Electronic Supplementary Material (ESI) for RSC Advances
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1 Supplementary information

2 Experimental

Chemicals: Disodium tetracarbonylferrate dioxane complex [$Na_2Fe(CO)_4$, 100%], oleic acid [OA, 99%], oleylamine [OLA, 70%] and dioctyl ether [99%] were purchased from Sigma Aldrich, UK LLC. Platinum (II) acetylacetonate [$Pt(acac)_2$, 98%] was purchased from ACROS organics. Dioctyl ether was degassed by bubbling N_2 through it overnight, all other reagents were used without further purification or treatment.

Two methods were employed, one under flowing N_2 on a Schlenk line and the other in a sealed Parr 4744 autoclave. The ratio of Fe to Pt precursor was kept at 1 : 1 or 2 : 1 whilst OA and OLA amounts were varied (**Table S1**) and the volume of solvent was kept constant (20 mL).

Reaction on a Schlenk line: $Na_2Fe(CO)_4$ was weighed in a glove box into a 100 mL round bottomed flask containing a 15 mm magnetic stirrer bar. The round bottomed flask was transferred to a readily purged Schlenk line equipped with a Liebig condenser. Degassed dioctyl ether (20 mL) was added to

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the flask followed by OLA and OA in this order (for amounts see **Table S1**). $Pt(acac)_2$ (0.25 mmol, 100 mg) was added to the flask with a positive N_2 flow. The mixture was stirred for 5 minutes at 500 rpm and subsequently heated up to 100 °C at a rate of ~20 °C.min^{-1}, the temperature was maintained at 100 °C for 25 min during which the reaction mixture turned a dark green colour. After 25 min, the vessel was heated further to 260 C° at ~20 °C.min^{-1} where it was held for 30 min before cooling back to room temperature. Then, the product was transferred to a 50 mL falcon centrifuge tube; 2 x 5 mL hexane was used to rinse the emptied round bottomed flask and added to the product.

Reaction in an autoclave: $Na_2Fe(CO)_4$ was weighed in a glove box into a 45 mL Parr Teflon liner. The vessel was moved to a Schlenk line where the N_2 exhaust was flowed over the top of the vessel whilst dioctyl ether, OLA and OA were injected into the vessels through a septum in this order (for amounts see **Table S1**). $Pt(acac)_2$ (0.25 mmol, 100 mg) was added to the flask taking care to avoid introducing air into the vessel. The digestion vessel was gently agitated by hand before being assembled with the autoclave jacket and placed into an oven (Memmert, model UFP400). A pre-set program was run (**Table S2**). The oven was cooled to room temperature. The following day, the product was transferred to a 50 mL falcon centrifuge tube; 2 x 5 mL hexane was used to rinse the emptied Teflon liner and added to the product.

To clean the product the following protocol was used and can be scaled up as appropriate. 10 mL product was diluted with 20 mL ethanol and centrifuged at 10018 g (Heraeus, Biofuge Stratos) for 10 min, the supernatant was removed and the precipitate was sonicated in 10 mL hexane. This process was performed a total of 3 times. The product was dispersed in a minimal amount of hexane (0.1 - 0.5 mL) for transfer to a 1.5 mL glass vial from which the hexane could be removed under reduced pressure.

Table S1. Amounts of surfactant and their respective ratios with the amount of metal precursor used. Samples **1** – **6** were performed with equiatomic metal precursor amounts ($Na_2Fe(CO)_4$, 0.25 mmol, 87 mg), samples **7** – **26** were synthesized with Fe : Pt ratio 2 : 1 ($Na_2Fe(CO)_4$, 0.5 mmol, 173 mg), numbers in brackets indicate samples synthesized under autoclave conditions.

Sample number	OA / mmol	OLA / mmol	Ratio [Fe] : [OA]	Ratio[Pt] : [OLA]
1S (4A)R	12.0	4	48	16
2 (5)R	8.0	8	32	32
3 (6)R	4.0	12	16	48
7 (17A)	16.0	0	32	0
8 (18)	12.8	3.2	25.6	12.8
9 (19)R	12.0	4	24	48
10 (20)	10.7	5.3	21.3	16
11 (21)R	8.0	8	16	21.3
12 (22)	5.3	10.7	10.7	8
13 (23)R	4.0	12	8	32
14 (24)	3.2	12.8	6.4	42.7
15 (25)	0.0	16	0	48
16 (26)	0.0	0	0	0

The morphology (size and shape) of the synthesized nanoparticles were studied by transmission electron microscopy (TEM). Grids were prepared using the same solution used for dynamic light

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scattering. TEM images were obtained using a JEOL JEM 1200 EX operating at an acceleration voltage of 120 kV in conjunction with Digital Micrograph™ (version 1.83.842). Size was measured using Pebbles software, which uses an algorithm to calculate the edge of individual particles and their respective dimensions[25]. Minor diameters are reported as the narrowest part particles, while major diameters are the widest, equivalent diameters are averages of the two.

Crystallography of MNPs was investigated by X-ray diffractometry (XRD) using a PanAlytical X'Pert system, equipped with a Co Kα source (λ = 1.789 Å) and an automatic diversion slit for illumination of a constant area of sample. Samples were deposited as a concentrated liquid onto a kapton coated zero background single silica crystal plate and left to dry before analysis. The diffraction pattern was collected within the range 20 - 90 degrees 2θ. Aspect ratios calculated from TEM images were used with the Scherrer equation to calculate the size of crystalline domains.

Zero field cooled (ZFC), field cooled (FC) curves, and hysteresis loops at 5 K and 300 K were obtained using a Superconducting Quantum Interference device equipped with a vibrating sample magnetometer magnetic property measurement system (SQUID VSM MPMS).

Infra-red (IR) spectra were obtained using a Perkin Elmer Spectrum 100 Fourier transform infra-red FTIR, equipped with a Ge universal attenuated total reflectance accessory (UATR). Samples were deposited as a powder onto the UATR sample stage and compressed until the IR monitor indicated that enough signal was gained to generate a spectrum.

Inductively coupled plasma atomic emission spectroscopy (ICP-AES) was performed on a thermo iCAP 6500 duo view spectrometer or a Shimadzu ICPS 7000 Sequential Plasma Spectrometer.

Thermal gravimetric analysis was performed on a Seiko TG/DTA6200 instrument with a heating rate of 20 ^{0}C.min^{-1} under an air atmosphere.

Table S2. IMNPut parameters used for high pressure syntheses of FePt, (SPWT = set point wait), flap indicates how open the exhaust is, fan indicates how fast hot air is being circulated within the oven.

Step	Time hh : mm	Period hh : mm	Temp °C	Type	Flap	Fan
1	0 : 01	0 : 01	100	Spwt	10%	MAX
2	0 : 26	0 : 25	100	Next	50%	50%
3	0 : 41	0 : 15	250	Spwt	50%	50%
4	1 : 41	1 : 00	250	Next	50%	50%
5	2 : 41	1 : 00	20	End	Open	OFF

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Table S3. Shape (the most efficient model for sizing the particles is in brackets), major, minor and equivalent diameters, the resulting aspect ratio, and crystallographic data of samples **1** - **26**; in the crystalline diameter column, suffixes 'min' and 'maj' indicate whether it is closest to the TEM determined minor or major diameter respectively. All data were extracted from **Figure S2 Figure S5**, (where not applicable, the notation NA is used).

Sample	Amount OA / mmol	Amount OLA / mmol	Shape	Major diameter / nm	Minor diameter / nm	Equivalent Diameter / nm	Aspect ratio	Peak/ angle 2θ	Crystalline diameter/ nm
1 S1 : 1	12	4	Cubic (ellipsoid)	7.4 ± 0.8	5.2 ± 0.6	6.8 ± 0.5	1.2	46.7	6.6 maj
2	8	8	Cubic	8.3 ± 0.7	6.1 ± 0.8	6.5 ± 0.6	1.4	46.9	7.7 maj
3	4	12	Cubic	9.7 ± 3.3	5.4 ± 1.3	6.8 ± 1.2	2.0	46.8	8.7 maj
4 A1 : 1	12	4	Cubic (ellipsoid)	6.3 ± 0.9	4.8 ± 1.0	5.4 ± 0.9	1.3	46.8	6.8 maj
5	8	8	Cubic	6.1 ± 0.8	4.0 ± 0.6	4.8 ± 0.6	1.5	46.8	6.6 maj
6	4	12	Cubic (ellipsoid)	6.6 ± 0.8	5.6 ± 0.7	6.1 ± 0.7	1.2	46.9	6.3 maj
7S2 : 1	16	0	Cubic (ellipsoid)	7.6 ± 1.2	6.4 ± 0.8	7.0 ± 0.9	1.2	46.8	6.5 min
8	12.8	3.2	Cubic	6.1 ± 0.6	4.5 ± 0.5	4.8 ± 0.5	1.4	46.9	5.9 maj
9R	12	4	Cubic (ellipsoid)	6.3 ± 0.6	5.3 ± 0.5	5.7 ± 0.5	1.2	46.8	5.3 min
10	10.7	5.3	Cubic (ellipsoid)	5.5 ± 0.8	4.7 ± 0.6	5.1 ± 0.6	1.2	46.9	5.0 min
11R	8	8	Cubic	7.3 ± 0.7	5.5 ± 0.7	5.7 ± 0.5	1.3	46.9	6.0 min
12	5.3	10.7	Cubic (ellipsoid)	5.2 ± 1.1	4.3 ± 0.7	4.7 ± 0.7	1.2	47.0	4.9 maj
13R	4	12	Cubic	9.2 ± 2.9	4.5 ± 0.7	6.4 ± 1.1	2.2	47.0	7.6 maj
14	3.2	12.8	Cubic (ellipsoid)	5.7 ± 0.9	4.8 ± 0.7	5.2 ± 0.7	1.2	46.9	4.9 min
15	0	16	NA	Aggregate	Aggregate	Na	Na	47.0	3.6
16	0	0	NA	Aggregate	Aggregate	Na	Na	Na	Amorphous
17A2 : 1	16	0	Ellipsoid	4.7 ± 1.6	4.0 ± 1.2	4.3 ± 1.3	1.2	46.8	6.5 maj
18	12.8	3.2	Ellipsoid	6.3 ± 0.8	5.5 ± 0.7	5.8 ± 0.7	1.2	46.9	5.9 maj
19R	12	4	Cubic (ellipsoid)	5.8 ± 0.7	5.1 ± 0.5	5.4 ± 0.4	1.2	46.8	5.5 min

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20	10.7	5.3	Cubic (ellipsoid)	8.0 ± 1.0	6.5 ± 0.8	7.2 ± 0.7	1.2	47.0	7.5 maj
21R	8	8	Cubic (ellipsoid)	5.7 ± 0.7	4.8 ± 0.7	5.02 ± 0.7	1.2	47.0	5.3 maj
22	5.3	10.7	Cubic (ellipsoid)	6.2 ± 1.5	5.2 ± 1.2	5.7 ± 1.3	1.2	47.0	6.2 maj
23R	4	12	Mixture (ellipsoid)	6.9 ± 1.2	5.6 ± 1.0	6.2 ± 1.0	1.3	47.0	7.3 maj
24	3.2	12.8	Ellipsoid	6.3 ± 0.8	5.2 ± 0.5	5.7 ± 0.5	1.2	47.0	5.5 min
25	0	16	NA	Aggregate	Aggregate	Na	Na	46.9	4.4
26	0	0	NA	Aggregate	Aggregate	Na	Na	47.0	5.0

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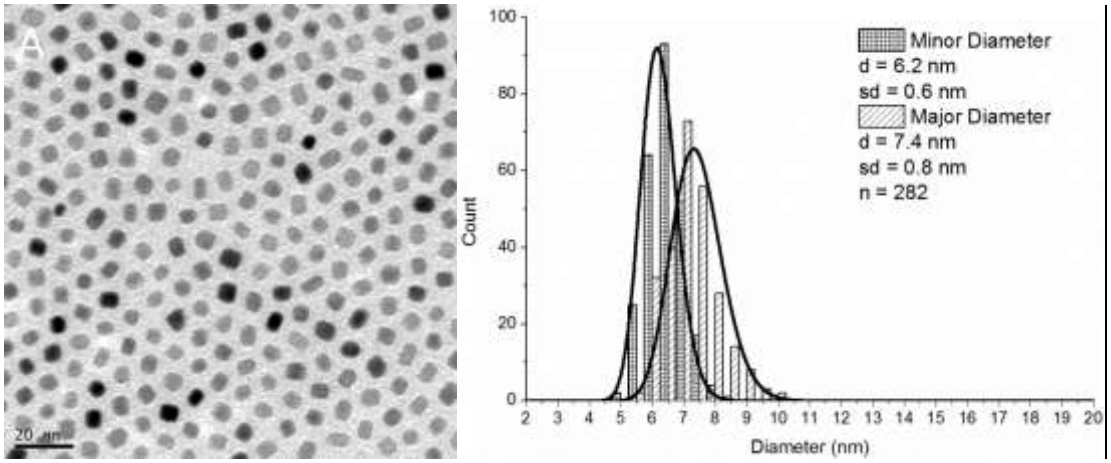
Table S4. Elemental composition (%), saturation magnetisation values (M_S) and blocking temperatures data of samples **1** – **26**. **1** – **3** and **7** – **16** were synthesised on a Schlenk line, **4** – **6** and **17** – **26** in an autoclave**.** All data were extracted from **Figure S7** Figure **S8**.

Sample	Amount OA / mmol	Amount OLA / mmol	Fe / %	300 K, M_S / emu.g^{-1}	T_B / K
1 S1 : 1	12	4	10	0.02	21
2	8	8	10	0.7	42
3	4	12	14	1.0	67
4 A1 : 1	12	4	12	0.5	28
5	8	8	17	1.4	34
6	4	12	20	4.9	47
7S2 : 1	16	0	-	0.1	23
8	12.8	3.2	-	0.1	21
9R	12	4	4	0.1	16
10	10.7	5.3	-	0.4	32
11R	8	8	24	0.7	35
12	5.3	10.7	-	0.6	32
13R	4	12	21	3.1	65
14	3.2	12.8	-	2.0	35
15	0	16	-	14.6	88
16	0	0	-	7.4	>300
17A2 : 1	16	0	-	0.2	26
18	12.8	3.2	-	0.8	30
19R	12	4	10	1.0	28
20	10.7	5.3	-	2.3	61
21R	8	8	36.3	2.9	54
22	5.3	10.7	-	23.4	~
23R	4	12	35	25.1	54/110
24	3.2	12.8	-	23.8	57
25	0	16	-	8.3	70
26	0	0	-	13.0	98



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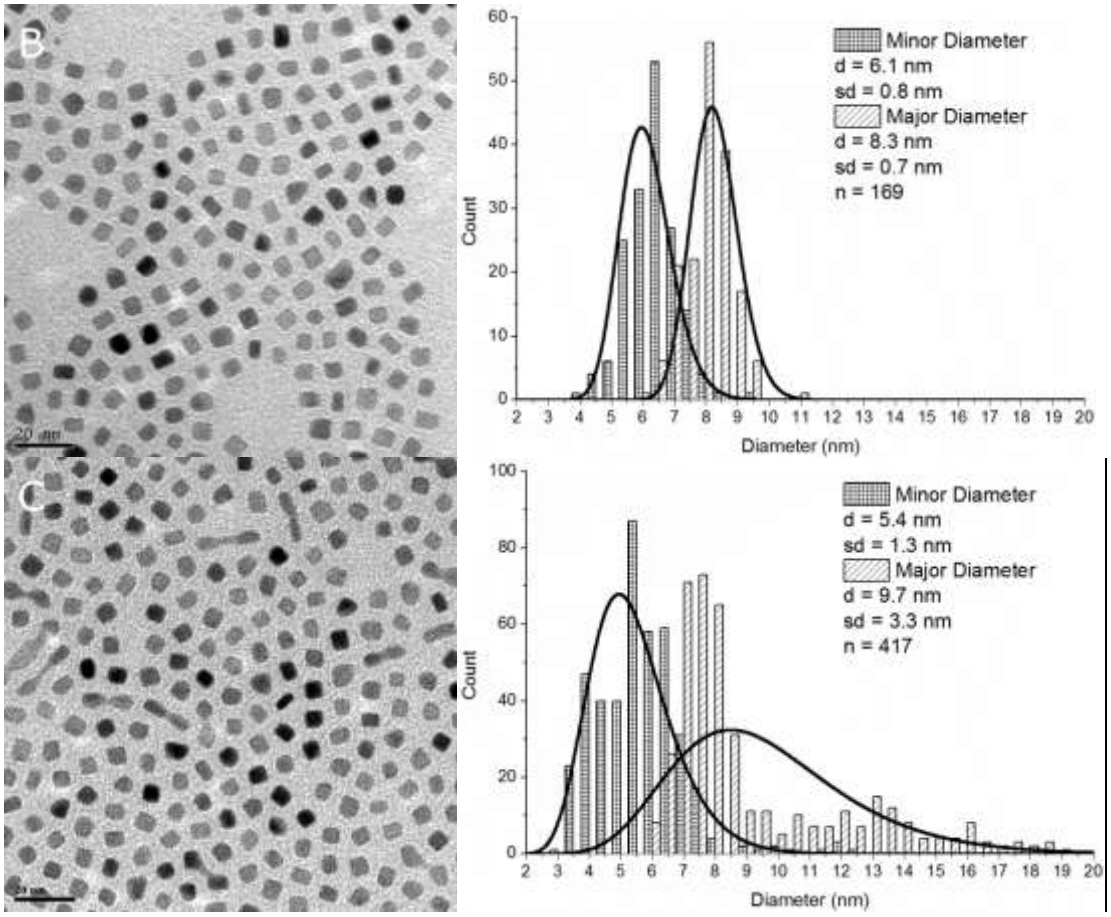

Figure S1. TEM images, samples **1 – 3**, synthesized under Schlenk conditions with equimolar Fe : Pt precursor amounts and changing amounts of OA and OLA, **A,** OA : OLA = 12 : 4 mmol; **B**, OA : OLA = 8 : 8 mmol; **C**, OA : OLA = 4 : 12 mmol.

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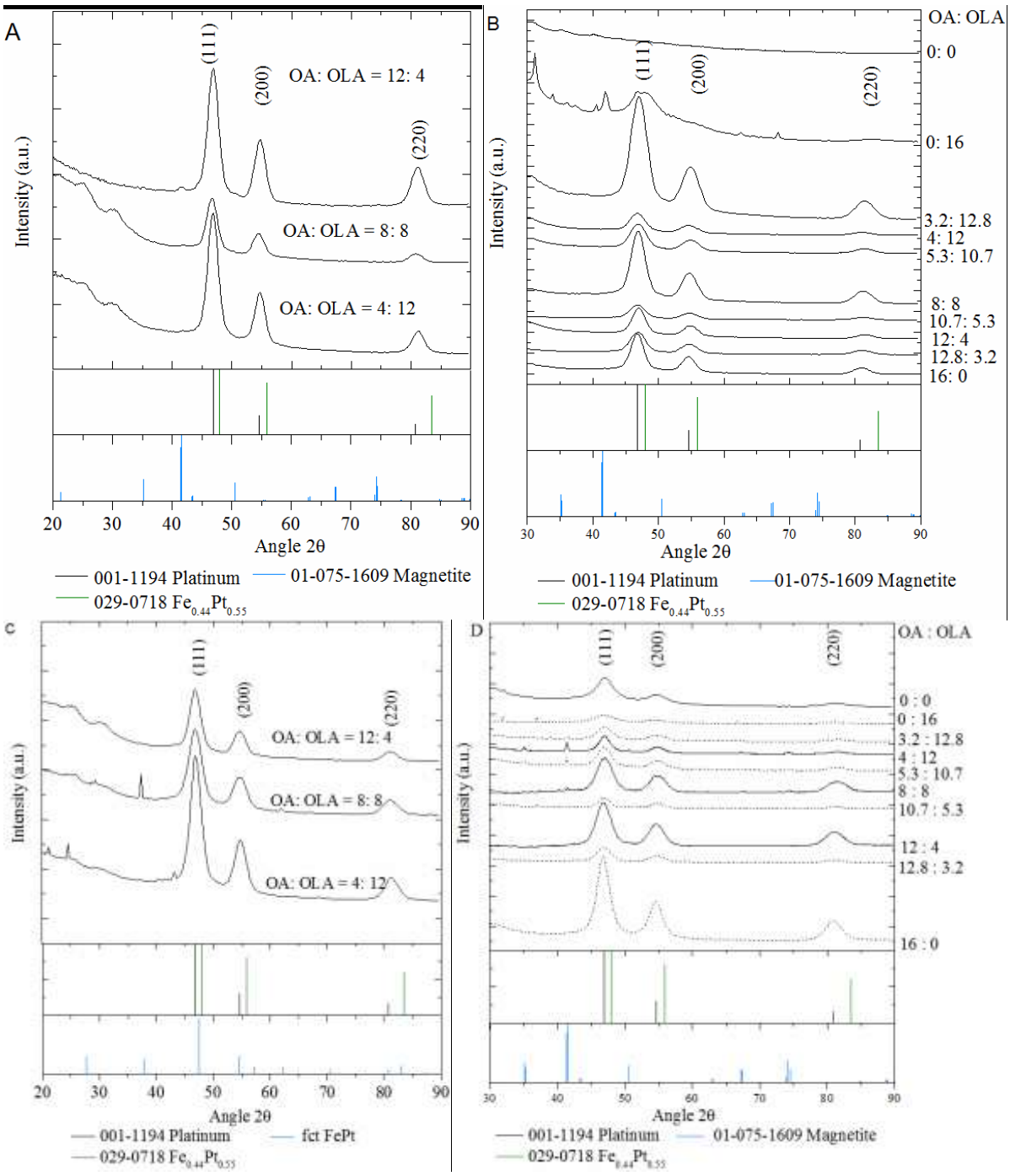

Figure S2. XRD crystallographic data of samples synthesized on a Schlenk line A, B; and in an autoclave C, D. A and C, were synthesized with equiatomic metal precursor amounts and B and D with Fe : Pt precursor ratio of 2 : 1. Amounts of surfactant are detailed in mmol as ratios.

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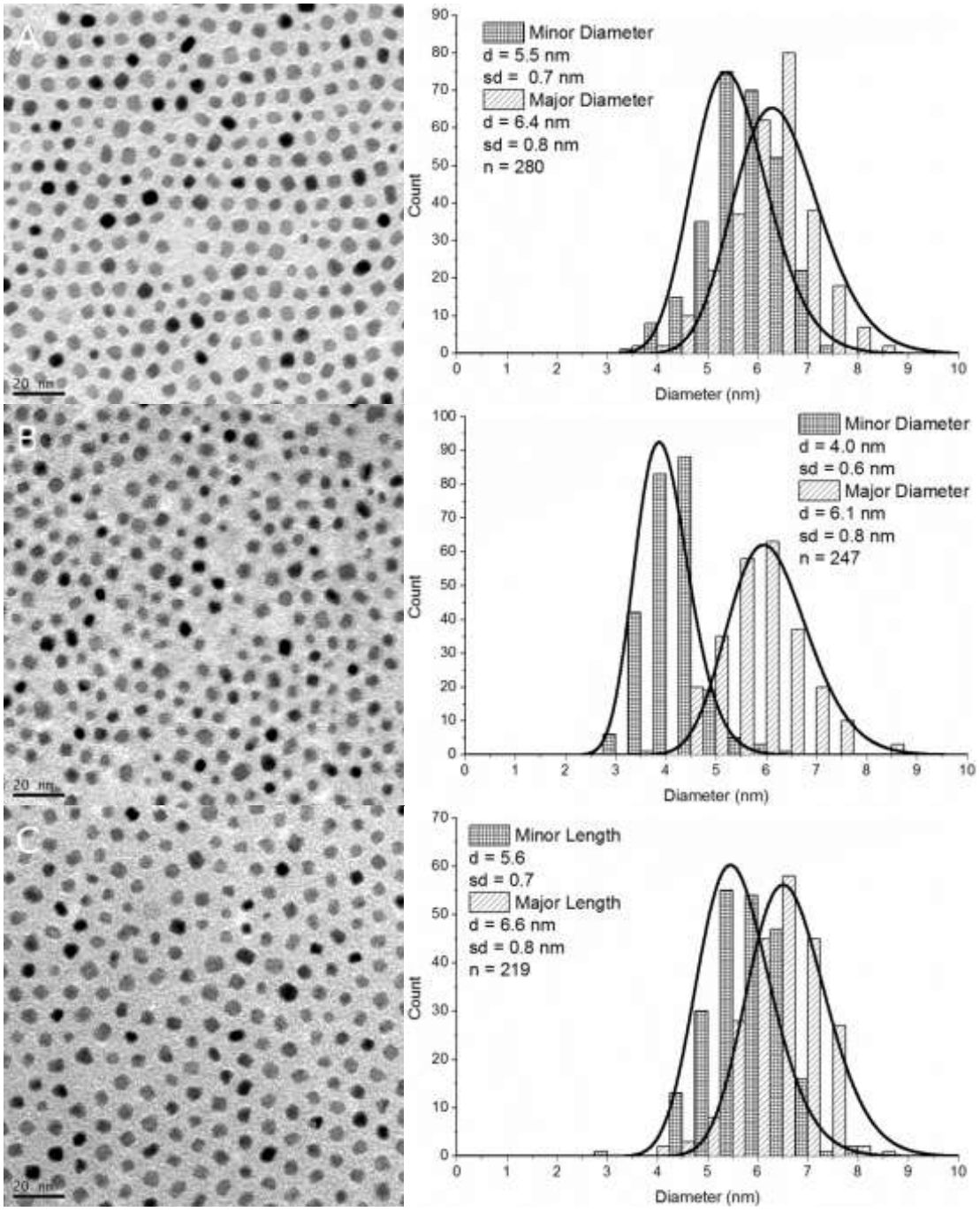
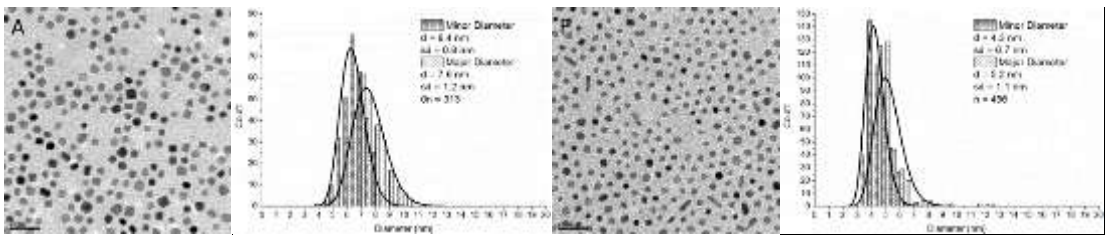


Figure S3. TEM images, samples **4 – 6**, synthesized under autoclave conditions with equimolar Fe : Pt precursor amounts and changing amounts of oleic acid and oleylamine, **A,** OA : OLA = 12 : 4 mmol; **B,** OA : OLA = 8 : 8 mmol; **C**, OA : OLA = 4 : 12 mmol.



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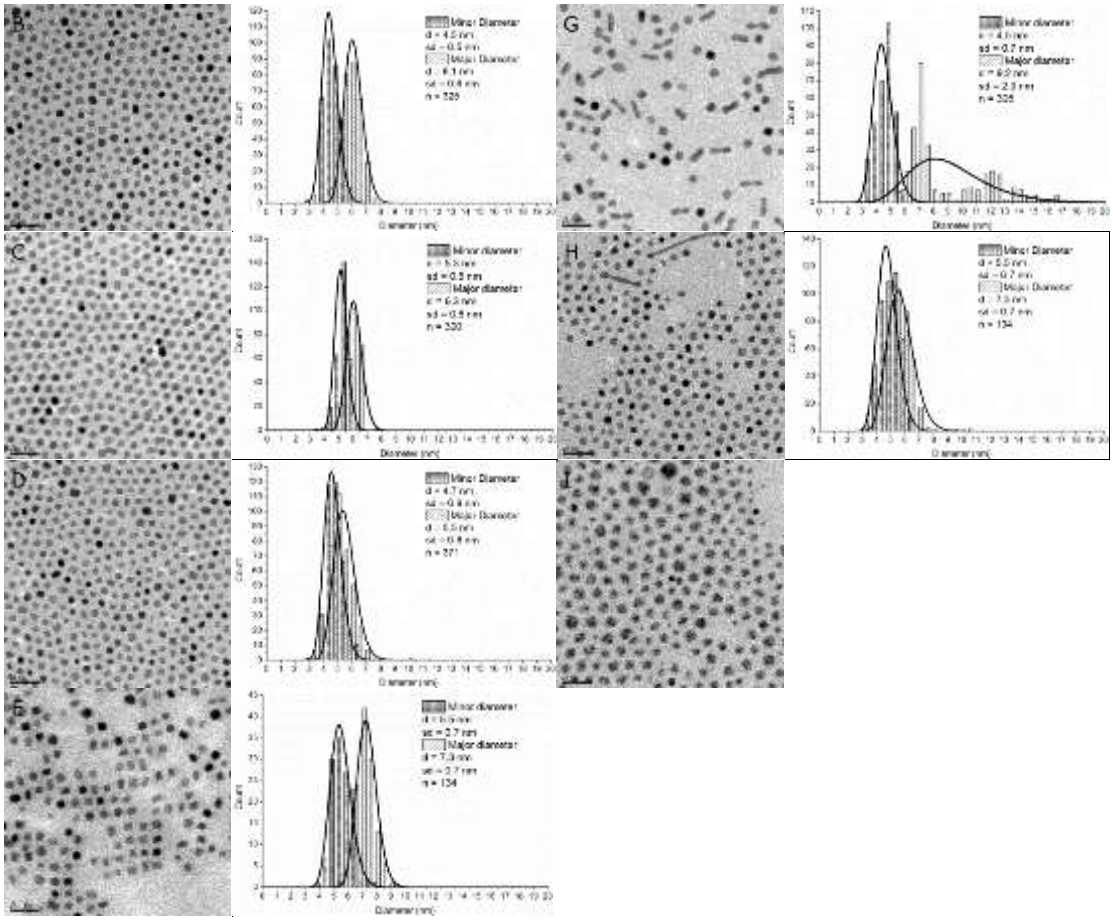
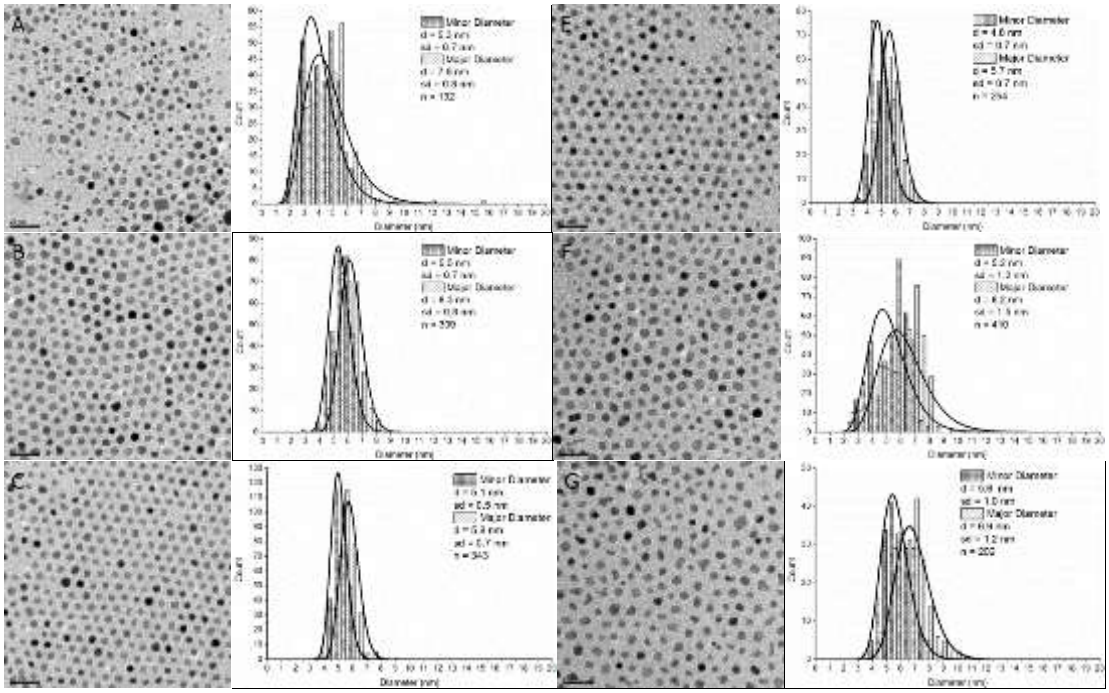


Figure S4. TEM images, samples **7 - 15**, synthesized under Schlenk conditions with Fe : Pt precursor ratio of 2 : 1 and changing amounts of oleic acid and oleylamine, **A**, OA : OLA = 16 : 0 mmol; **B**,OA : OLA = 12.8 : 3.2; **C**, OA : OLA = 12 : 4 mmol; **D**, OA : OLA = 10.7 : 5.3 mmol; **E**, OA : OLA = 8 : 8 mmol; **F**, OA : OLA = 5.3 : 10.7 mmol; **G**, OA : OLA = 4 : 12 mmol; **H**, OA : OLA = 3.2 : 10.7 mmol.



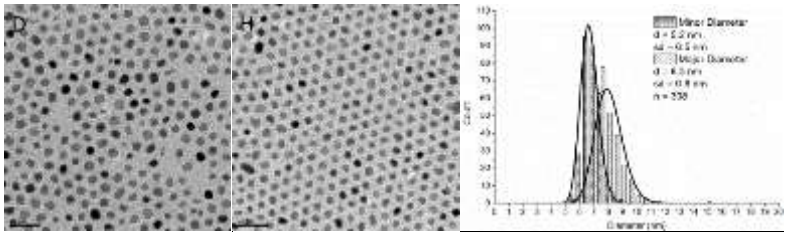


Figure S5. TEM images, samples **17 - 24**, synthesized under autoclave conditions with Fe : Pt precursor ratio of 2 : 1 and changing amounts of oleic acid and oleylamine, **A**, OA : OLA = 16 : 0 mmol; **B**, OA : OLA = 12.8 : 3.2; **C**, OA : OLA = 12 : 4 mmol; **D**, OA : OLA = 10.7 : 5.3 mmol; **E**, OA : OLA = 8 : 8 mmol; **F**, OA : OLA = 5.3 : 10.7 mmol; **G**, OA : OLA = 4 : 12 mmol; **H**, OA : OLA = 3.2 : 10.7 mmol.

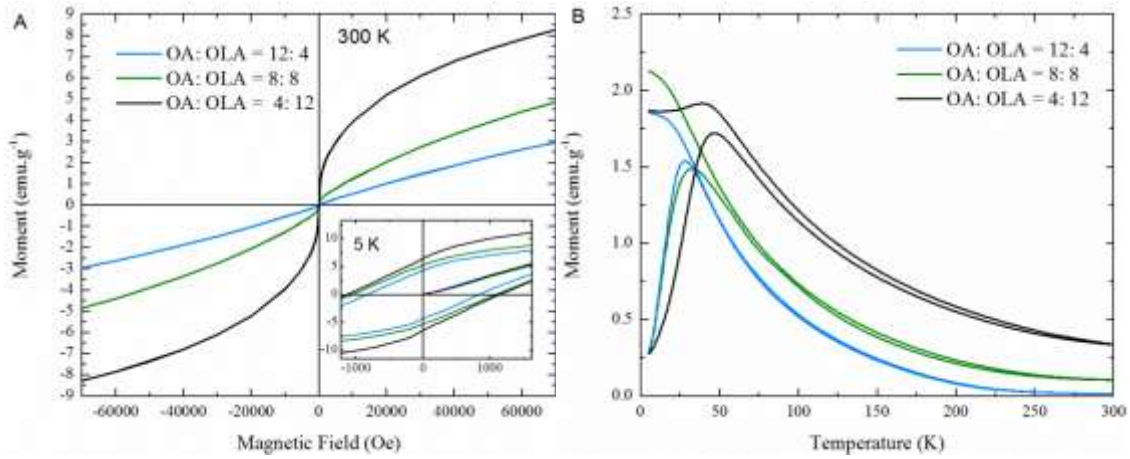


Figure S6. SQUID saturation magnetisation A, and blocking temperature curves B of samples synthesised in an autoclave with amount ratios (mmol) of OA : OLA; 12 : 4; 8 : 8; and 4 : 12.

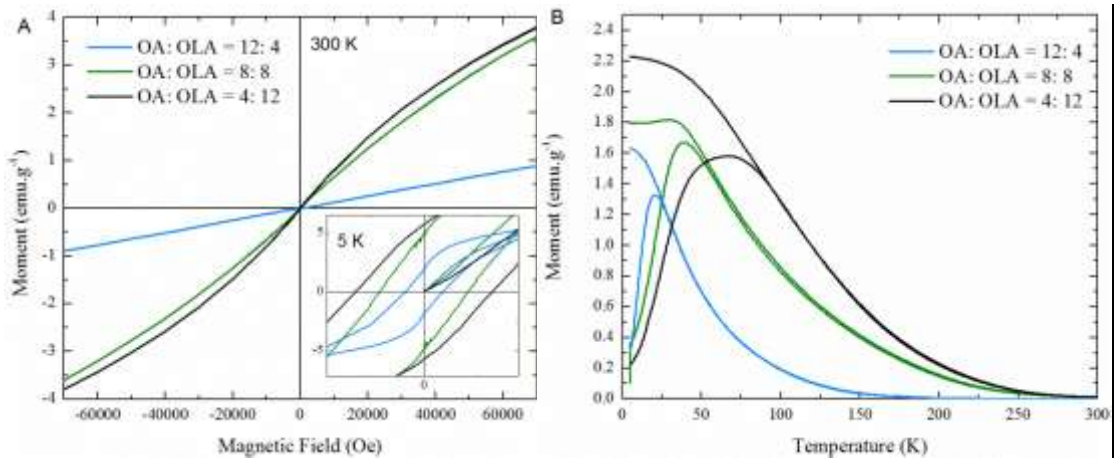


Figure S7. SQUID magnetic data of samples synthesized with equiatomic metal precursor amounts; A and B, hysteresis loops (300 K) and ZFCFC curves of samples synthesized under Schlenk conditions (samples **1 – 3**).

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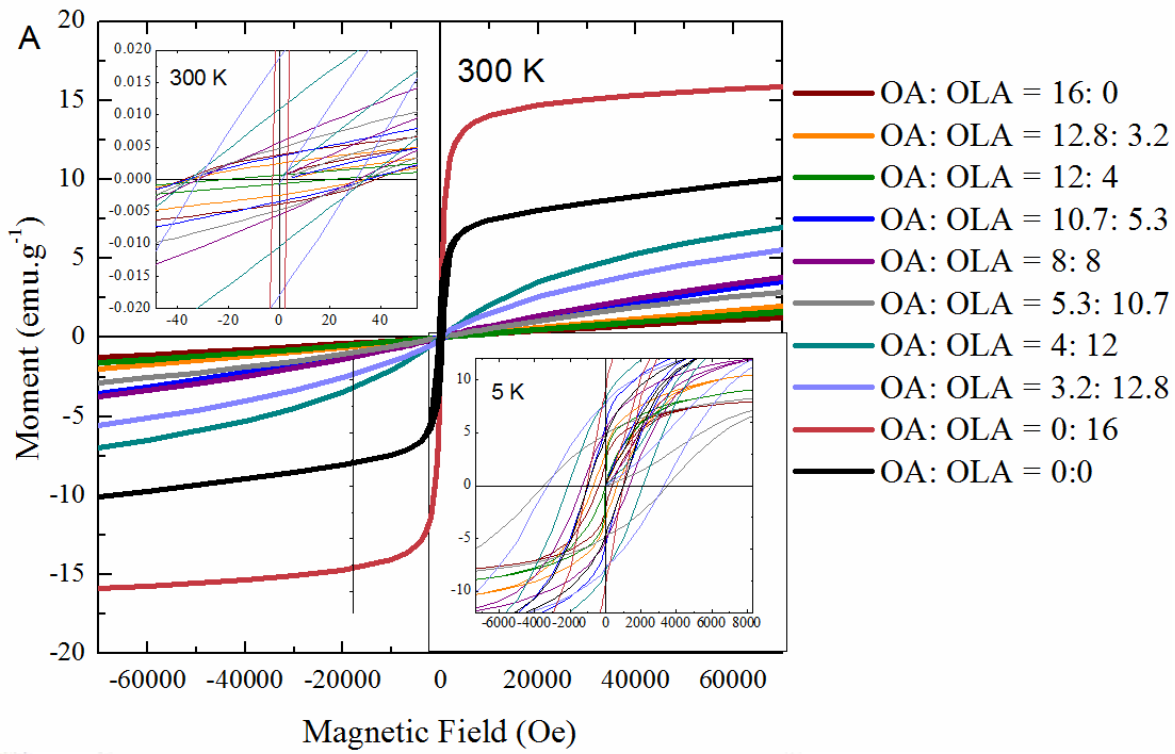

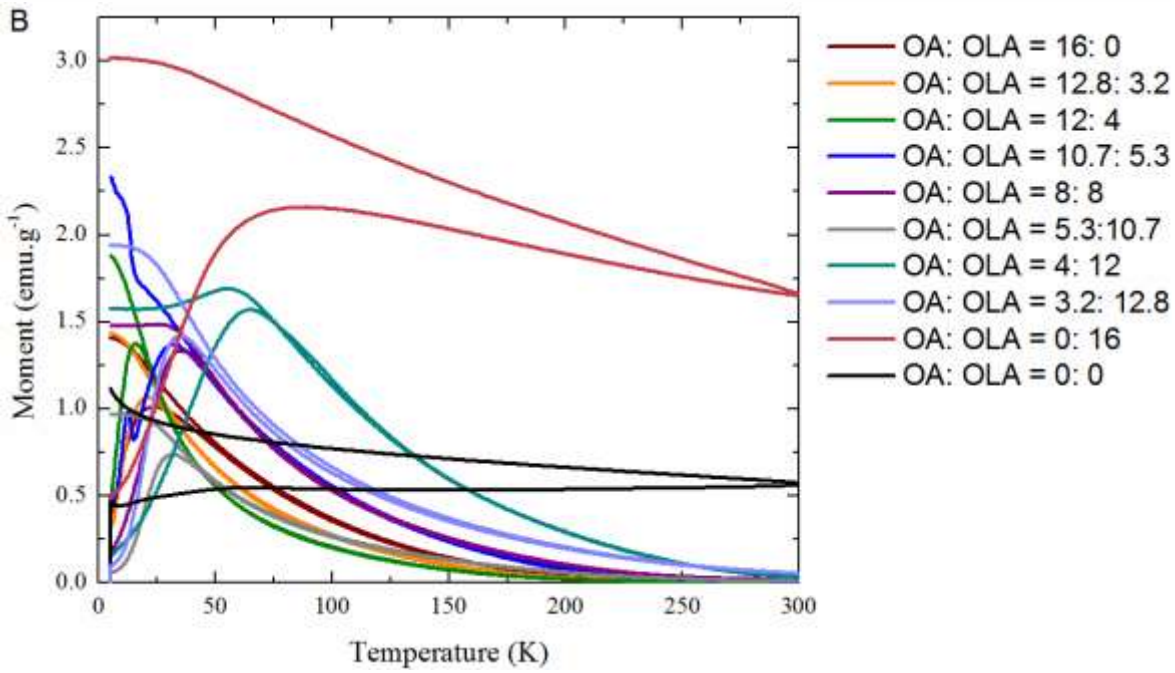

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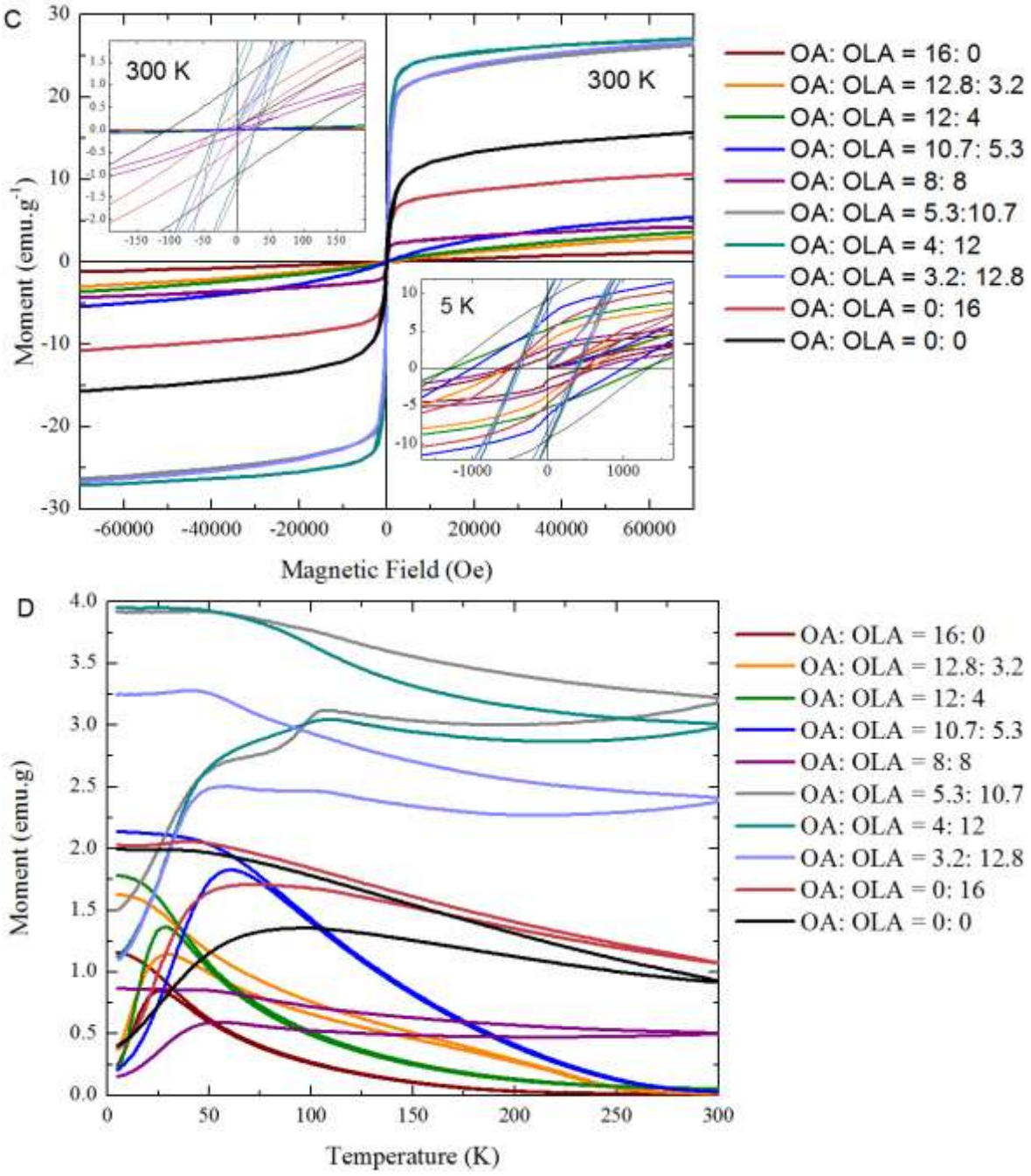

Figure S8. SQUID magnetic data of samples synthesized with Fe : Pt ratio of 2 : 1; A and C, Hystereses loops and B and D, ZFCFC curves of samples synthesized under Schlenk (samples **7** – **16**) and autoclave conditions respectively (samples **17** – **26**).