

Electronic supplementary information for:

**Aqueous synthesis of polyhedral “brick-like” iron oxide nanoparticles for hyperthermia and  $T_2$  MRI contrast enhancement**

Matthew Worden<sup>a</sup>, Michael A. Bruckman<sup>b</sup>, Min-Ho Kim<sup>d</sup>, Nicole F. Steinmetz<sup>bc</sup>, James M. Kikkawa<sup>e</sup>, Catherine LaSpina<sup>a</sup> and Torsten Hegmann<sup>\*af</sup>

<sup>a</sup> Department of Chemistry and Biochemistry, Kent State University, Kent, OH

<sup>b</sup> Department of Biomedical Engineering, Case Western Reserve University, Cleveland, OH

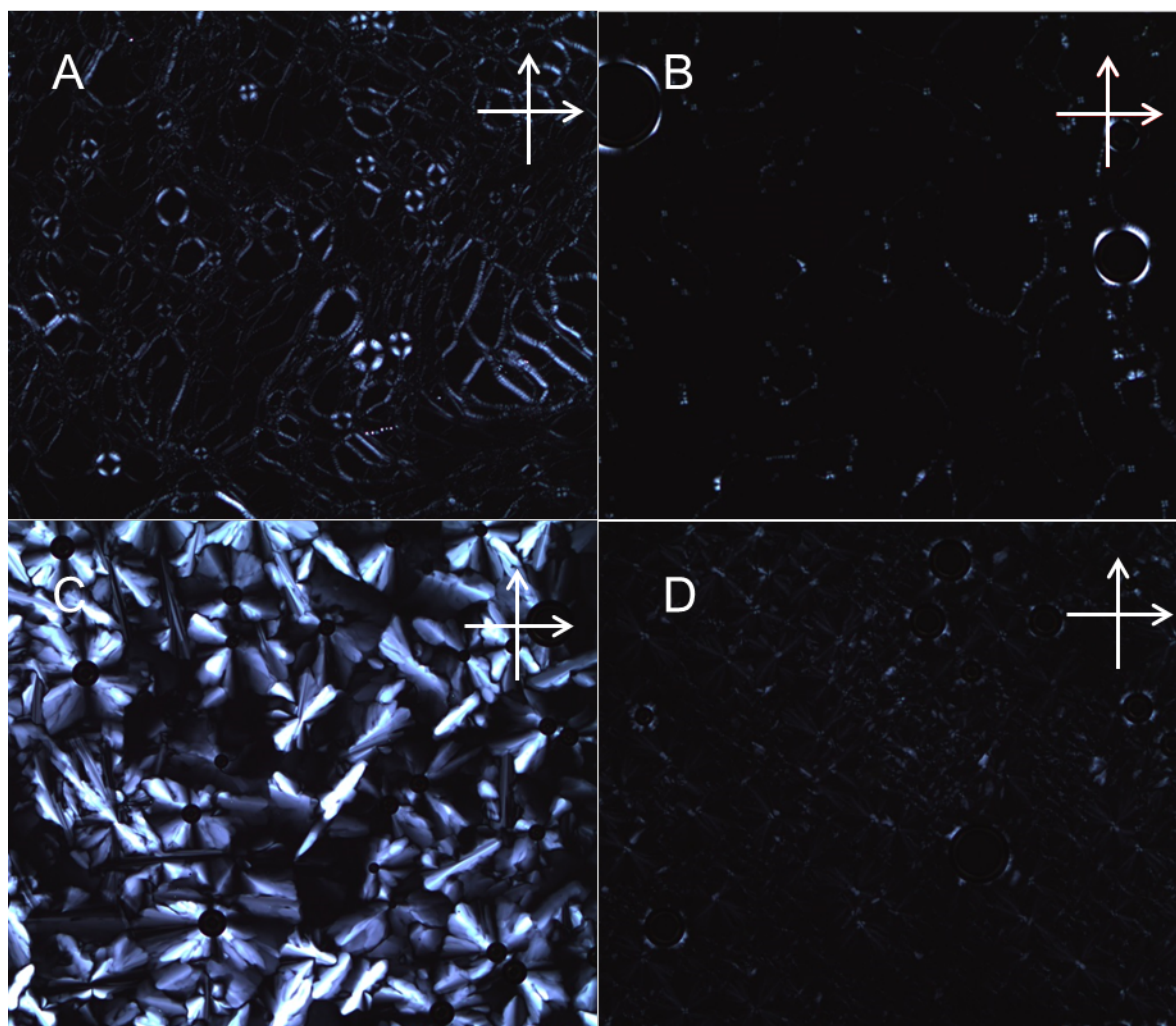
<sup>c</sup> Departments of Radiology, Materials Science and Engineering, and Macromolecular Science and Engineering, Case Western Reserve University, Cleveland, OH

<sup>d</sup> Department of Biological Sciences, Kent State University, Kent, OH

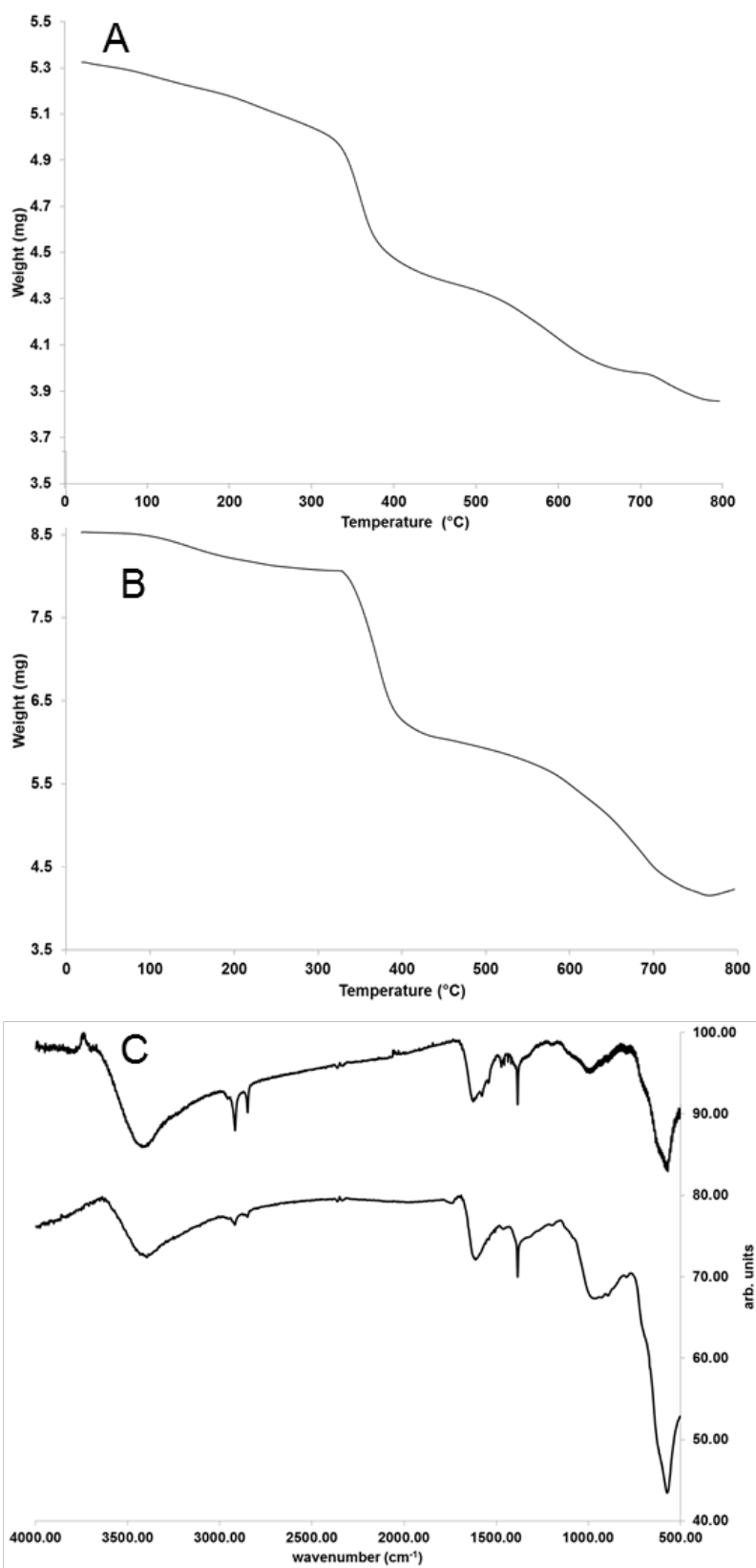
<sup>e</sup> Department of Physics & Astronomy, University of Pennsylvania, Philadelphia, PA

<sup>f</sup> Liquid Crystal Institute, Chemical Physics Interdisciplinary Program, Kent State University, Kent, OH

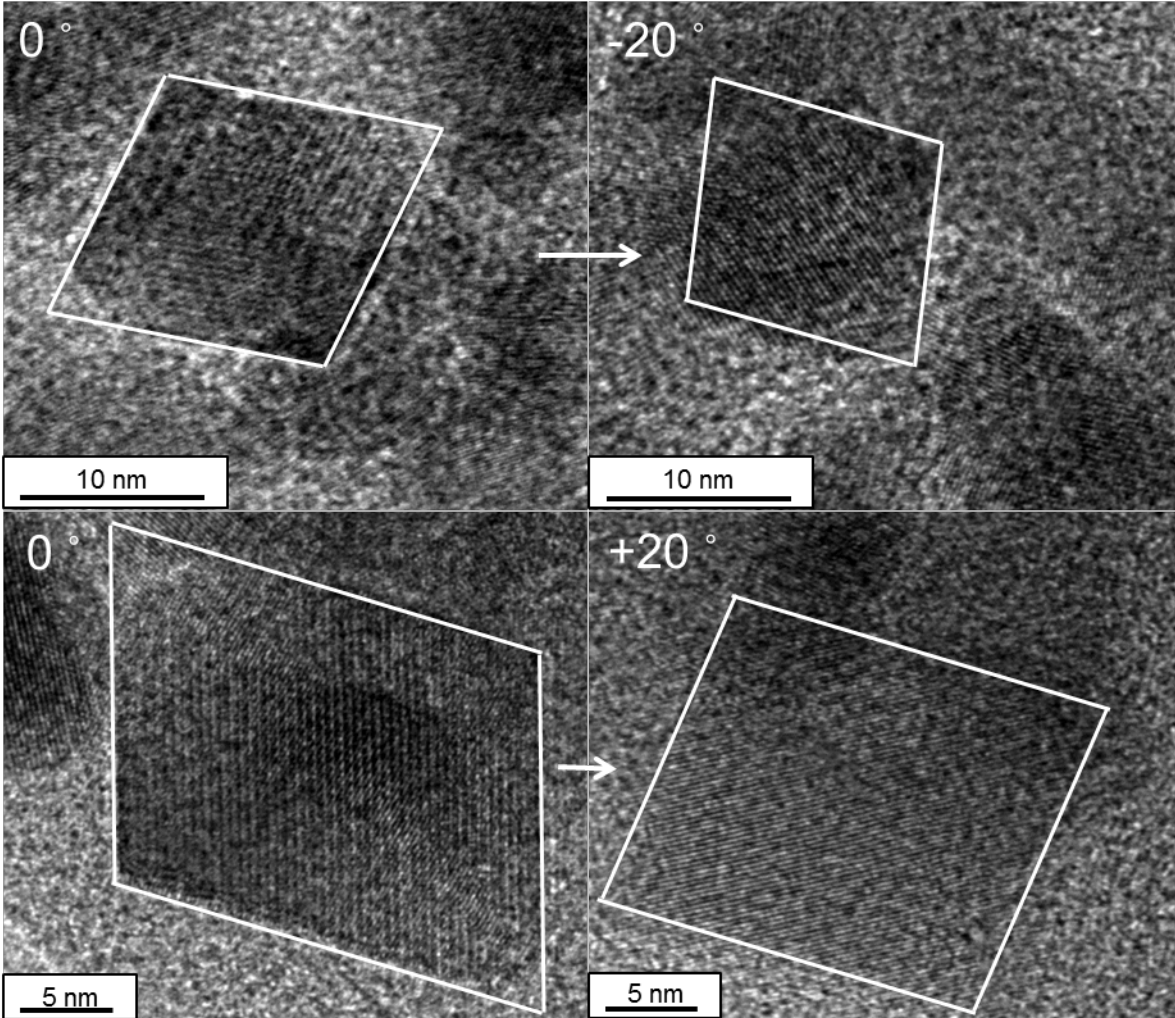
Email: [thegmann@kent.edu](mailto:thegmann@kent.edu); phone: 1 (330) 672 7770; fax: 1 (330) 672 2796



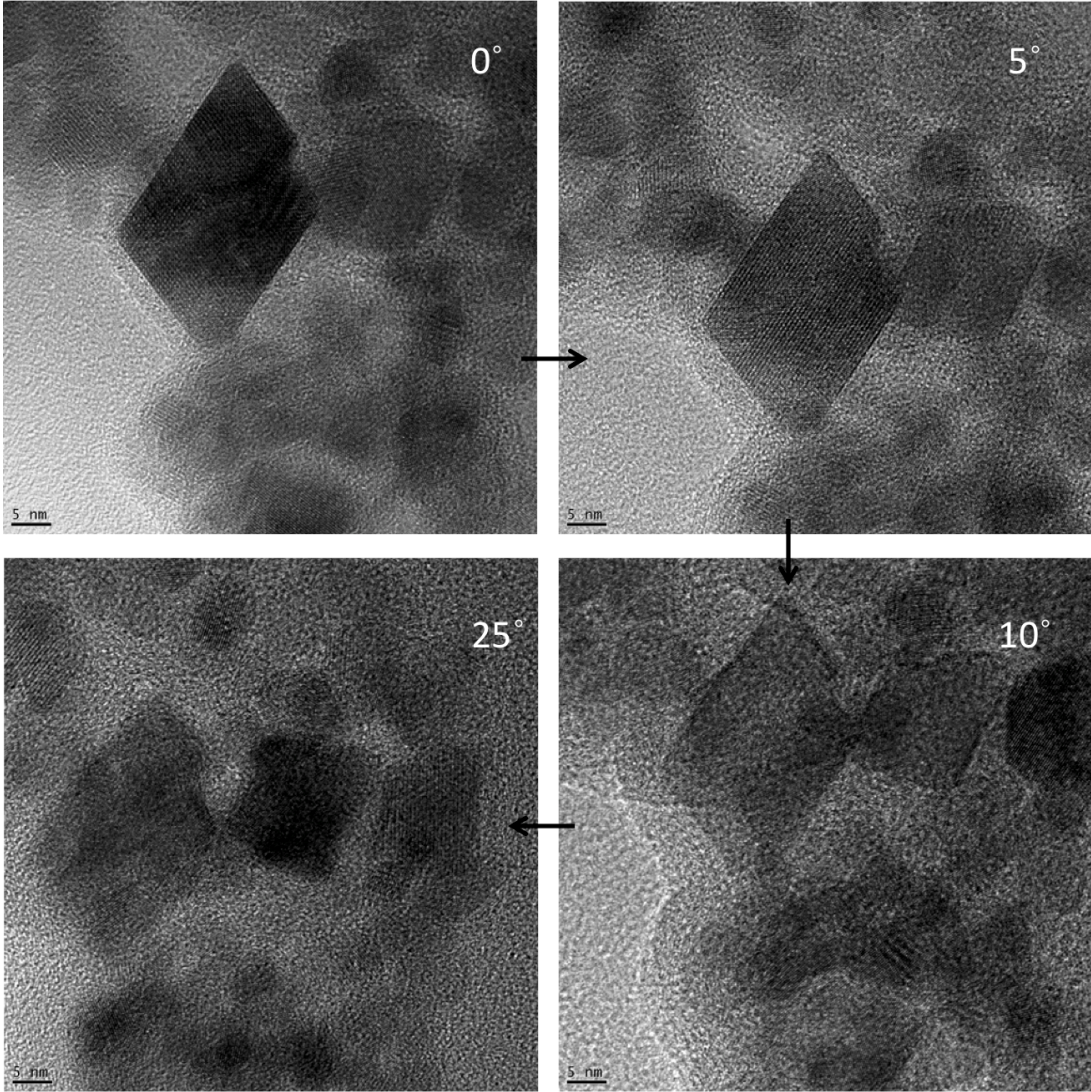
**S1:** POM images under crossed polarizers showing birefringent textures of lyotropic phases. **A** shows an image of a 50% mixture of Triton X45 in water at 35 °C; **B** shows the same along with a 2:1 mixture of  $\text{FeCl}_3$  and  $\text{FeCl}_2$ , which represents the condition of the reaction mixture before hydrolysis with NaOH occurs. Fingerprint textures typical of a lamellar phase can be seen in both. **C** shows an image of a 50% mixture of Triton X100 in water at 30 °C; **D** shows the same along with a 2:1 mixture of  $\text{FeCl}_3$  and  $\text{FeCl}_2$ . Focal conic textures typical of a hexagonal phase can be seen in both (limited transmission due to presence of iron salts), although the inclusion of the iron precursors does lower the transition temperature slightly.

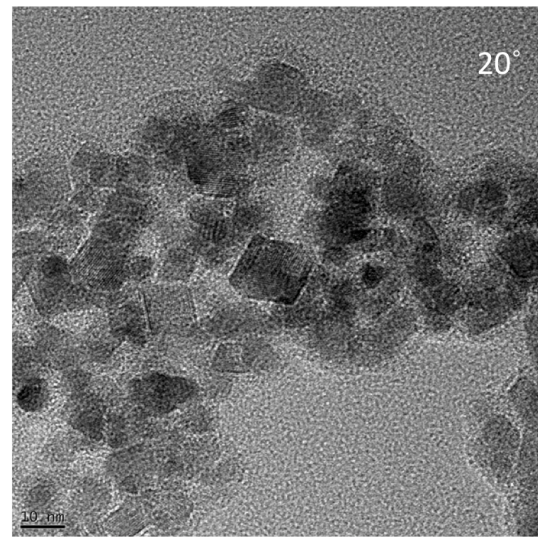
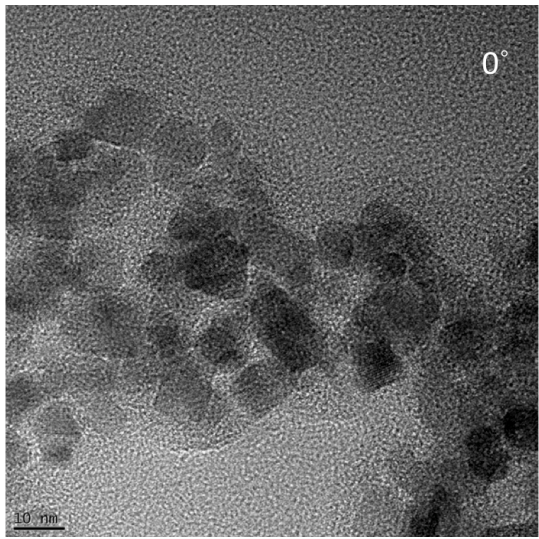
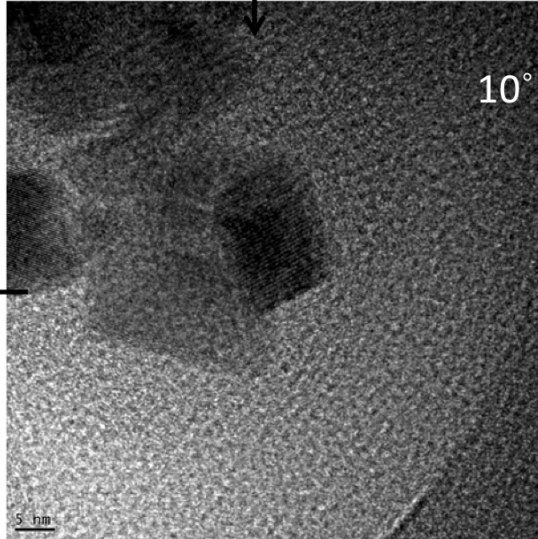
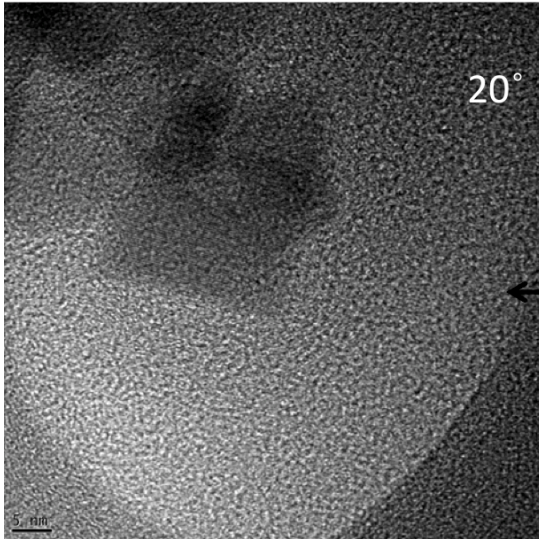
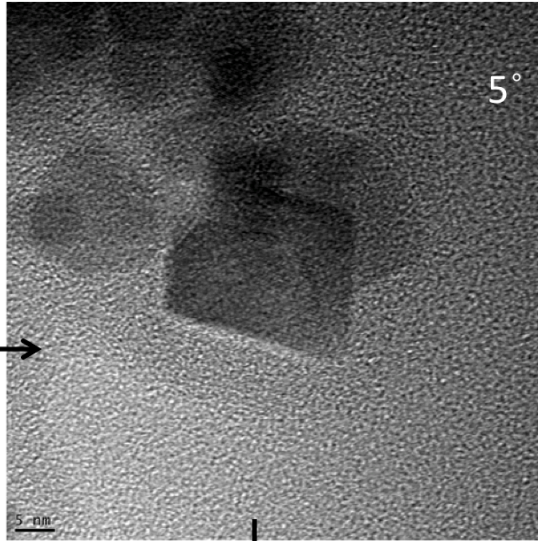
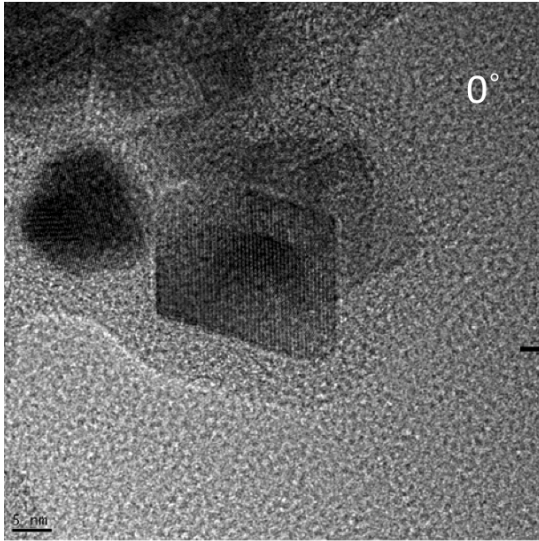


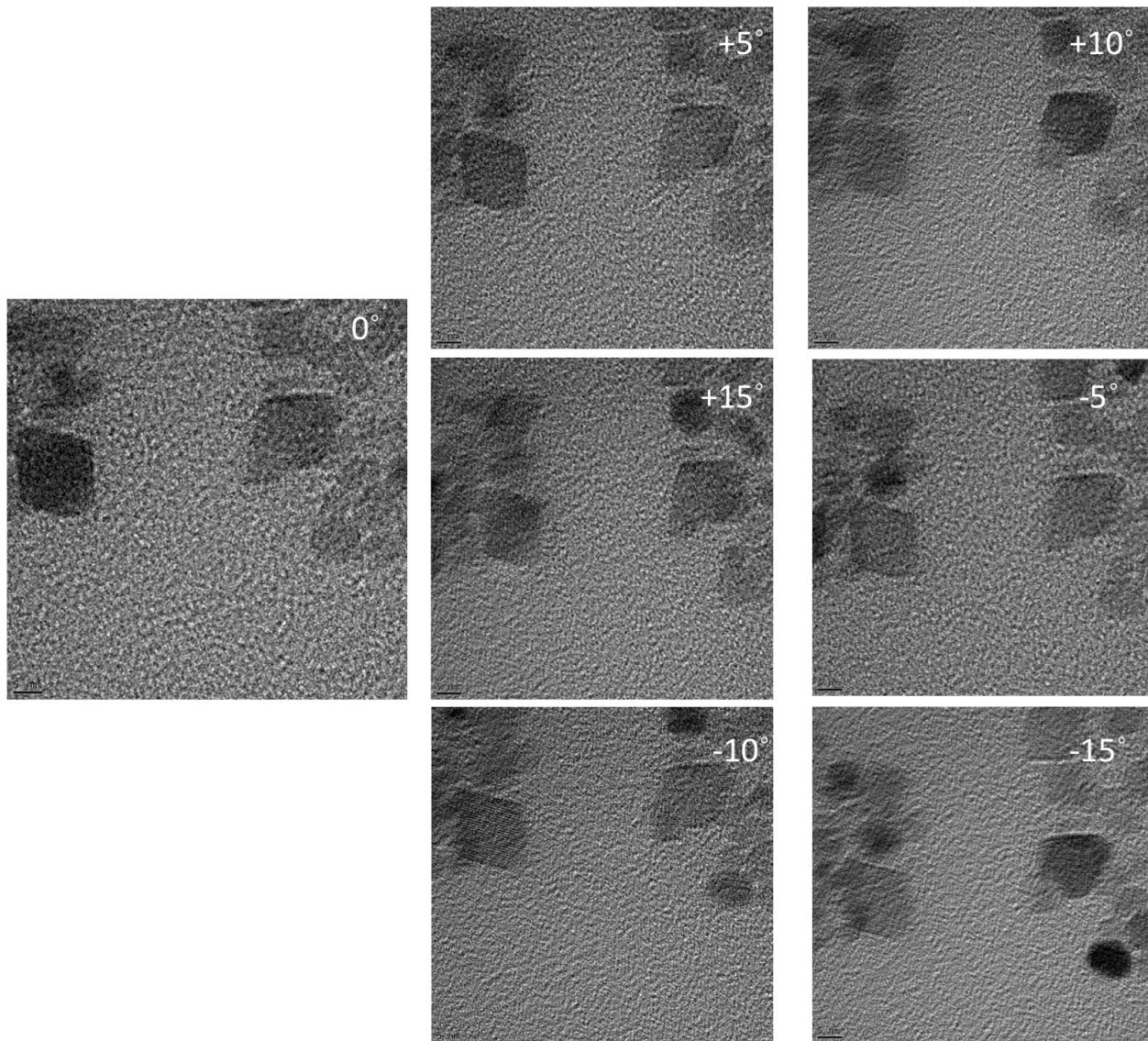
**S2:** FT-IR and TGA data for **S-IONBs**. Figures A and B show weight-loss vs. temperature profiles of **S-IONBsX100** (~27% weight loss) and **S-IONBsX45** (~46% weight loss), respectively. Figure C shows FT-IR spectra of **S-IONBsX45** (top) and **S-IONBsX100** (bottom). The broad peaks centered at ~3400 cm<sup>-1</sup> corresponds to O-H stretching; the sharp peaks at ~2915 and ~2852 cm<sup>-1</sup> correspond to C-H stretching; the broad peaks at ~1600 cm<sup>-1</sup> are indicative of COO- stretching; the broad peaks centered around ~1000 cm<sup>-1</sup> are due to Si-O-R stretching; and the large asymmetric peak at ~590 cm<sup>-1</sup> correspond to stretching modes associated with Fe<sup>2+</sup>-O and Fe<sup>3+</sup>-O.



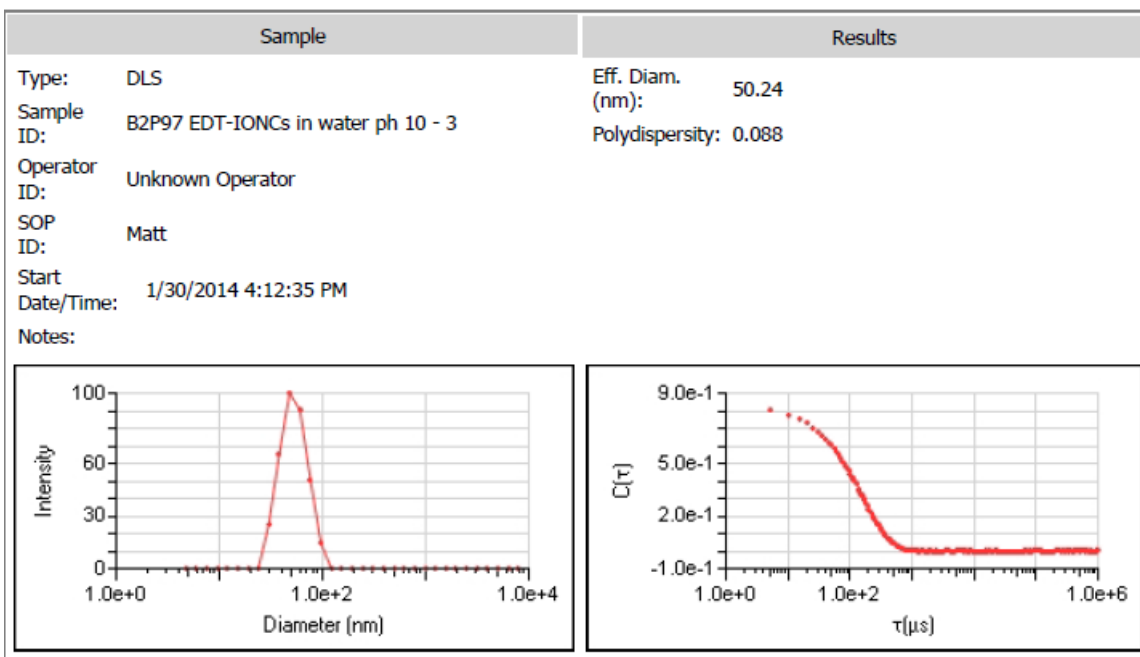
**S3:** TEM images showing particles from **IONBsX45** at different viewing angles. The top left set shows a typical rhombohedral particle, and the top right shows the same particle when viewed at an alpha (in plane) tilt of negative 20°. The bottom left shows a different rhombohedral particle, and the bottom right shows the same particle when viewed at an alpha (in plane) tilt of positive 20°. Both sets of images demonstrate how changes in viewing angle change the apparent internal angles of the particles and thus affect the evaluation of the particle morphology.



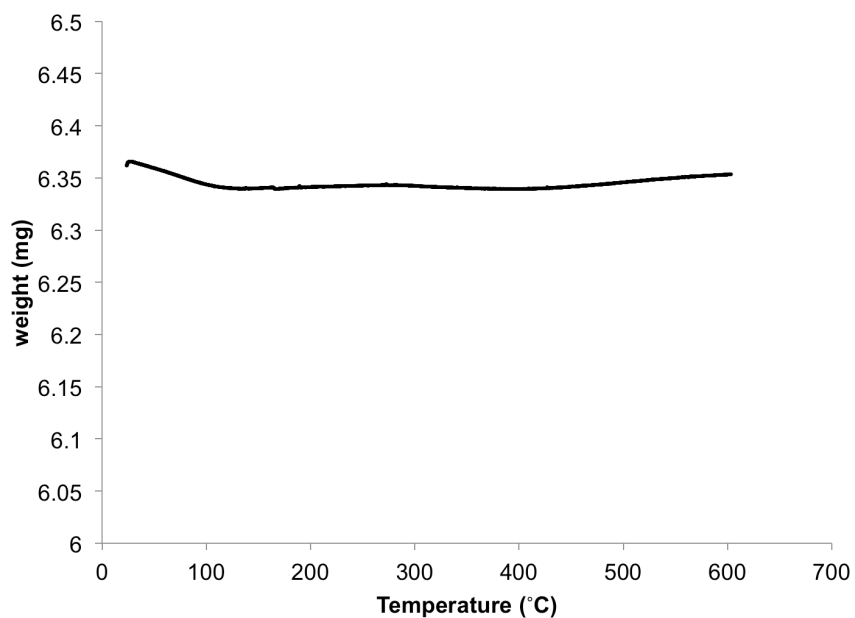




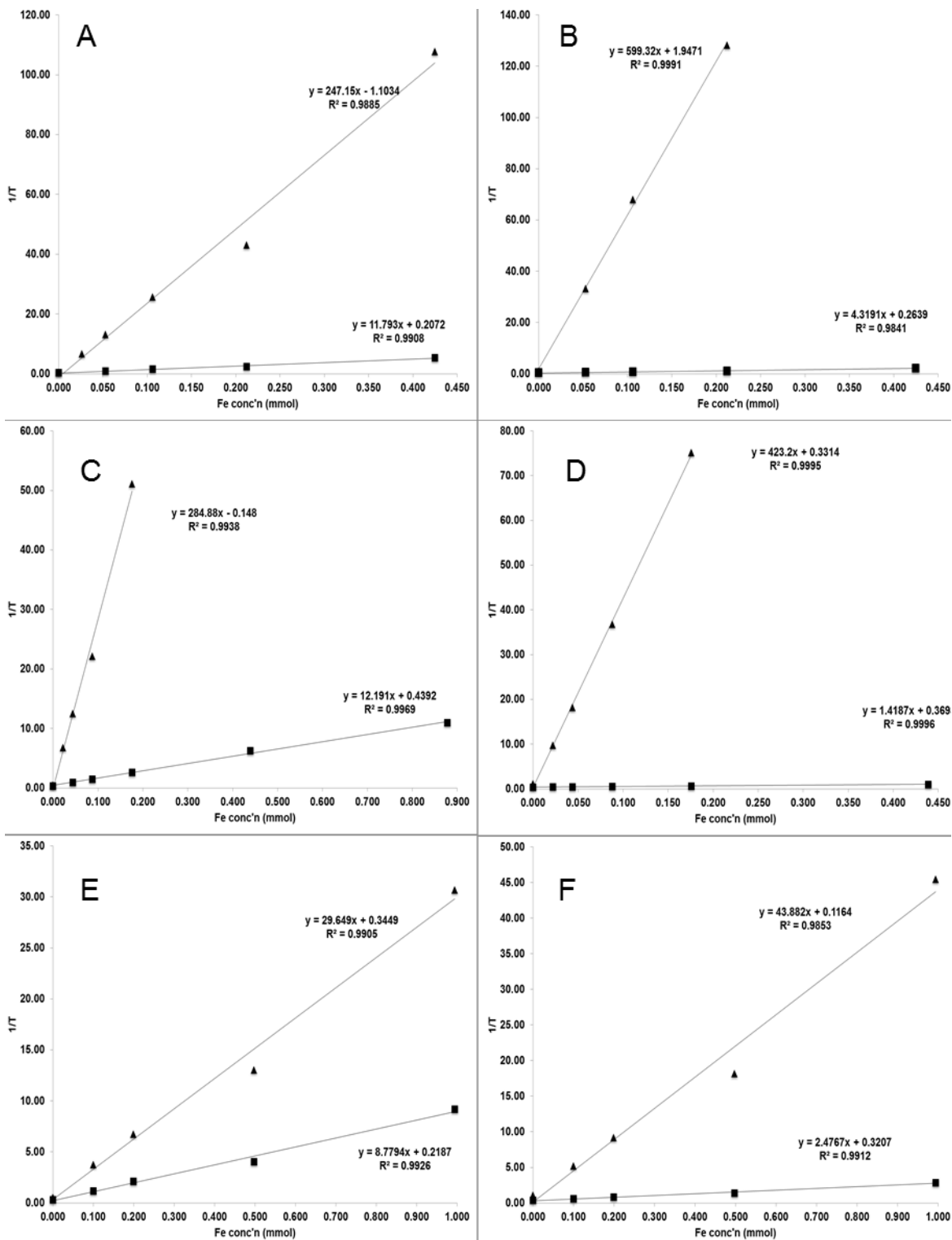
**S4:** Additional TEM images showing particles from **IONBsX45** at different viewing angles as indicated in the individual images (tilting is from left to right with respect to the images shown).



S5: DLS output file showing a plot of the size distribution of *S*-IONBsX45 in water.

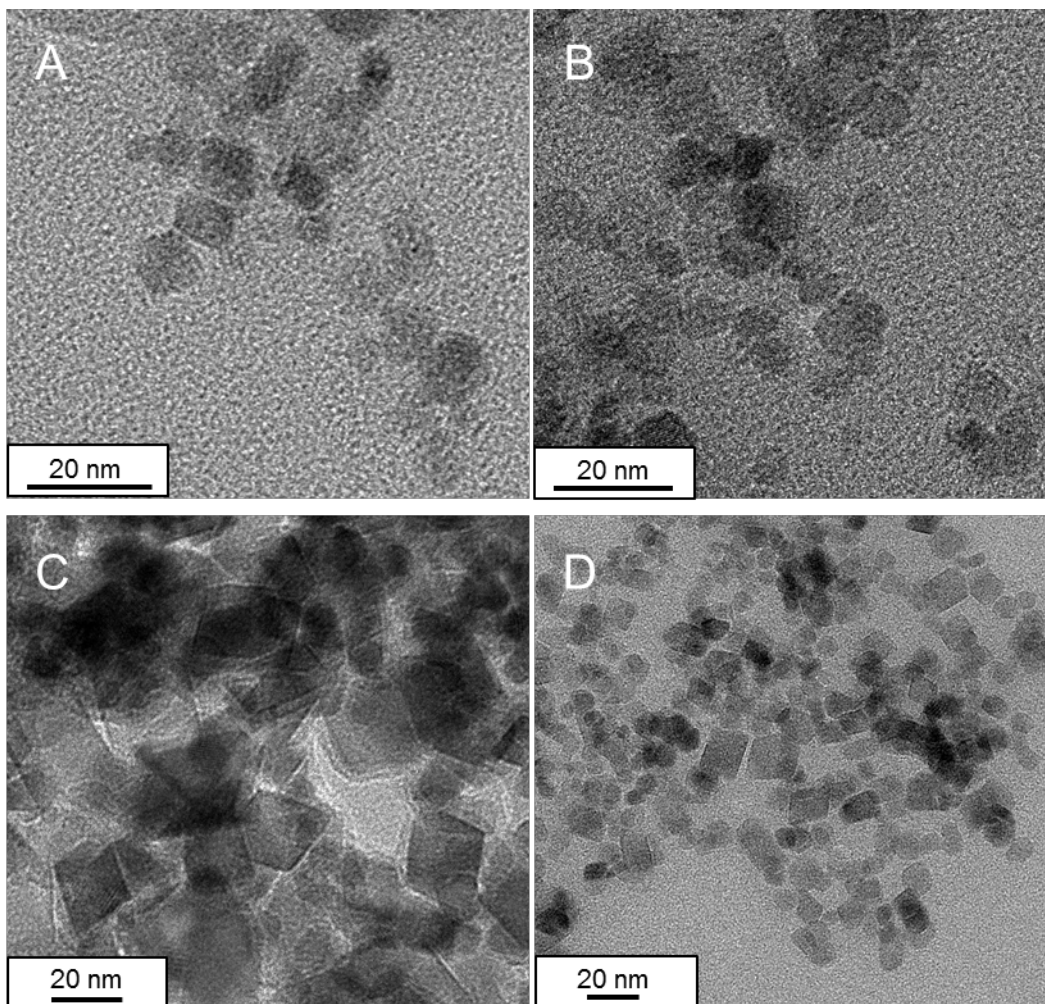


S6: TGA plot of bare **IONBsX45** (dried in a vacuum oven for 2 hours prior) showing effectively no weight loss, i.e. all surfactant molecules from the synthesis are washed off prior to conversion into the silanized IONBs (*S*-IONBs).



S7: Plots used to determine relaxivity values for various particles.  $y$ -Axes show the inverse of the relaxation time;  $x$ -axes show Fe concentration in mM. Figures A and B are for *S*-IONBsX100 at 1.5 T and 7 T, respectively; C and D are for *S*-IONBsX45; E and F are for *S*-IONPs. Triangle markers correspond to  $1/T_2$  values; square markers correspond to  $1/T_1$  values. Equations for determining the slope of each line are included in the inset for each plot.





S8: TEM images of *S-IONBsX100* (A, B), and *S-IONBsX45* (C, D).