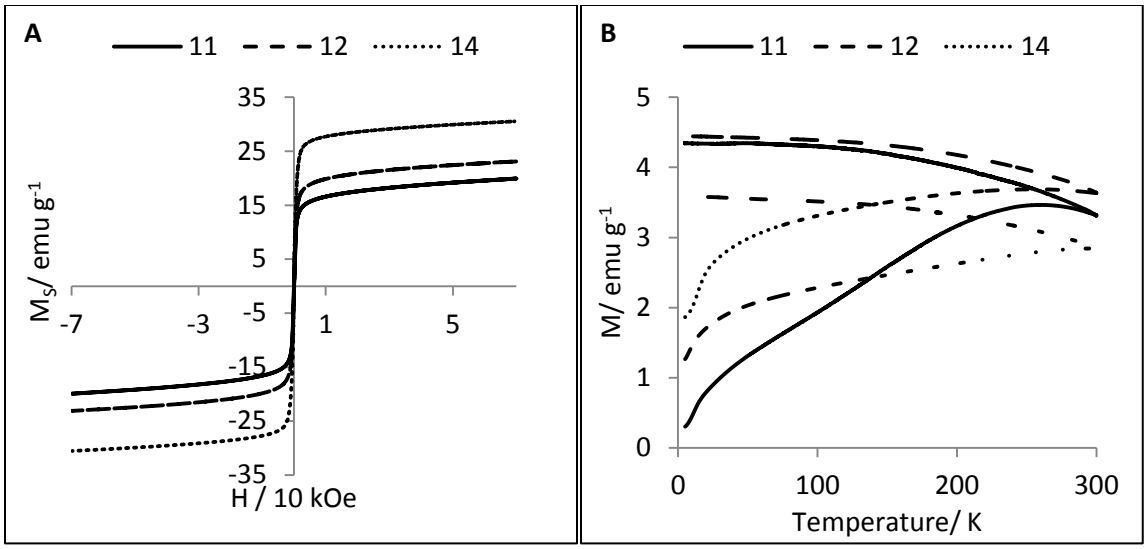


Figure S10. **A,** hysteresis loops at 300 K and **B**, ZFC/FC (100 Oe) curves of samples prepared in dioctyl ether with constant OA to OLA amount ratio of 1: 6 and increasing tSf; **11**, 10.5 mmol; **12**, 17.5 mmol; **14**, 31.5 mmol.

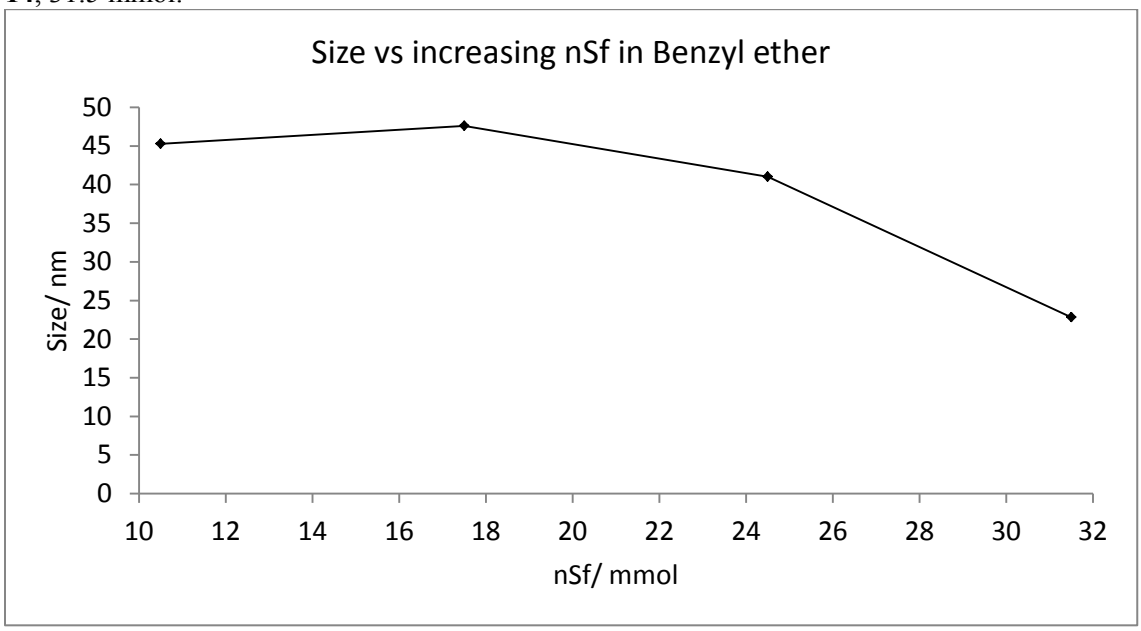


Figure S11. TEM (**20** – **23**) average sizes of mcNPs synthesized in dibenzyl ether with constant amount ratio of OA: OLA equal to 1: 6 and increasing tSf.

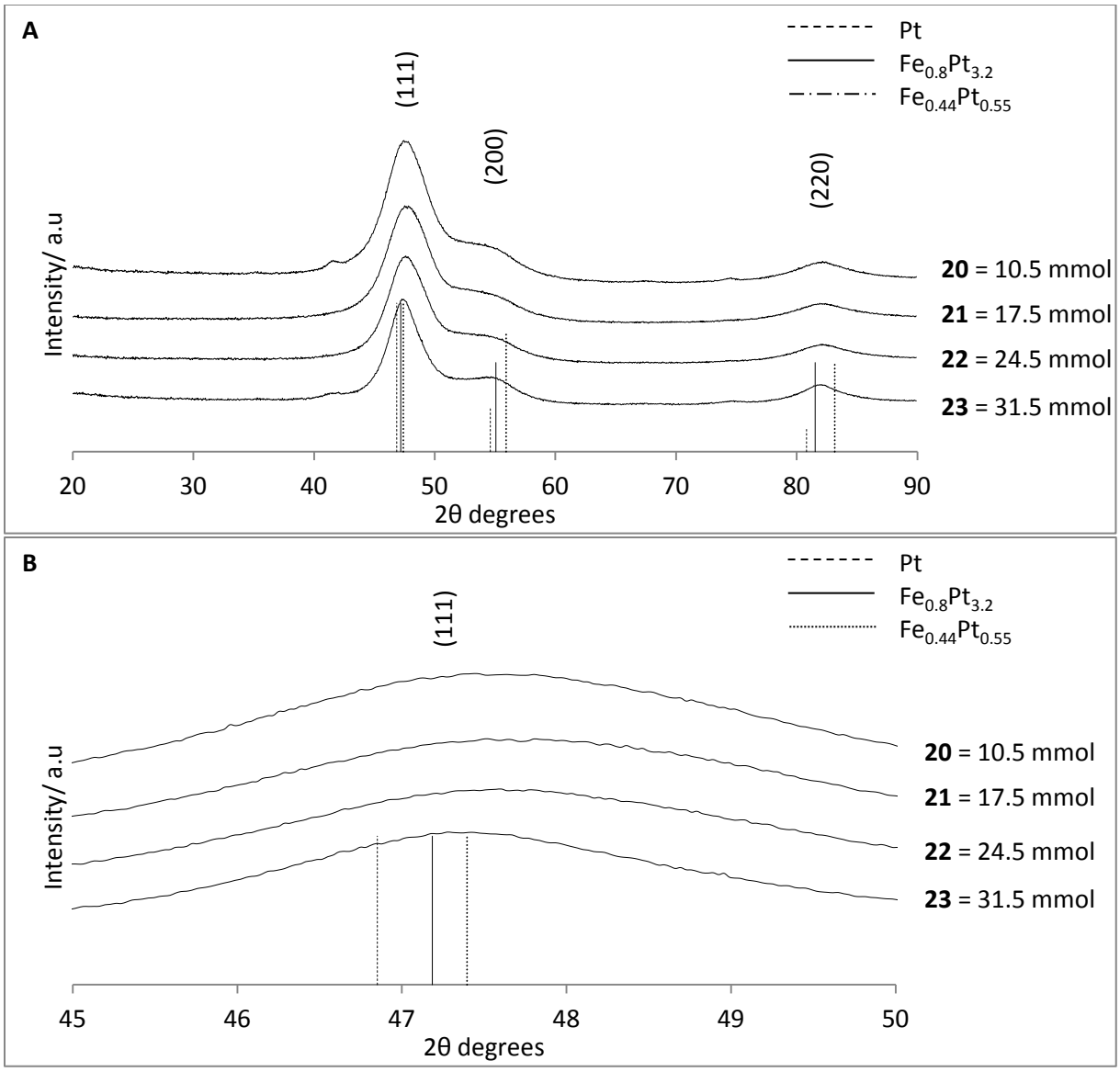


Figure S12. XRD patterns of samples **20** to **23**; **A**, 20 – 90 degrees 2θ; **B**, 45 -50 degrees 2θ; products synthesized in dibenzyl ether with an amount ratio of OA: OLA equal to 1: 6 and increasing tSf; **20**, 10.5 mmol; **21**, 17.5 mmol; **22**, 24.5 mmol; **23**, 31.5 mmol.

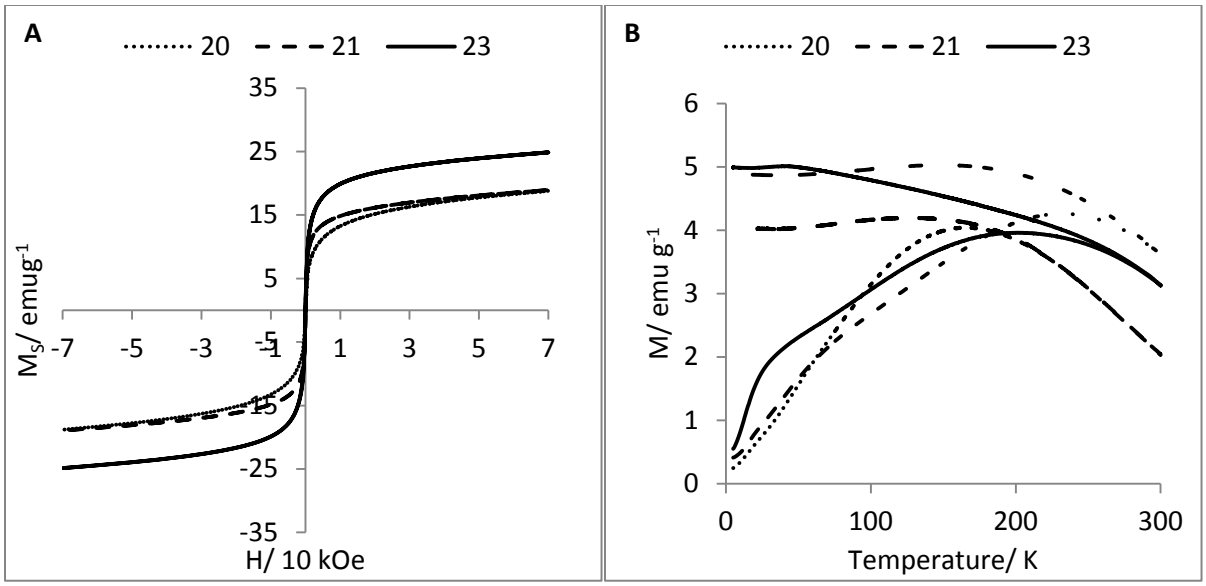


Figure S13. Hysteresis loops at 300 K, **A**; and ZFC/FC (100 Oe) curves of samples prepared with a constant amount ratio of OA: OLA equal to 1: 6 and increasing tSf; **20**, 10.5 mmol; **21**, 17.5 mmol; **23**, 31.5 mmol.

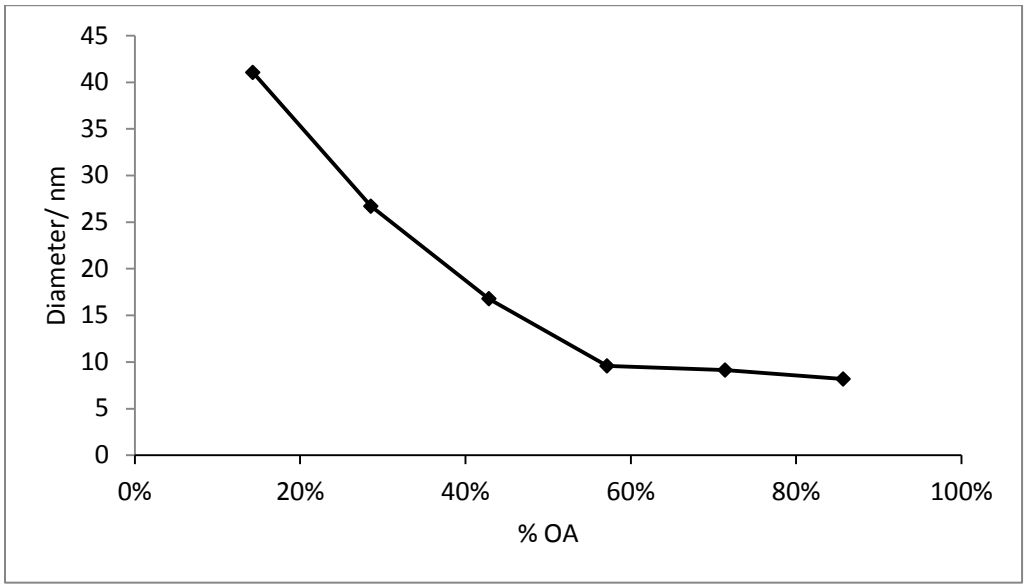


Figure S14. Samples **11** – **14**, fraction of OA used vs size at constant tSf in dioctyl ether.

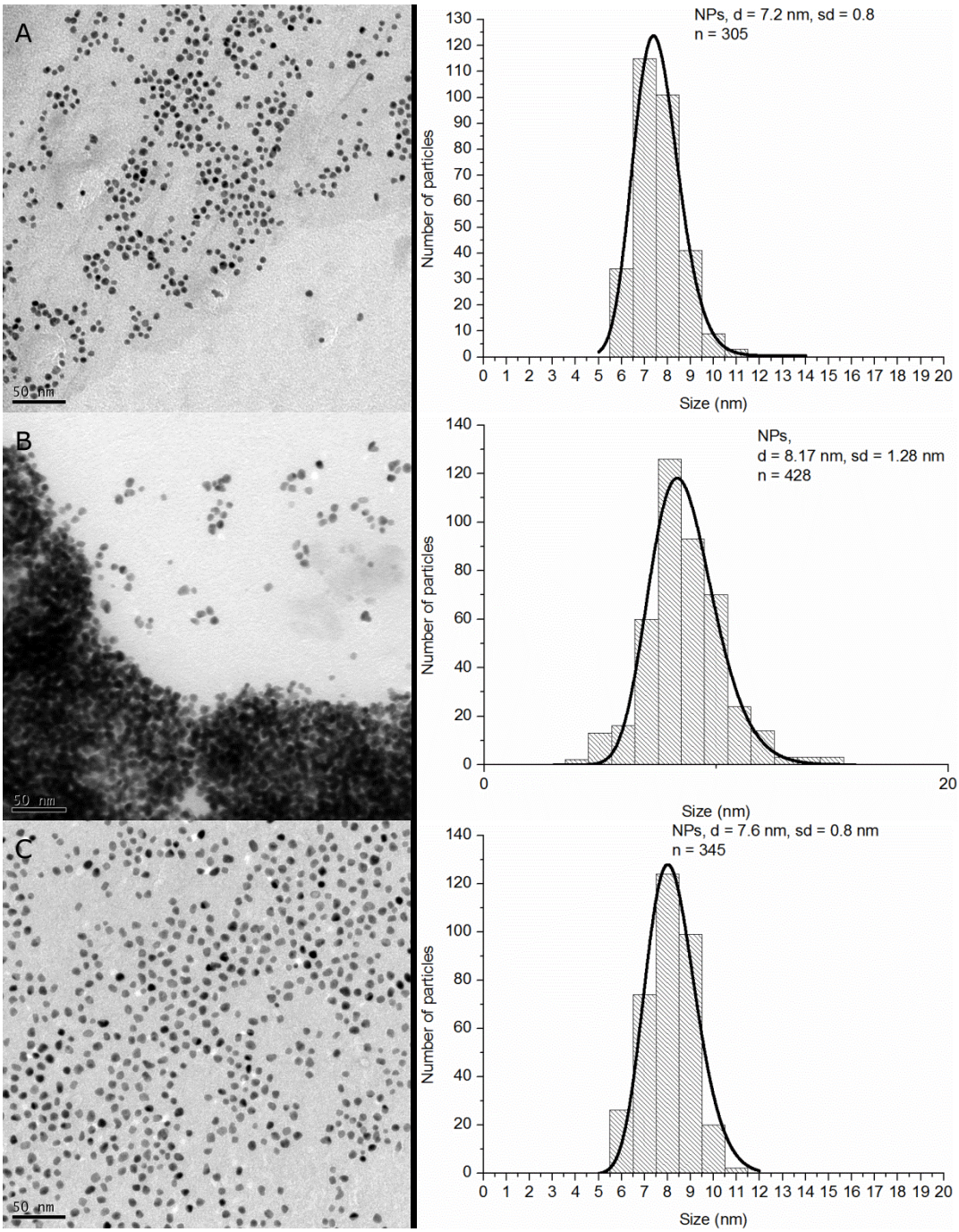


Figure S15. TEM images of discrete NPs (samples **29**, **19**, and **30)** synthesized in dioctyl ether with a constant amount ratio of OA: OLA equal to 6: 1 and increasing tSf; **A**, 10.5 mmol; **B**, 17.5 mmol; **C**, 31.5 mmol.

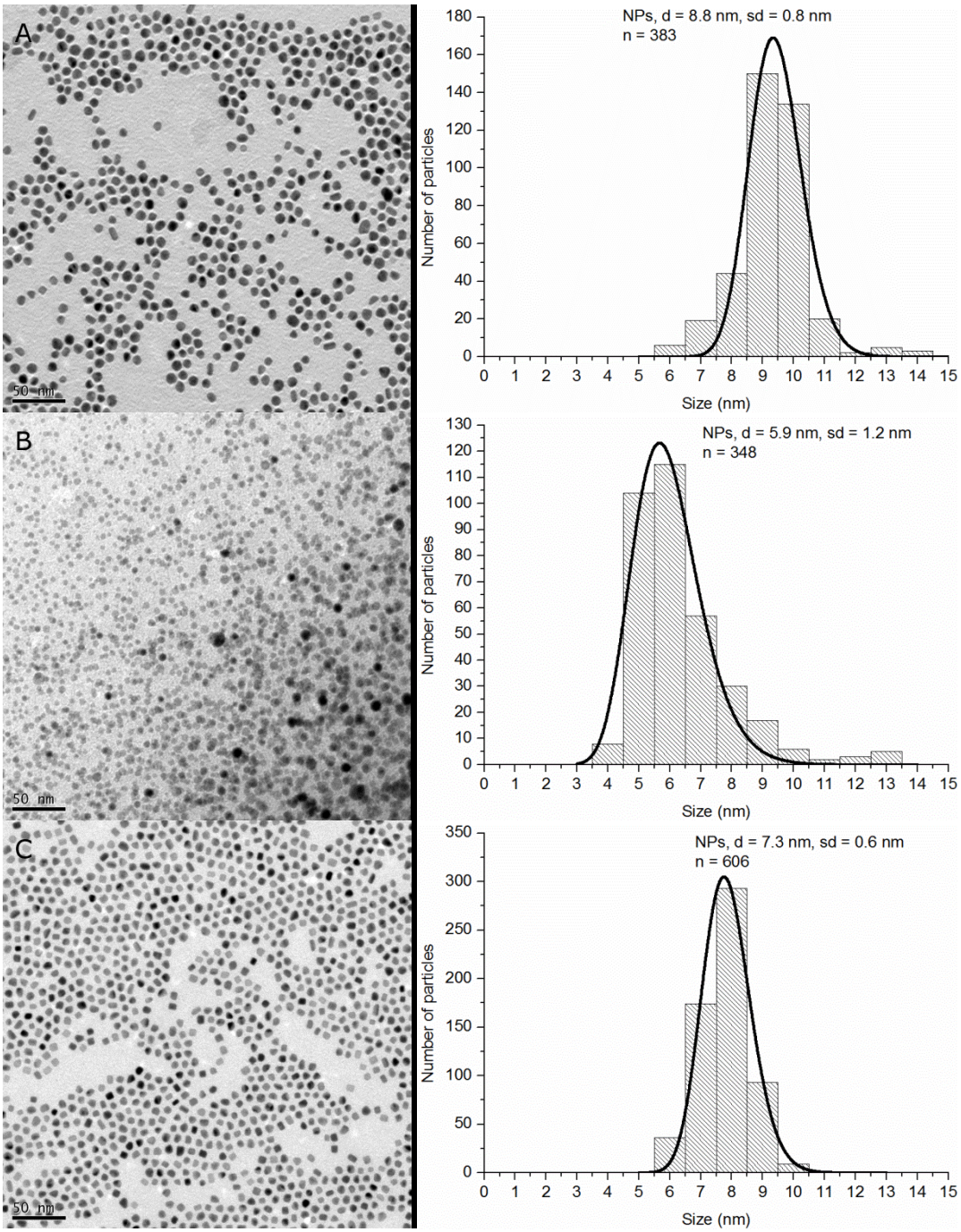


Figure S16. TEM images of discrete NPs (samples **31**, **28**, and **32)** synthesized in dibenzyl ether with an amount ratio of OA: OLA equal to 6: 1 and increasing tSf; **A**, 10.5 mmol; **B**, 17.5 mmol; **C**, 31.5 mmol.

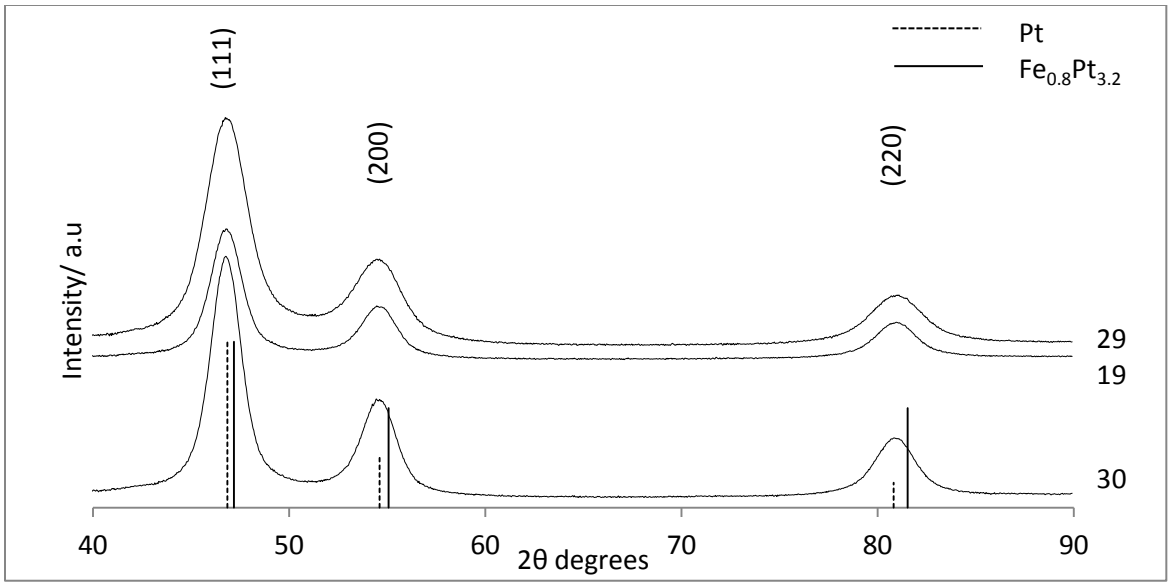


Figure S17. XRD patterns of discrete NPs synthesized in dioctyl ether with an amount ratio of OA: OLA equal to 6: 1 and increasing tSf; **29**, 10.5 mmol; **19**, 17.5 mmol; **30**, 31.5 mmol.

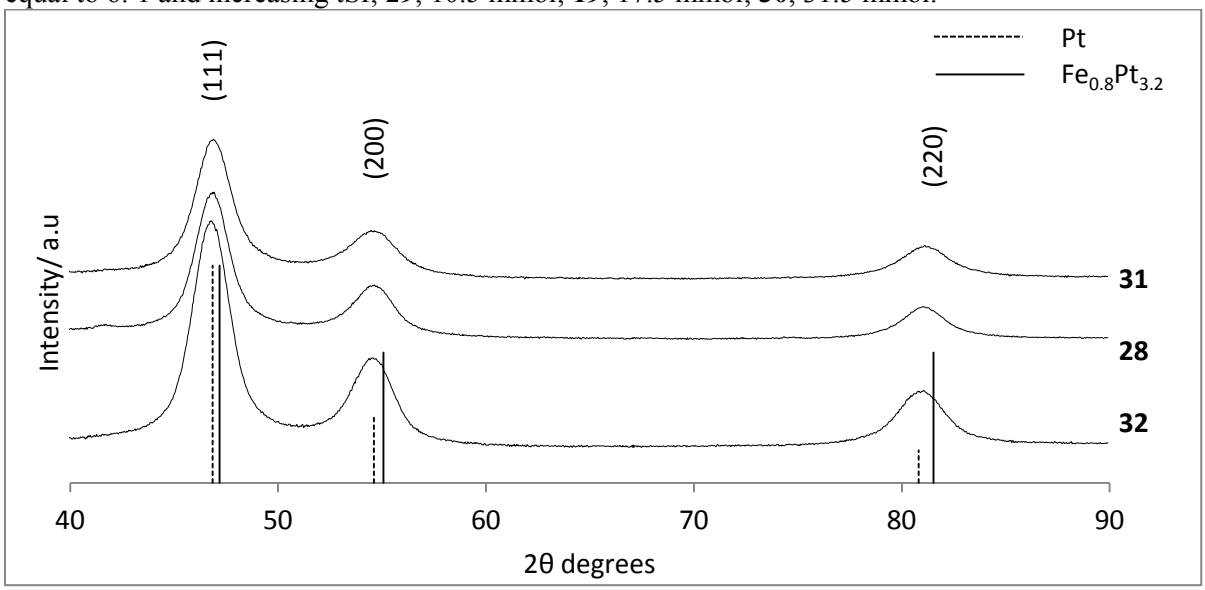


Figure S18. XRD patterns of discrete NPs synthesized in dibenzyl ether with a constant amount ratio of OA: OLA equal to 6: 1 and increasing tSf; **31**, 10.5 mmol; **28**, 17.5 mmol; **32**, 31.5 mmol.

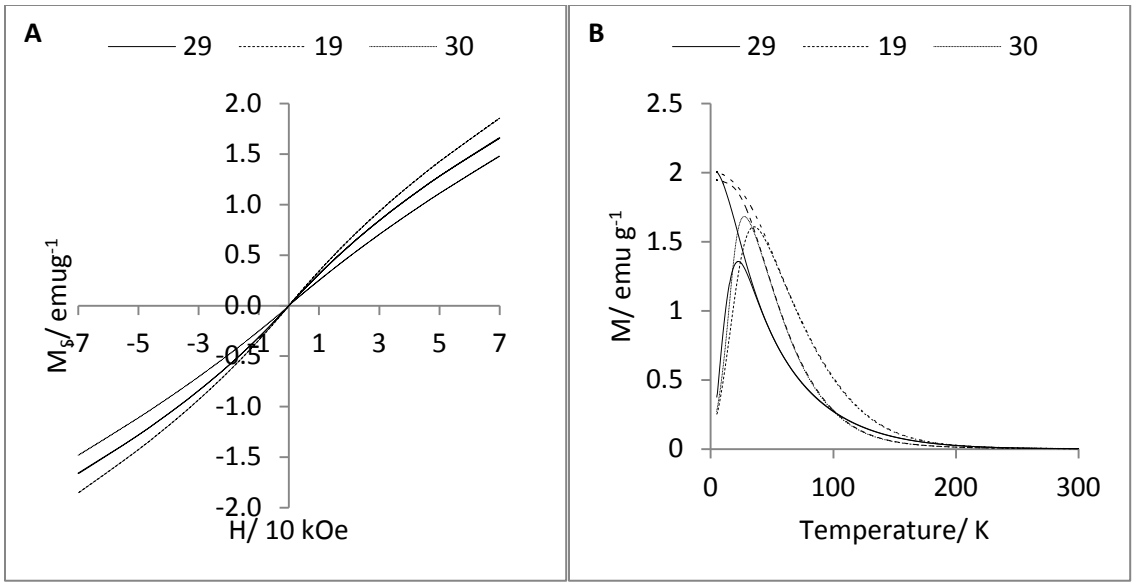


Figure S19. **A**, hysteresis loops at 300 K and **B**, ZFC/FC (100 Oe) curves of discrete NPs synthesized in dioctyl ether with an amount ratio of OA: OLA equal to 6: 1 and increasing tSf; **29**, 10.5 mmol; **19**, 17.5 mmol; **30**, 31.5 mmol.

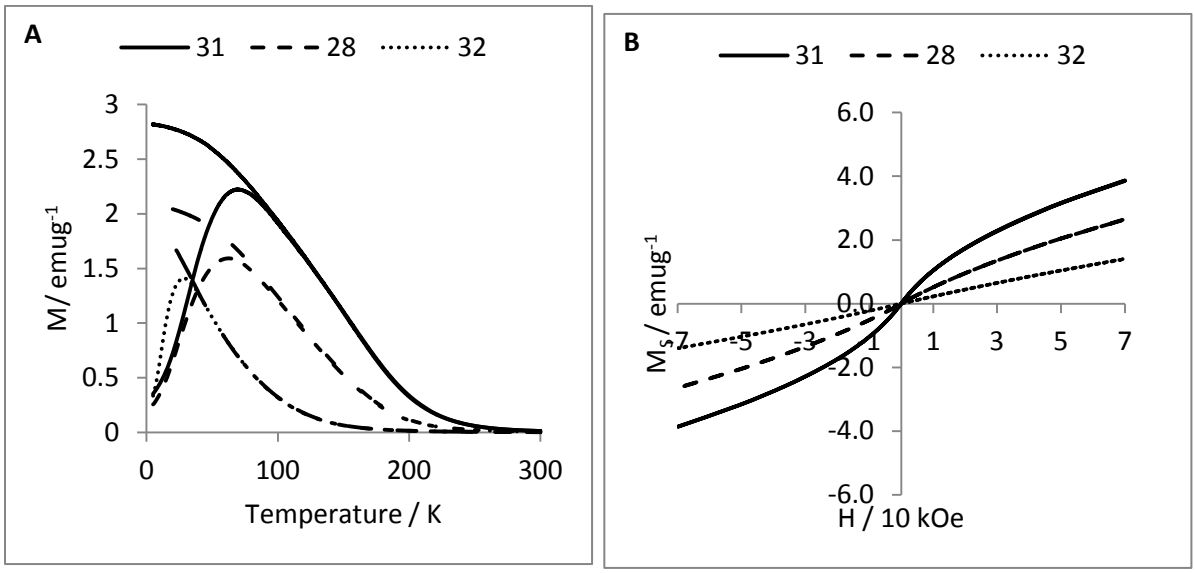
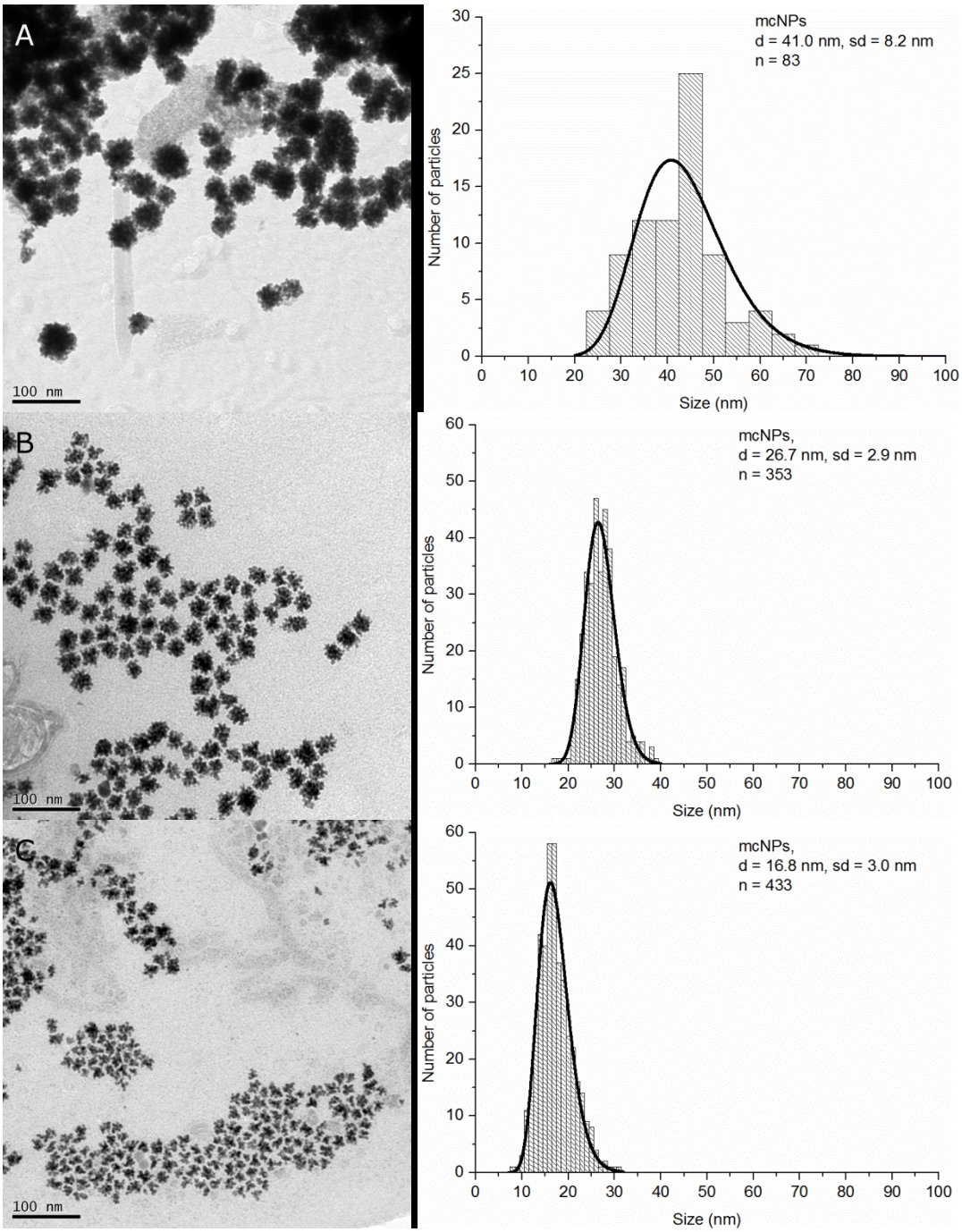


Figure S20. **A**, ZFC/FC curves (100 Oe) and **B**, hysteresis loops (300 K) of discrete NPs synthesized in dibenzyl ether with an amount ratio of OA: OLA equal to 6: 1 and increasing tSf; **31**, 10.5 mmol; **28**, 17.5 mmol; **32**, 31.5 mmol.



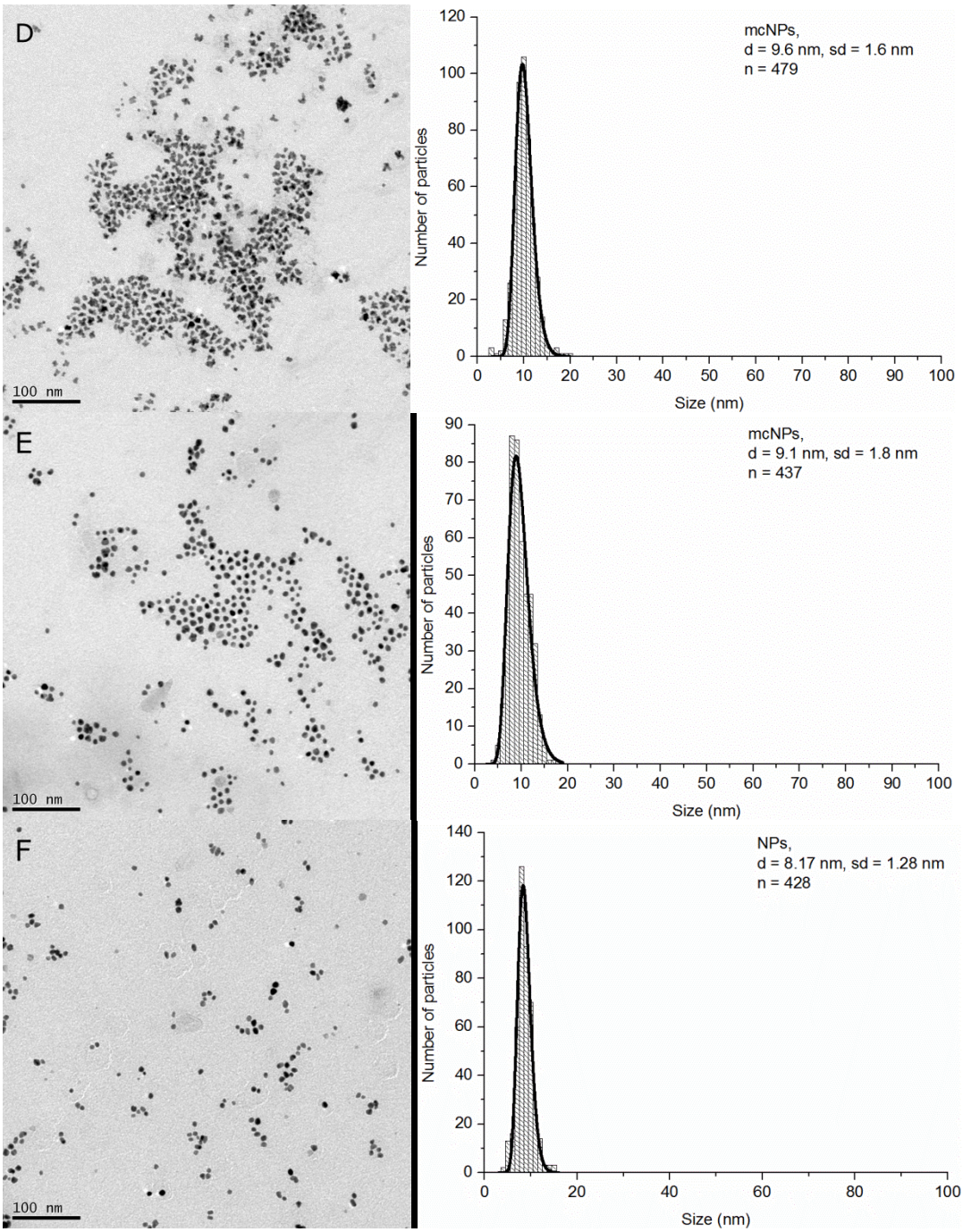


Figure S21. TEM images of mcNPs and discrete NPs synthesized in dioctyl ether with an increasing amount of OA and constant tSf (17.5 mmol, samples **12** and **15-19)**; **A**, 2.5 mmol (14%); **B**, 5 mmol (29%); **C**, 7.5 mmol (43%); **D**, 10 mmol (57%); **E**, 12.5 mmol (71%); **F**, 15 mmol (86%).

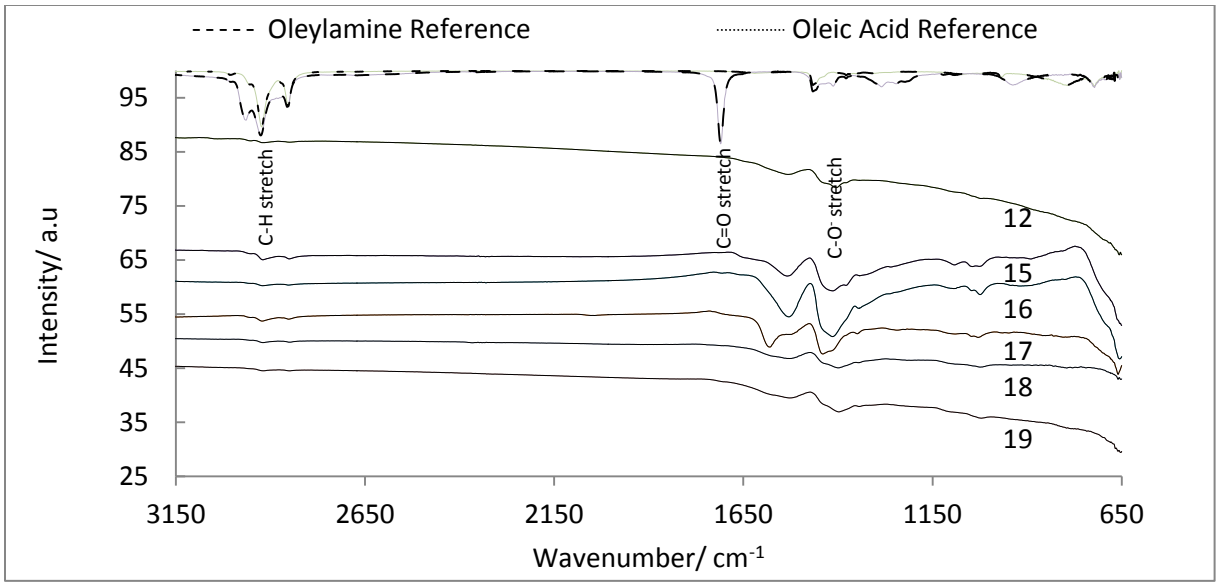
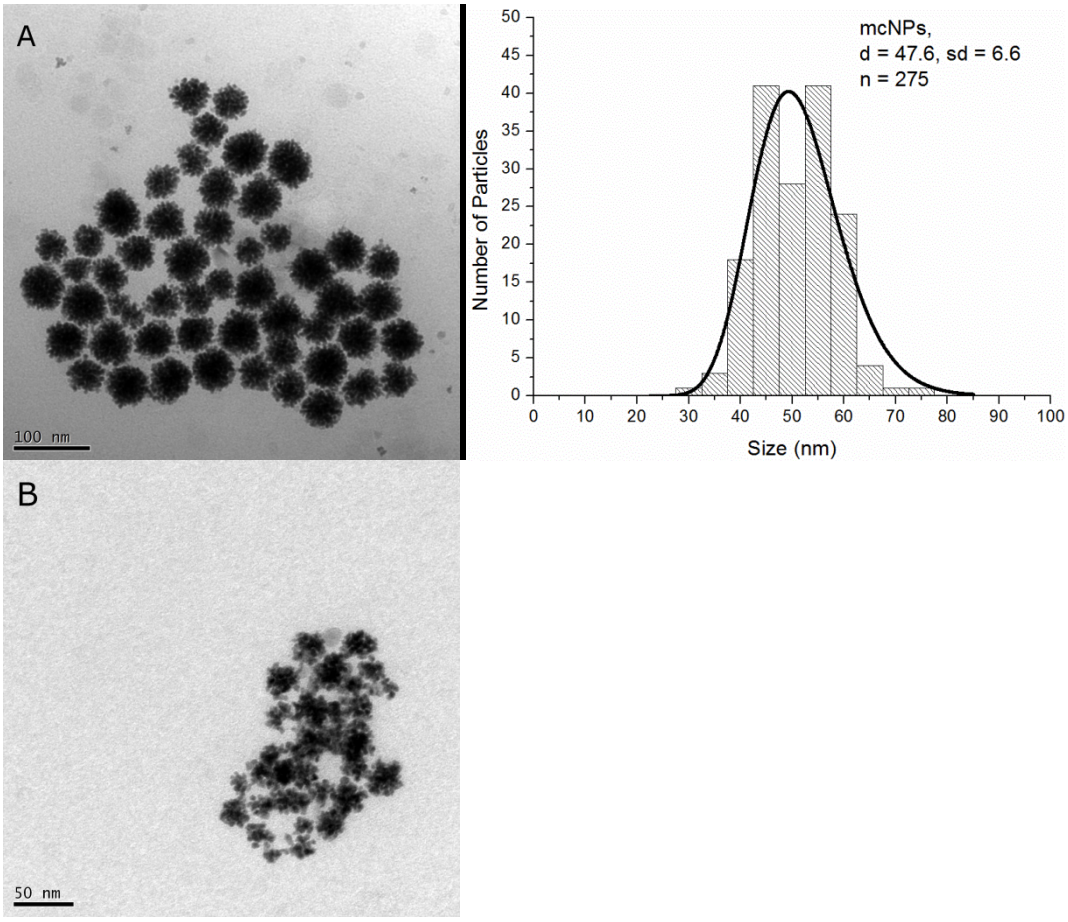


Figure S22. FTIR, mcNPs prepared with constant tSf (17.5 mmol) and increasing amount of OA; **12**, 2.5 mmol; **15**, 5 mmol; **16**, 7.5 mmol; **17**, 10 mmol; **18**, 12.5 mmol; **19**, 15 mmol.



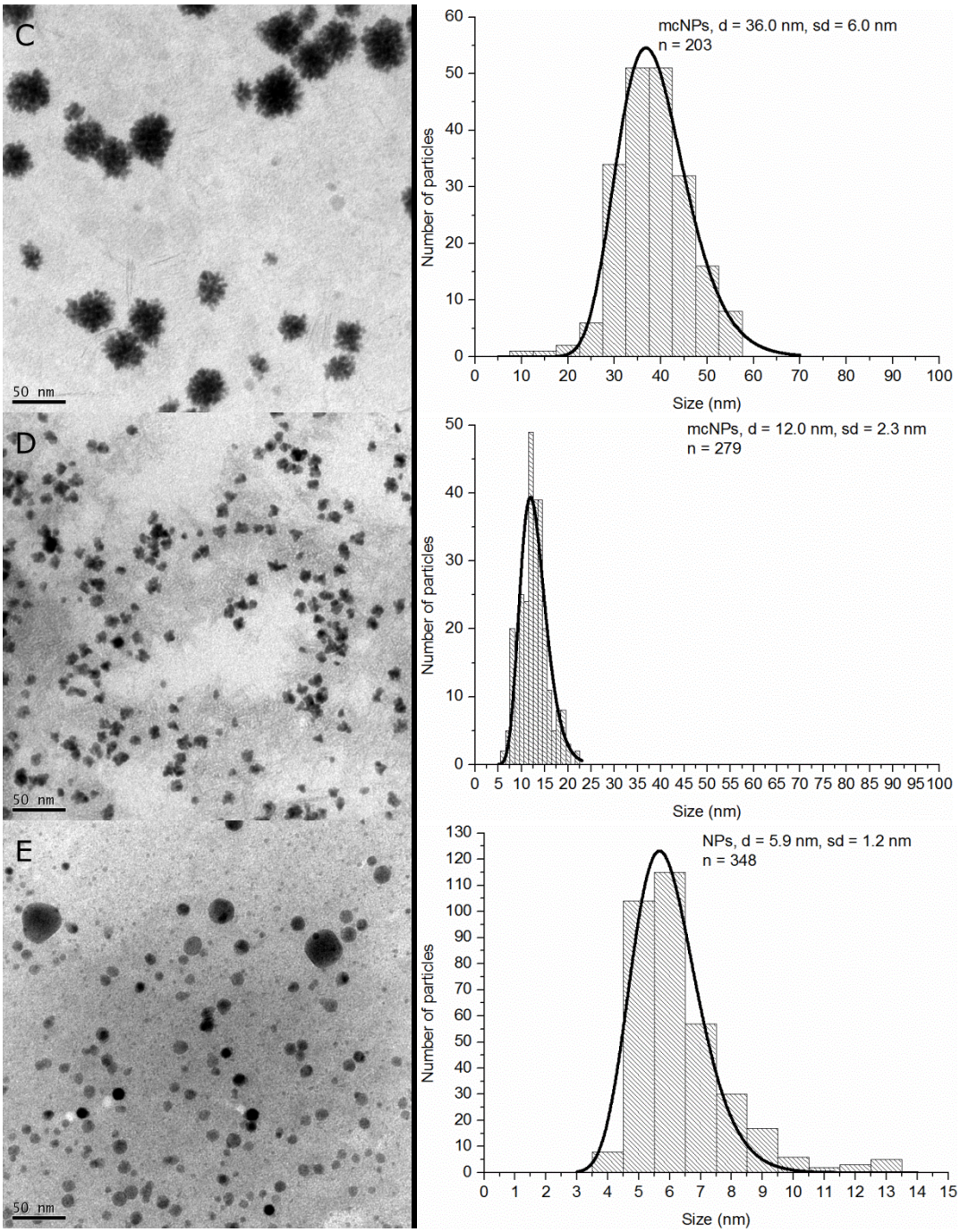


Figure S23. TEM images of mcNPs prepared in dibenzyl ether with constant tSf (17.5 mmol) and increasing amount of OA (samples **21** and **24-26** and **28)**; **A**, 2.5 mmol / (14%); **B**, 5 mmol (29%); **C**, 7.5 mmol (43%); **D**, 10 mmol (57%); **E**, 15 mmol (86%).

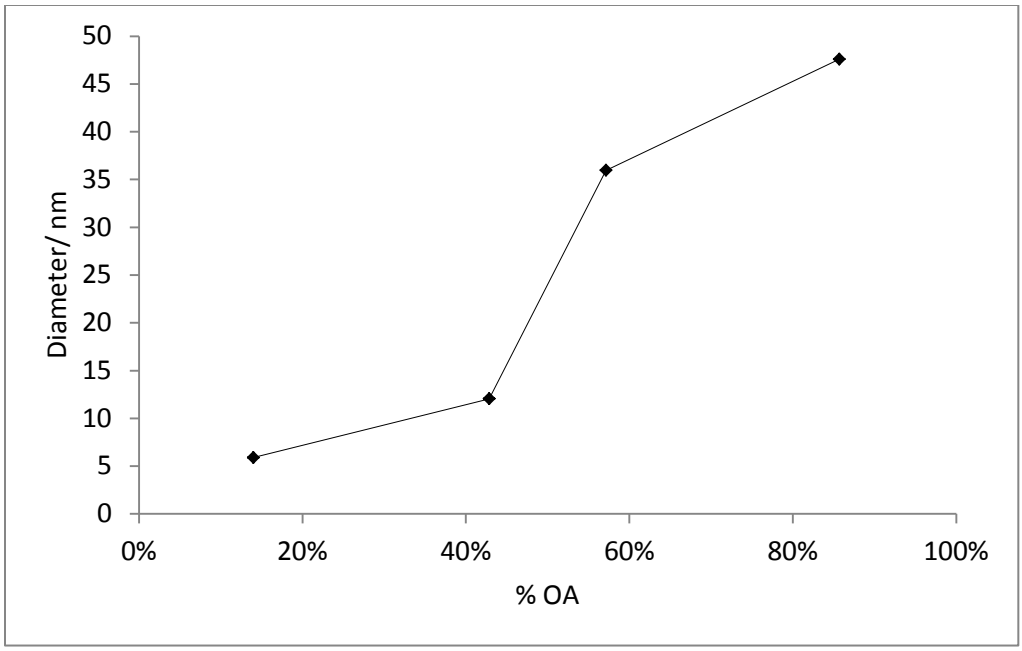


Figure S24. Samples **21**, **25**, **26**, **28**, fraction of OA used vs size determined by TEM in dibenzyl ether.

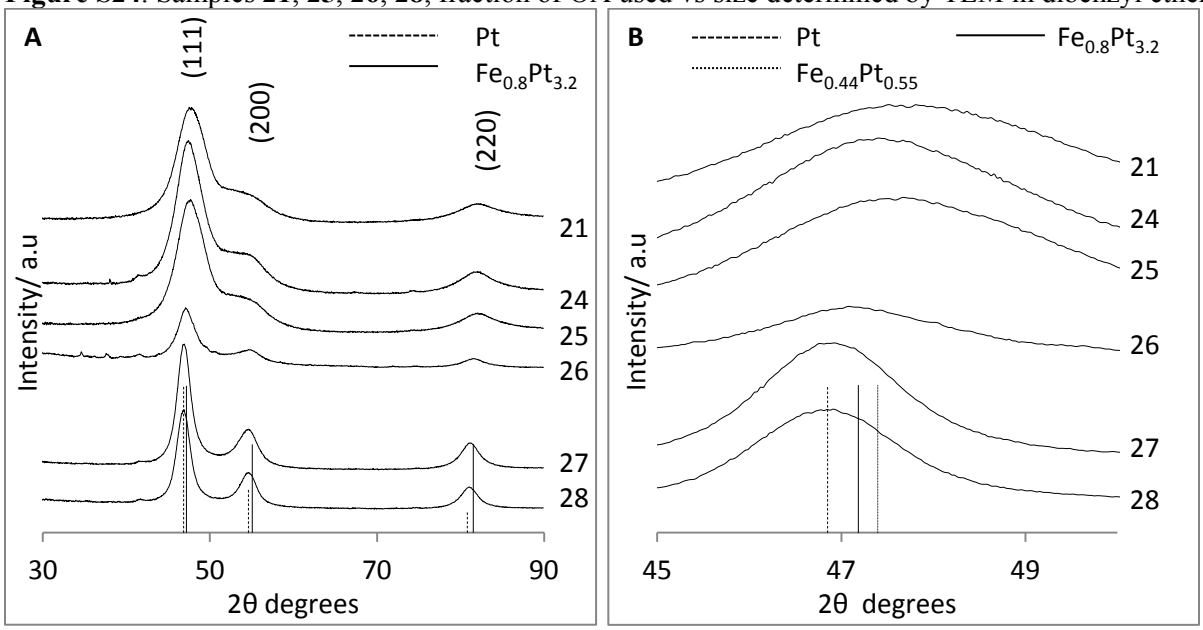


Figure S25. XRD patterns of mcNPs and discrete NPs synthesized in dibenzyl ether and increasing OA amount; **21**, 2.5 mmol (14%); **24**, 5 mmol (28%); **25**, 7.5 mmol (43%); **26**, 10 mmol (57%); **27**, 12.5 mmol (71%); **28**, 15 mmol (86%). **A** illustrates full diffraction pattern; **B** illustrates range 45 – 50 2θ.

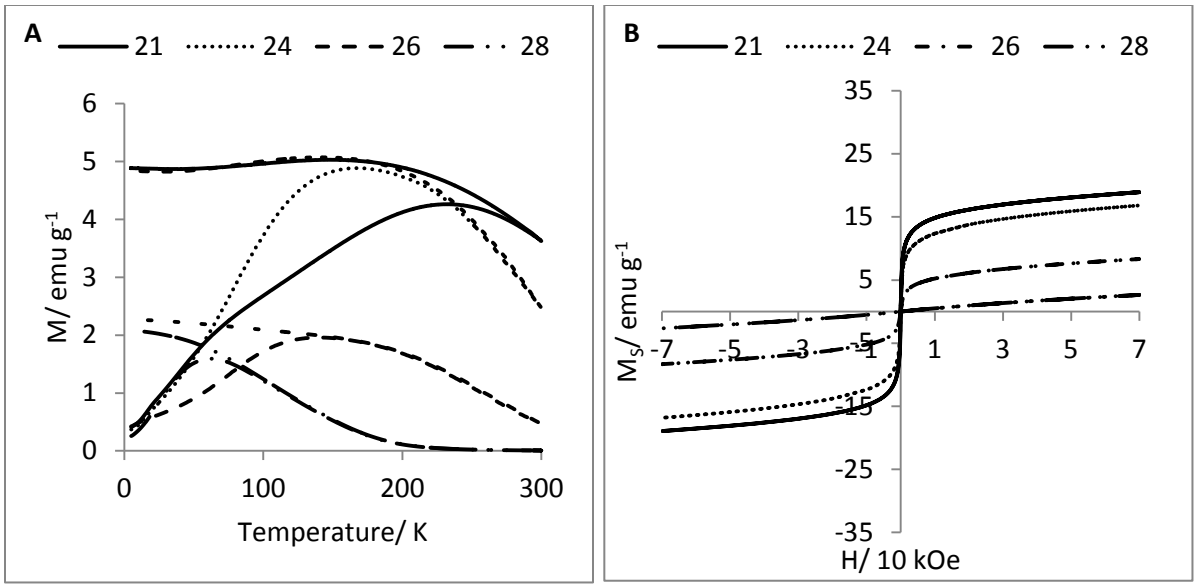


Figure S26. **A**, ZFC/FC (100 Oe) curves and **B**, hysteresis loops at 300 K of samples prepared in dibenzyl ether with constant tSf (17.5 mmol) and increasing amount of OA; **21**, 2.5 mmol (14%); **24**, 5 mmol (28%); **26**, 10 mmol (57%); **28**, 15 mmol (86%).

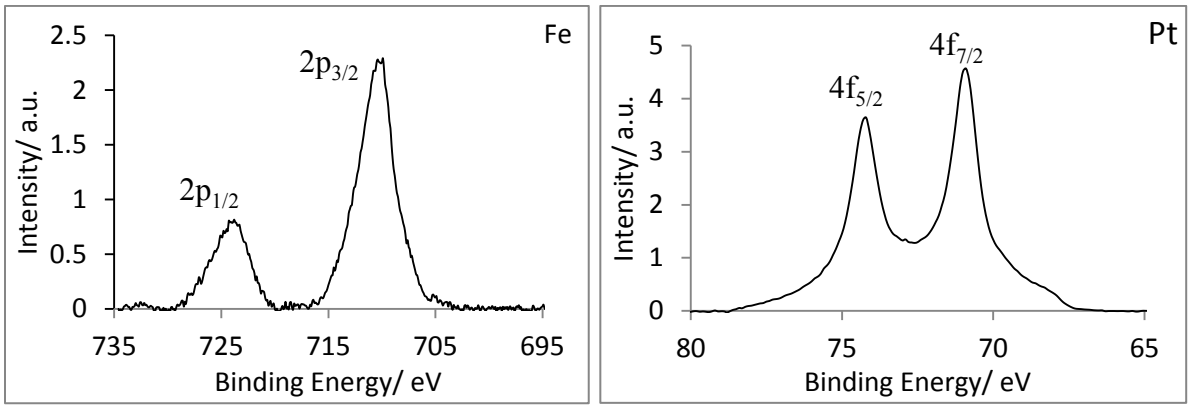


Figure S27. Typical XPS emission spectra for Fe 2p and Pt 4f orbitals respectively.

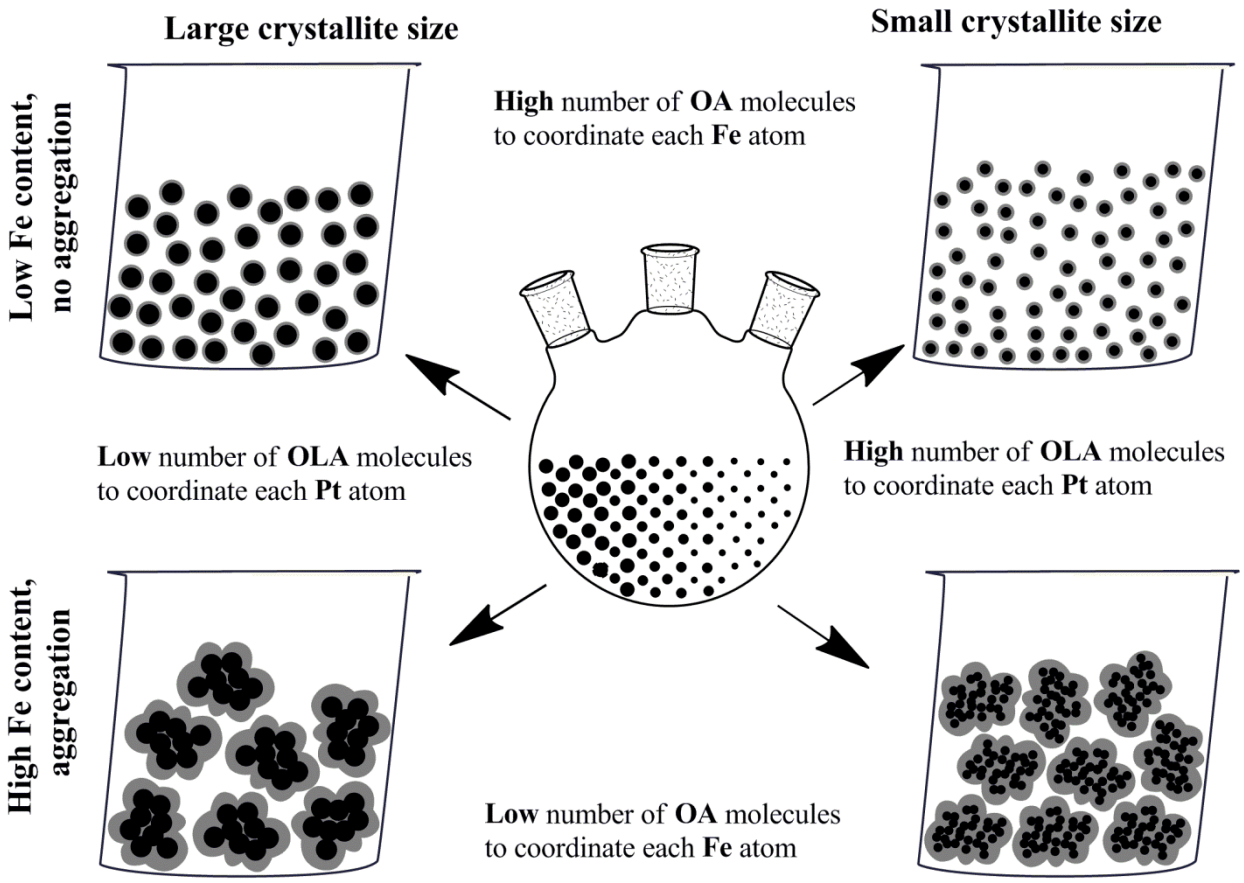


Figure S 28. FePt discrete NPs and mcNPs, varying in composition with the relative amounts of OA and OLA used.