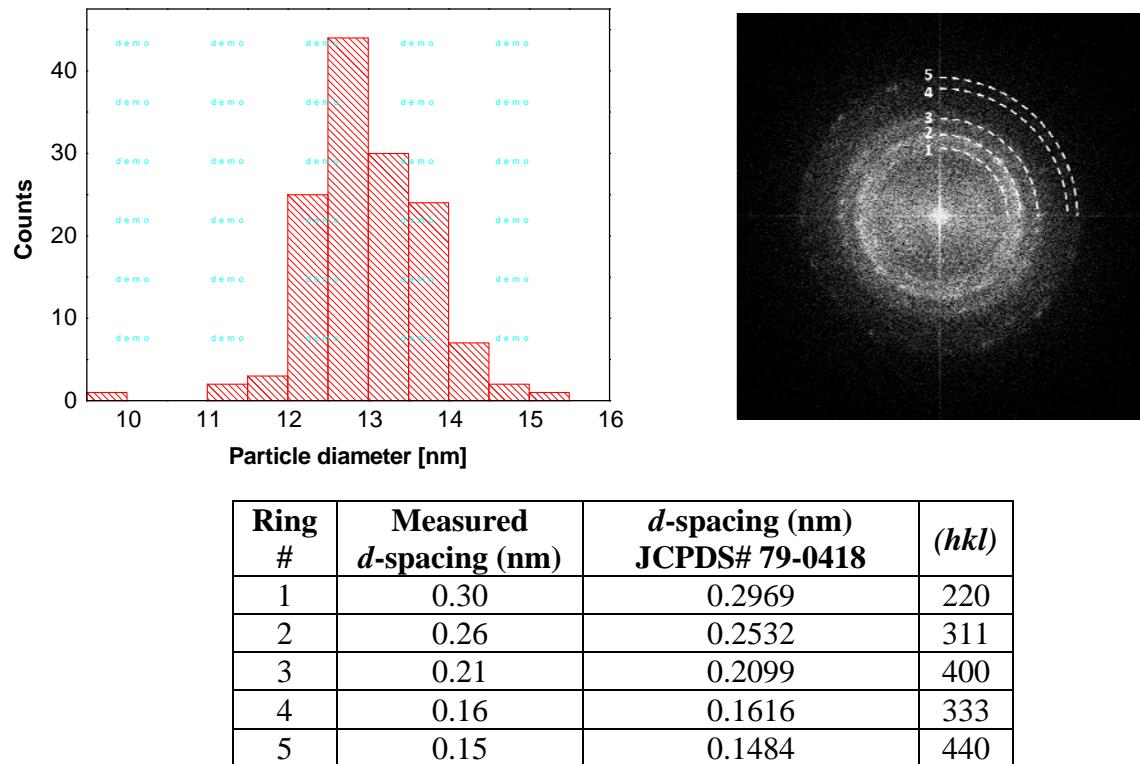
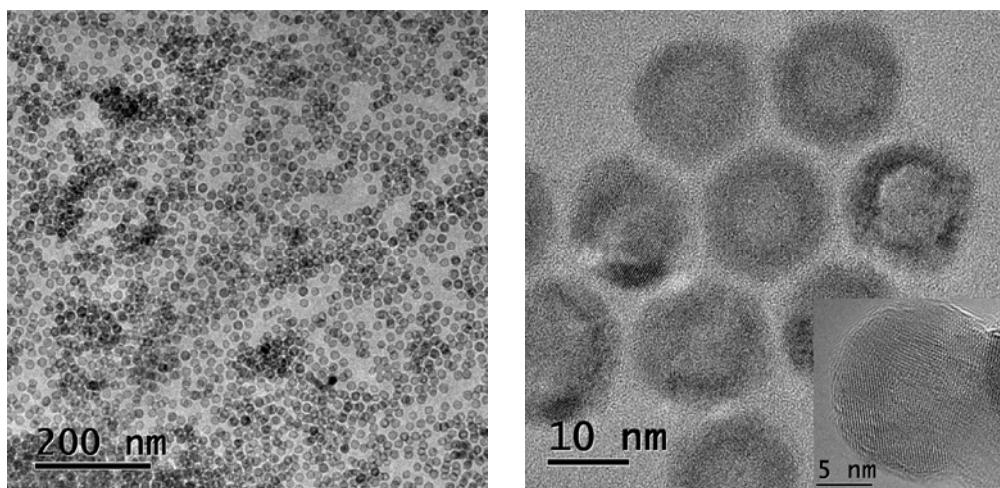


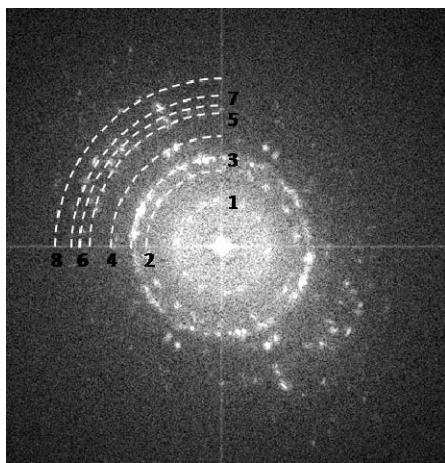
## The Role of Frozen Spins in the Exchange Anisotropy of Core–Shell Fe@Fe<sub>3</sub>O<sub>4</sub> Nanoparticles: Supporting Information



**Figure S1.** (Left) Statistical analysis of particle size ( $13.0 \pm 0.7$  nm,  $N=XXX$ ). (Right and bottom) electron diffraction analysis of Fe@Fe<sub>3</sub>O<sub>4</sub> core-shell nanoparticles, derived by FFT of the HRTEM image in Figure 1 (inset).

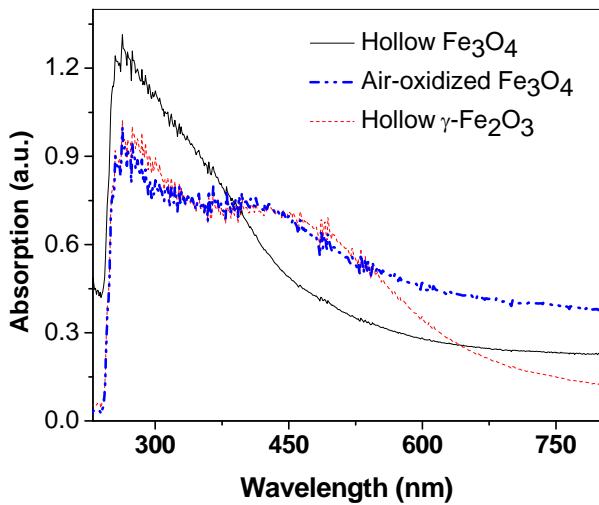


**Figure S2.** (Left) TEM image (Titan F-30, 300 kV) showing monodispersity of Fe<sub>3</sub>O<sub>4</sub> hollow nanoparticles. (Right) HRTEM images (Titan F-30, 300 kV) of the hollow nanoparticles.

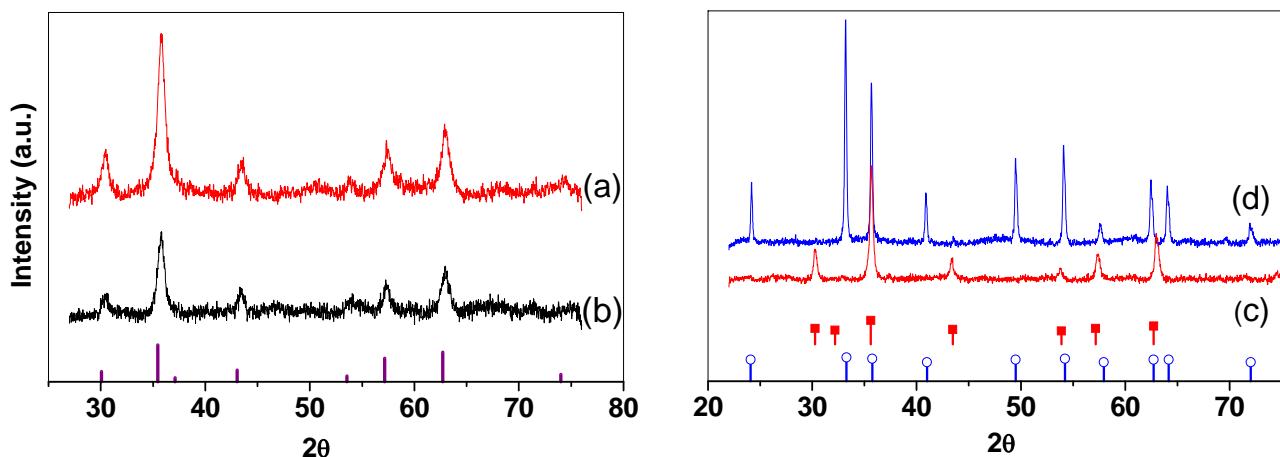


Ring #	Measured d-spacing (nm)	d-spacing (nm) JCPDS 79-0418	(hkl)
1	0.49	0.4848	111
2	0.29	0.2969	220
3	0.25	0.2532	311
4	0.21	0.2099	400
5	0.17	0.1714	422
6	0.16	0.1616	333
7	0.15	0.1484	440
8	0.13	0.1327	620

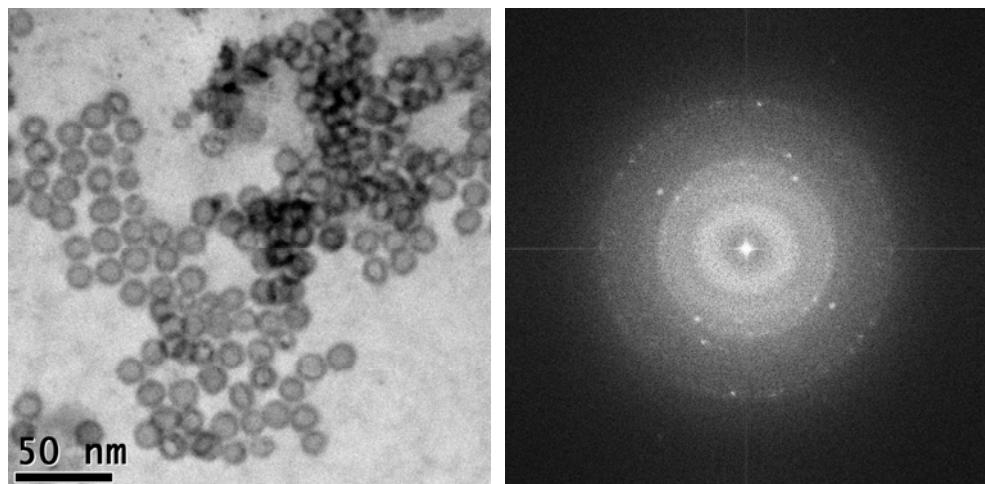
**Figure S3.** Electron diffraction analysis of hollow  $\text{Fe}_3\text{O}_4$  nanoparticles, using the FFT of a HRTEM image (Figure S2, inset).



**Figure S4.** Optical absorption spectra of freshly prepared hollow  $\text{Fe}_3\text{O}_4$  nanoparticles, by oxidation with  $(\text{CH}_3)_3\text{NO}$  (—), (ii) hollow iron-oxide nanoparticles prepared by oxidation in air (···), and hollow  $\gamma\text{-Fe}_2\text{O}_3$  nanoparticles prepared according to Ref. 16 (---).



**Figure S5.** (Left) X-ray diffraction spectra of hollow  $\text{Fe}_3\text{O}_4$  nanoparticles, before (a) and after (b) heating at  $500^\circ\text{C}$ . (Right) (c) X-ray diffraction spectra of  $\gamma\text{-Fe}_2\text{O}_3$  nanoparticles; (d) hollow iron-oxide nanoparticles with conversion to  $\alpha\text{-Fe}_2\text{O}_3$ , after heating at  $500^\circ\text{C}$ .



**Figure S6.** TEM image (Titan F-30, 300 kV) and corresponding FFT of aged nanoparticles after annealing at  $400^\circ\text{C}$  in argon, indicating a solid-to-hollow transition.