

A simple procedure for the production of large ferromagnetic cobalt nanoparticles

Rebecca O. Fuller,^{*a} Bee-Min Goh,^a George. A. Koutsantonis,^a Matthys J. Loedolff,^a Martin Saunders,^b and Robert C. Woodward^c

^a School of Chemistry and Biochemistry M310, The University of Western Australia, Crawley WA 6009, Australia.

^b Centre for Microscopy, Characterisation and Analysis, M010, The University of Western Australia, Crawley WA 6009, Australia.

^c School of Physics, M013, The University of Western Australia, Crawley WA 6009, Australia.

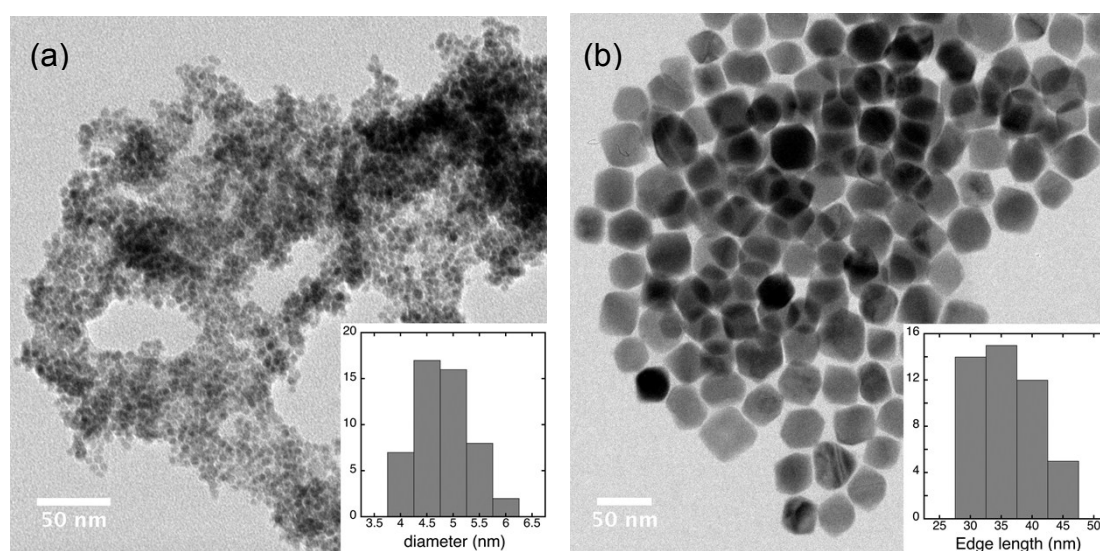


Figure S1: Bright Field TEM images of nanoparticles recovered from (a) the reaction solution (4.8 ± 0.7 nm) and (b) magnetic stirrer bar (36 ± 6 nm). The inset contains histogram of the average particle size.

Table S1: Reaction conditions for particles shown in Figure S1 and two repeat reaction conditions. The OA and TOPO are in 15 mL DCB and heated to reflux at a rate of $4^\circ\text{C}/\text{min}$. Prior to hot injection the $\text{Co}_2(\text{CO})_8$ is in 3 mL DCB heated 50°C .

Reaction	OA (mL)	TOPO (mg)	$\text{Co}_2(\text{CO})_8$ (g)	Temperature following hot injection ($^\circ\text{C}$)	Reaction time after injection (min)	Large particle Yield (g)	Large particle size (nm)
1	0.2	0.1059	0.54	130	22	0.101	36 ± 6
2	0.2	0.1054	0.55	140	20	0.159	25 ± 5
3	0.2	0.099	0.8	130	20	0.080	24 ± 3

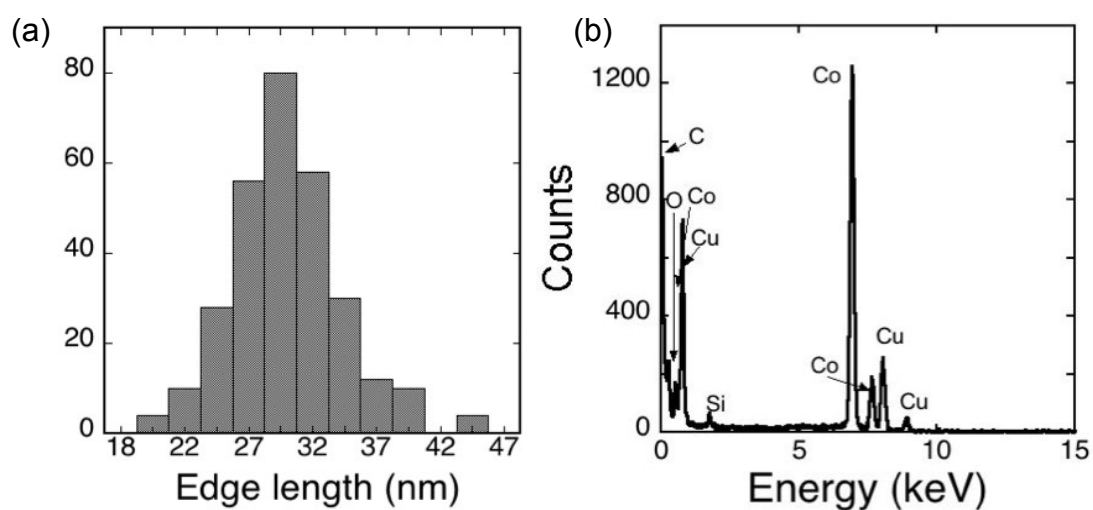
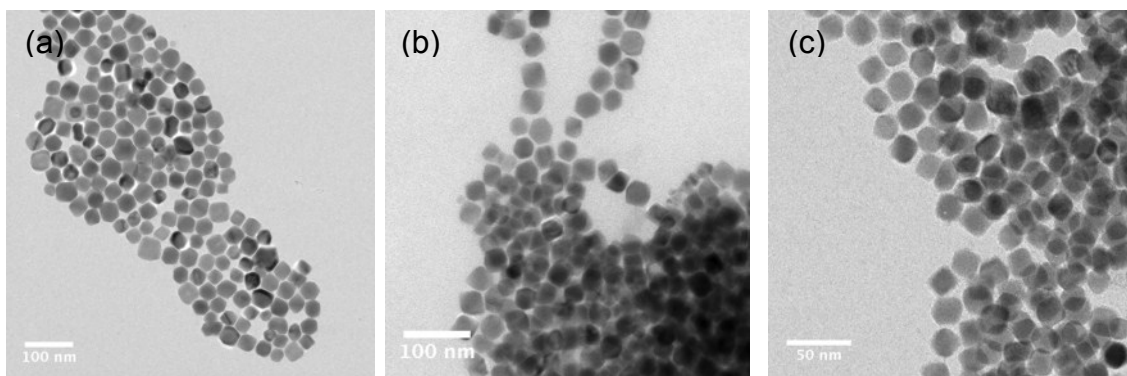


Figure S2: (a) Histogram of the size distribution of the particles (30 ± 4 nm) from bright field TEM images. Edge length was measured for 300 particles. (b) a typical EDX spectra of the particles.



independent reactions. Table S2 contains the reaction conditions used for each.

Table S2: Reaction conditions for particles shown in Figure 1 (a) and two repeat reaction (b) (c). Bright field TEM images for each are shown in Figure S3. The OA and TOPO are in 15 mL DCB and heated to reflux at a rate of 3 °C/min. Prior to hot injection the $\text{Co}_2(\text{CO})_8$ is in 4 mL DCB heated 50 °C. After hot injection the temperature is maintained to within 10 °C of the temperature following hot injection. Note the reaction is not stirred.

Reaction	OA (mL)	TOPO (mg)	$\text{Co}_2(\text{CO})_8$ (g)	Temperature following hot injection (°C)	Reaction time after injection (min)	Yield (g)	Particle size (nm)
(a)	0.2	0.1	0.54	140	24	0.086	30±4
(b)	0.2	0.0998	0.55	130	20	0.126	35±4
(c)	0.2	0.1	0.51	120	18	0.1	20±3

Table S3: A comparison of d-spacing values for SAED and XRD with ϵ -Co from Angew. Chem. Int. Ed. 38 1788 (1999) and P. A. Stadelmann. JEMS - EMS java version, 2004.

hkl	SAED d-spacing Figure 3 (nm)	SAED Figure 3 label	XRD d-spacing Figure 3 (nm)	d-spacing (Intensities %) Literature XRD ^[1, 2]	d-spacing ϵ -Co P. A. Stadelmann. JEMS - EMS java version, 2004
110	4.45	a	-	-	4.31
111	3.73	b	-	-	3.52
210	2.80	c	-	2.73 (4.5)	2.73
211	2.50	d	2.44	2.49 (0.8)	2.49
220	2.15	e	2.13	-	2.15
221	2.03	f	2.03	2.03 (100)	2.03
310	1.95	g	1.93	1.93 (58)	1.93
311	1.88	h	1.83	1.84 (25)	1.84
320	1.67	i		-	1.69
321	-	-	1.41	1.63(5)	
330	1.42	j	1.43	1.44(5)	1.44
420	-	-	1.35	1.37(5)	
510	1.22	k	1.195	1.195(21)	1.195
511	-	-		1.17(4)	1.17
520	1.15	l		1.13(19)	1.13

- [1] D. P. Dinega, M. G. Bawendi. *Angew. Chem. Int. Ed.* **1999**, 38, 1788-1791.
[2] V. F. Puentes, K. M. Krishnan, P. Alivisatos. *Appl. Phys. Lett.* **2001**, 78, 2187-2189.

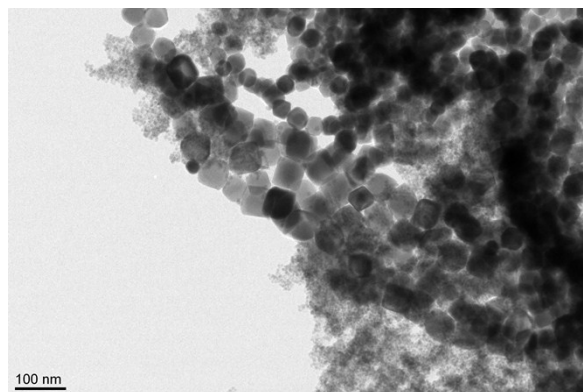


Figure S4: Bright field TEM of both large polycrystalline ϵ -Co particles and small single crystal ϵ -Co formed after 30 mins.

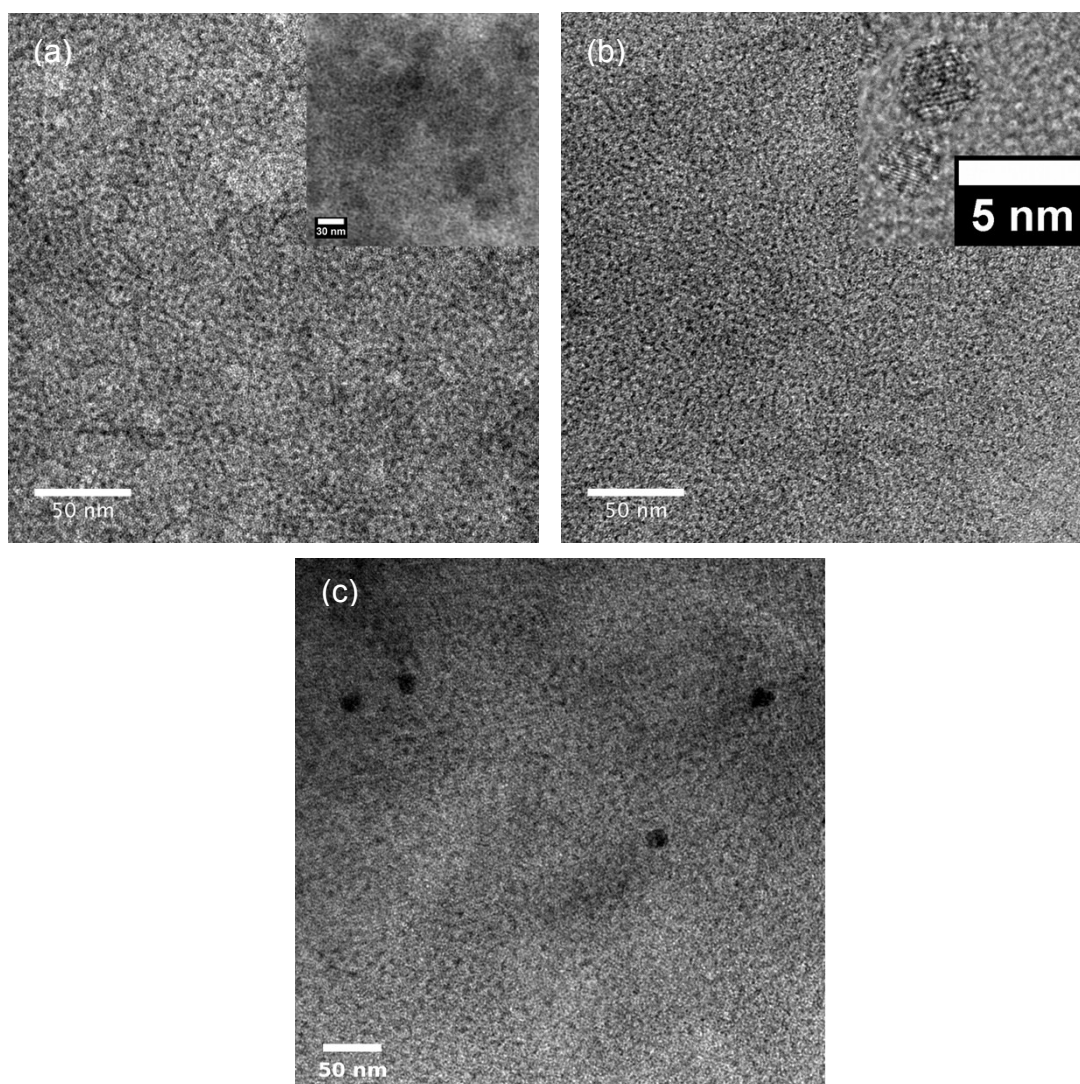


Figure S5: Bright field TEM of the reaction at 3 (a) and 6 (b) min. Only small crystals are present in the images. Additional techniques have been used to image the very small crystals present at these times. High-angle annular dark field scanning transmission electron microscopy (HAADF STEM) is shown in the inset to (a). The atomic contrast highlights the presence of the cobalt particles. At 6 min high resolution TEM is possible, the inset to (b) highlights the single crystal nature of the particles. (c) At 12 min both larger 15-20 nm Co particles are found in addition to smaller nanocrystals.

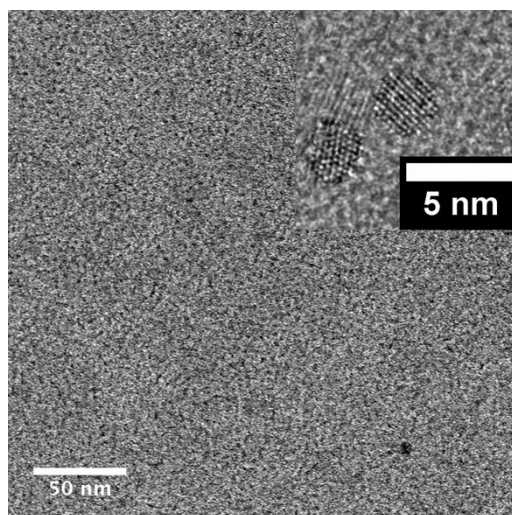


Figure S6: Bright field TEM image of the Co nanoparticles produced from a reaction with an increased amount of oleic acid. The inset contains a high resolution TEM image that illustrates the single crystal nature of individual particles.