

Supporting Information

Kinetics of Monodisperse Iron Oxide Nanocrystal Formation by “Heating-up” Process

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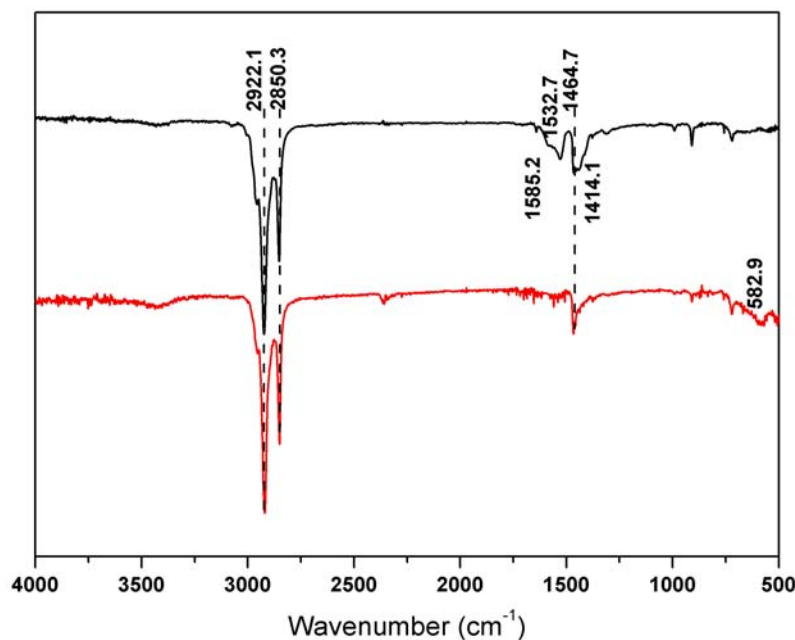


Figure S1. FTIR spectra of the reaction solution for the synthesis of iron oxide nanocrystals before (black line) and after (red line) the heating process. During the heating, the solution temperature was raised to 320 °C and held at that temperature for 30 min. The solvent used was 1-octadecene. The peaks at 2922.1 cm^{-1} and 2850.3 cm^{-1} are due to the C–H vibration of the alkyl chains of the solvent molecule and oleate ion. The peaks in the range from 1400 cm^{-1} to 1600 cm^{-1} are attributed to the carboxylate group of oleate ion coordinated to Fe ion. After heating, a new peak appeared at 582.9 cm^{-1} , which can be assigned as Fe–O vibration, indicating the formation of iron oxide nanocrystals.

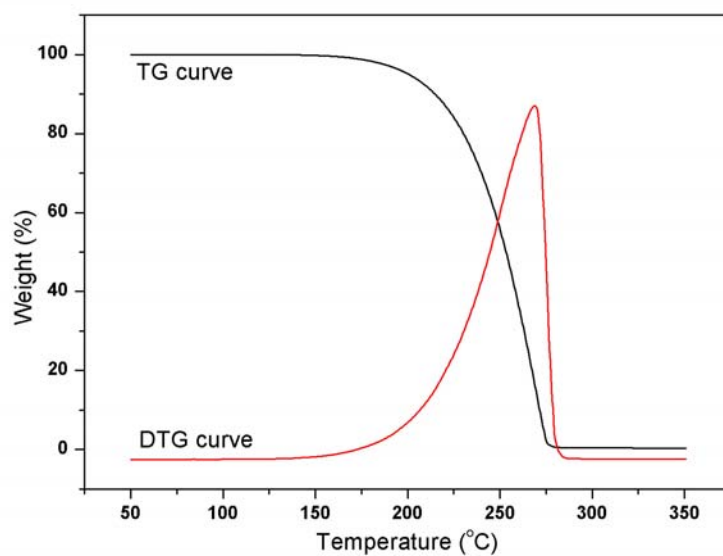


Figure S2. Thermal analysis data of pure oleic acid.

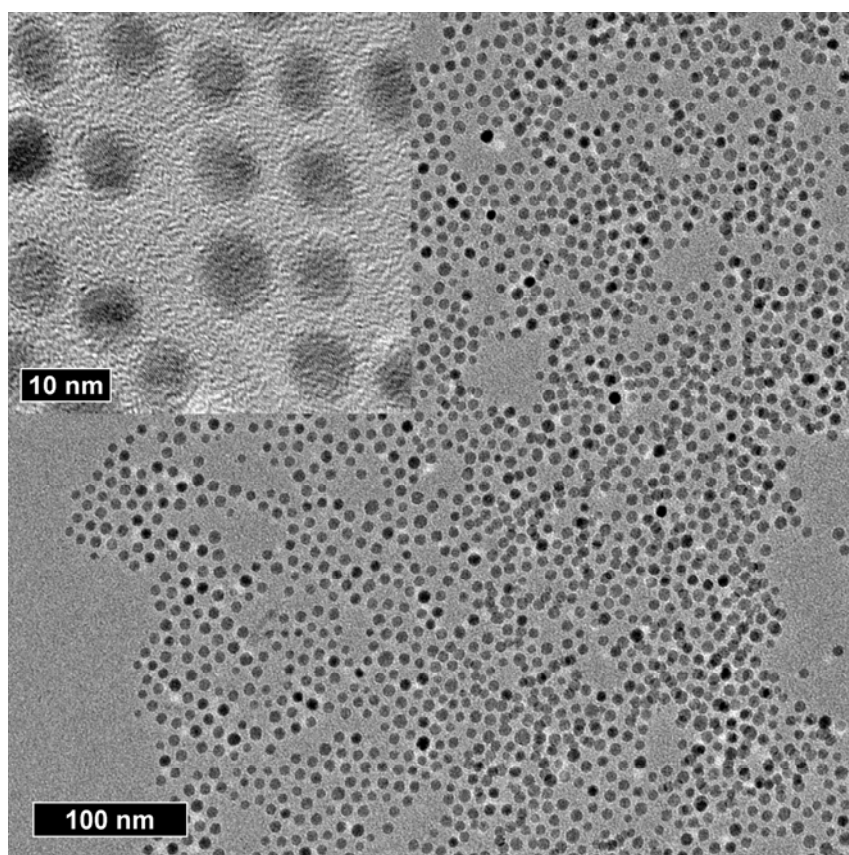


Figure S3. TEM images of iron oxide nanocrystals in the sample recovered after the TG-MS analysis.

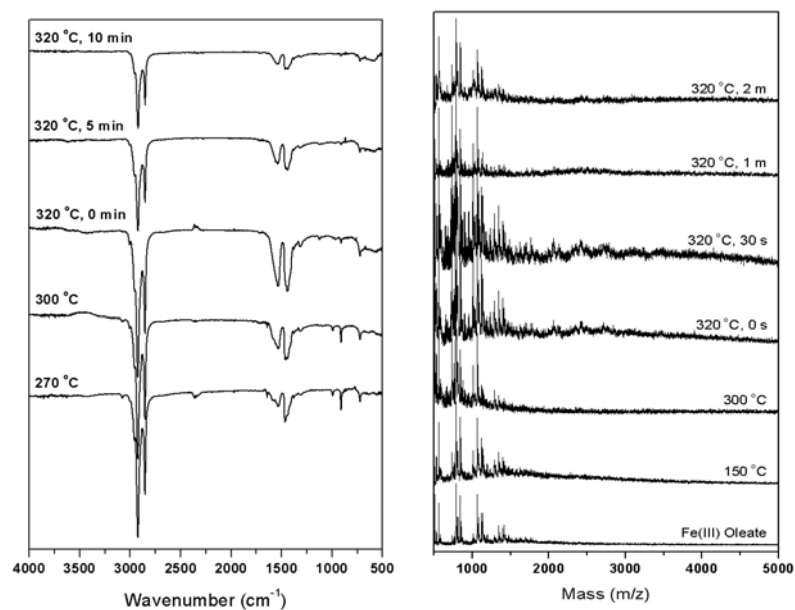


Figure S4. FTIR (left) and MALDI-TOF spectra (right) of the aliquots drawn from the solution during the heating process.

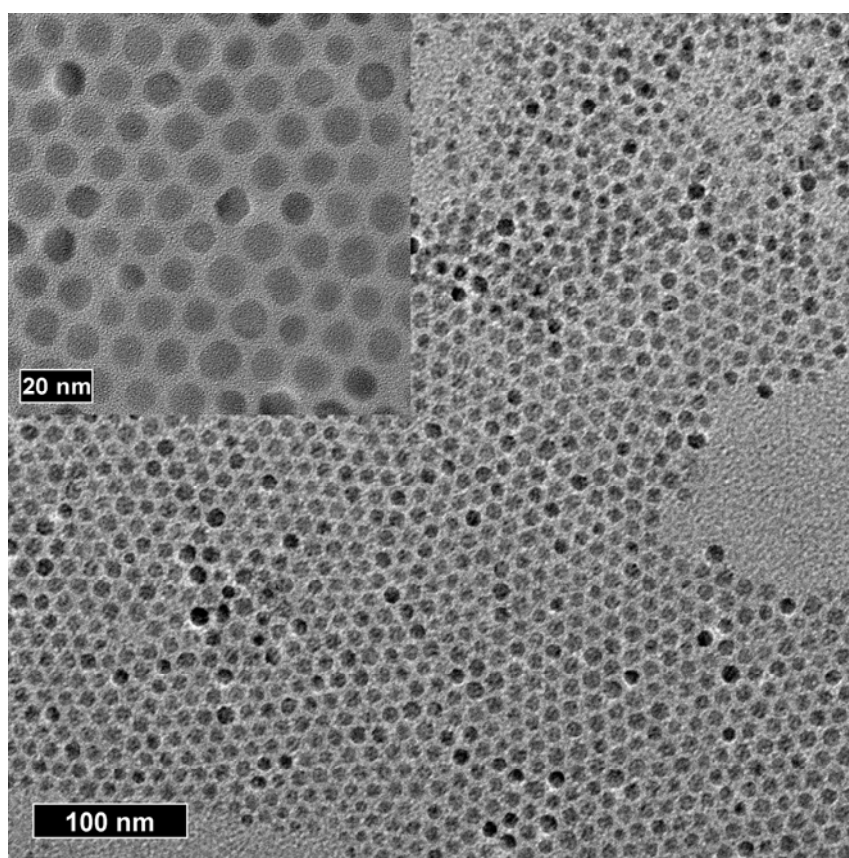


Figure S5. TEM images of iron oxide nanocrystals in the sample recovered after the in-situ SQUID measurement.

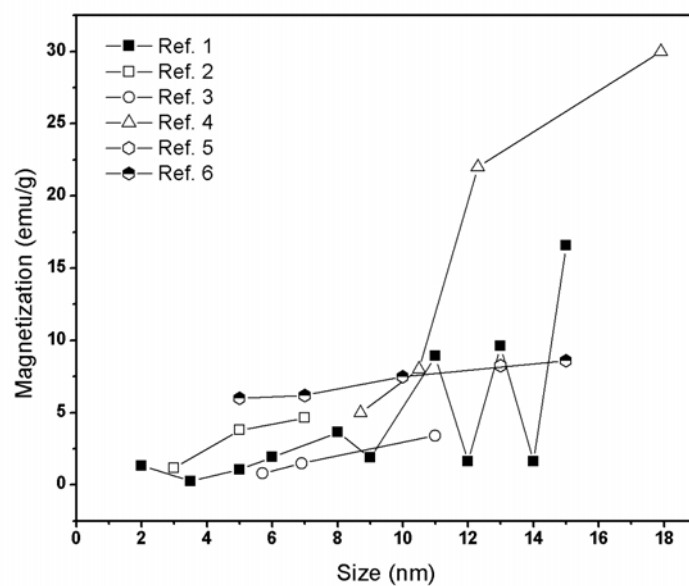


Figure S6. Plots of room temperature magnetization vs. size of various iron oxide nanocrystals (data taken from these 6 references).

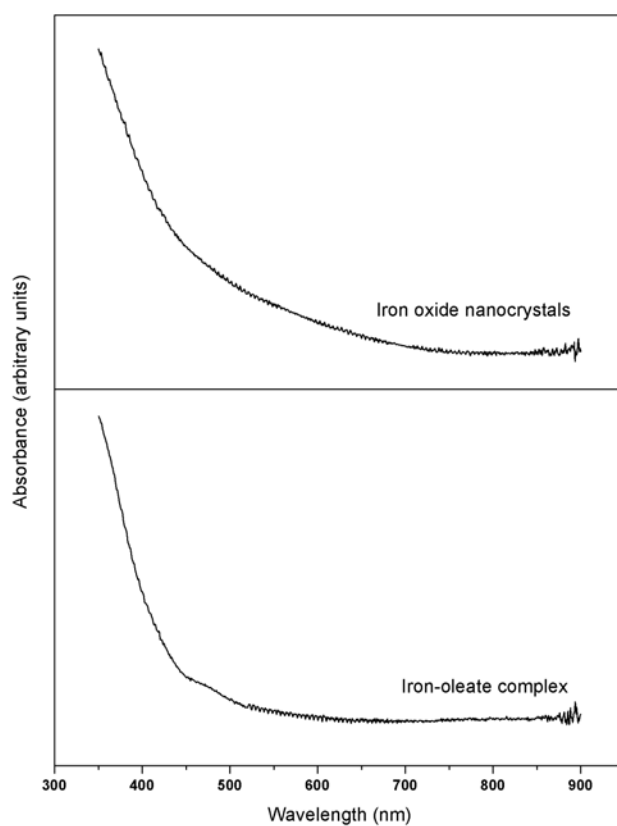


Figure S7. Optical absorption spectra of iron-oleate complex and iron oxide nanocrystals.

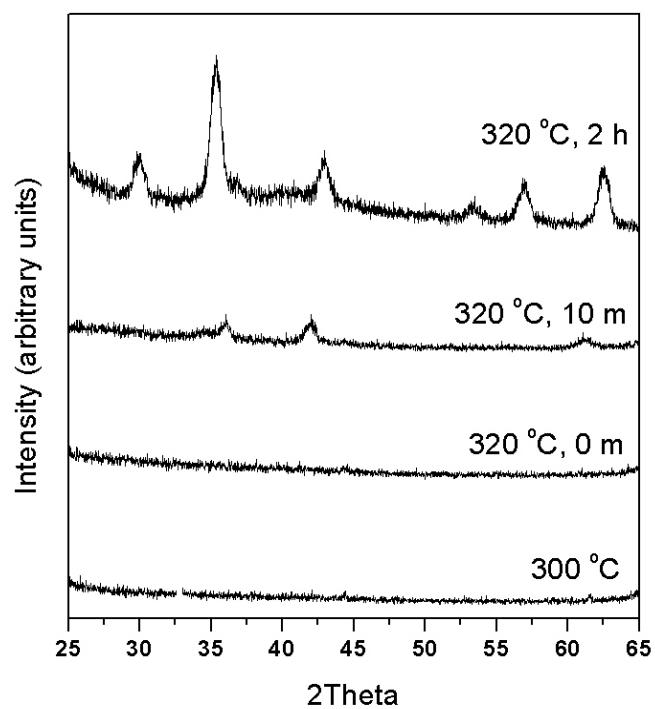
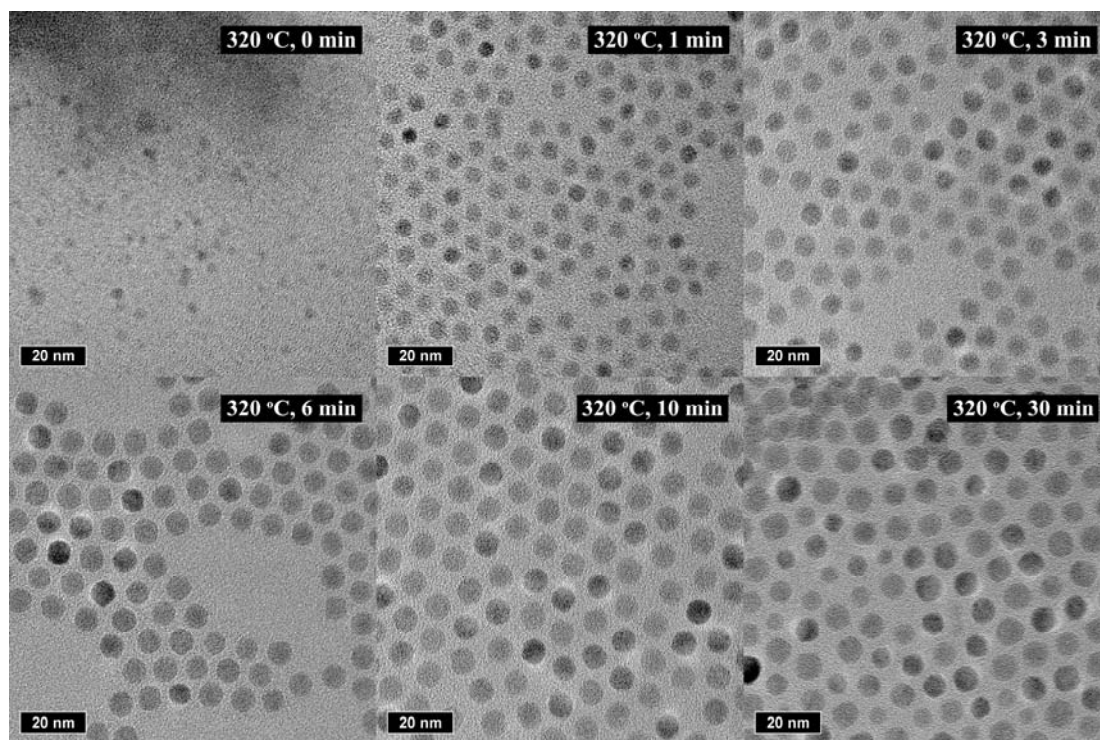


Figure S8. TEM and XRD data of the sample aliquots used for SEC analysis.

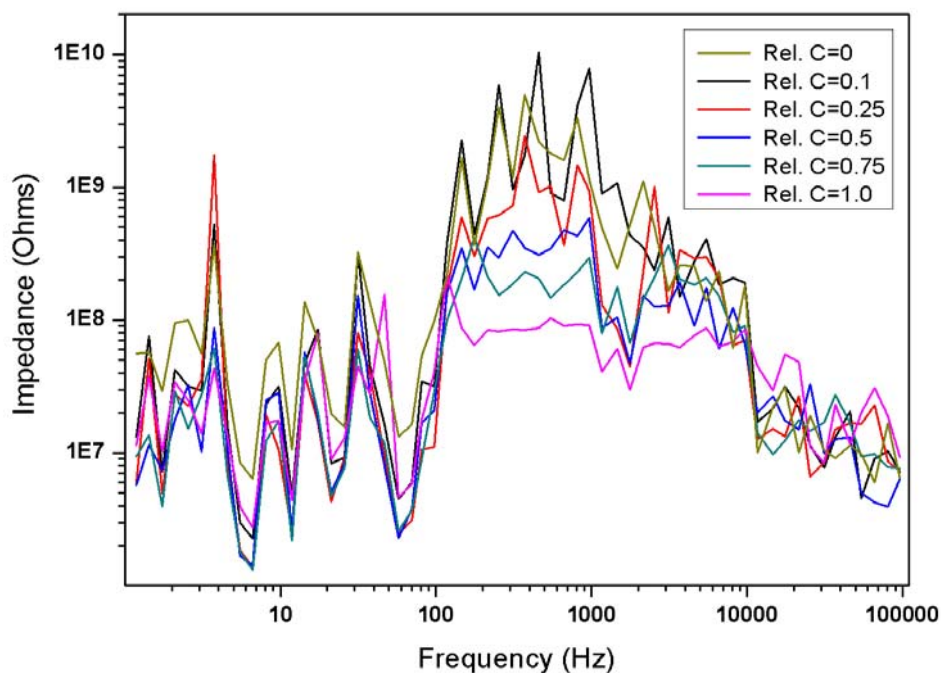


Figure S9. Bode plots of iron-oleate dispersions in THF (Rel. C means the relative concentration).

Table S1. Results for the recovery data with AC impedance method. Recovery experiments were performed by repeated measurements ($n = 5$) of reaction solutions spiked with different amounts of iron-oleate complex. The recoveries were calculated with reference to the calibration curve.

No.	Added relative iron-oleate concentration	Found/ relative iron-oleate concentration	Average	Recovery (%)	RSD (%)
1	0.25	0.25, 0.26, 0.25, 0.26, 0.27	0.26	104	3.2
2	0.30	0.32, 0.32, 0.29, 0.30, 0.31	0.31	103	4.2
3	0.50	0.49, 0.55, 0.52, 0.48, 0.50	0.51	102	5.4
4	0.60	0.61, 0.59, 0.58, 0.60, 0.58	0.59	98	2.2
5	0.80	0.81, 0.80, 0.78, 0.78, 0.77	0.78	98	2.1

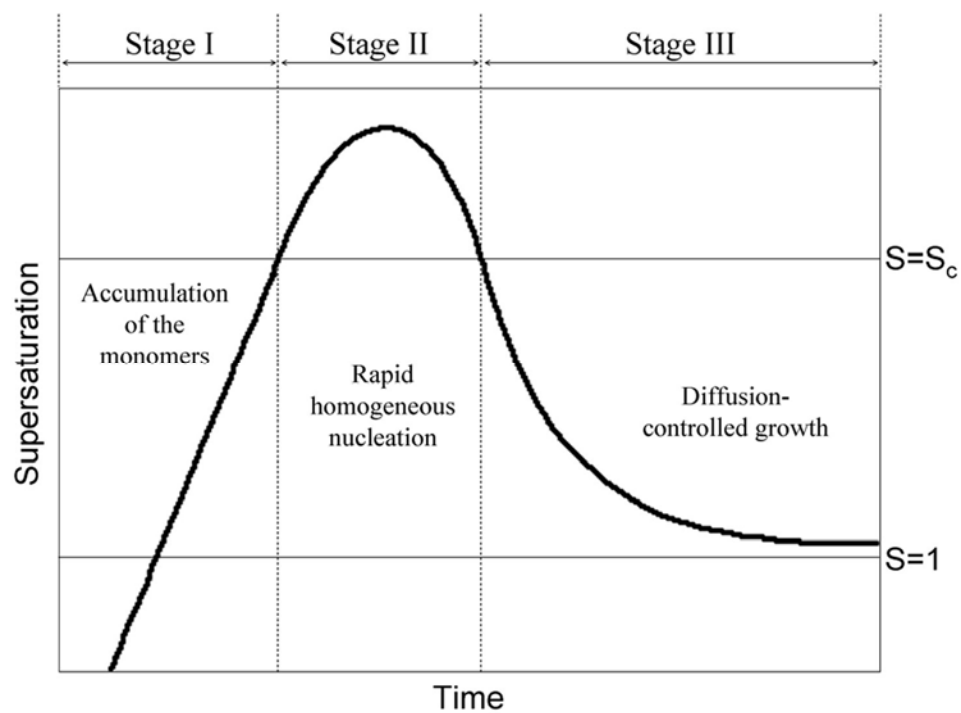


Figure S10. The LaMer diagram. S_c is the critical supersaturation, the minimum supersaturation level for the homogeneous nucleation.

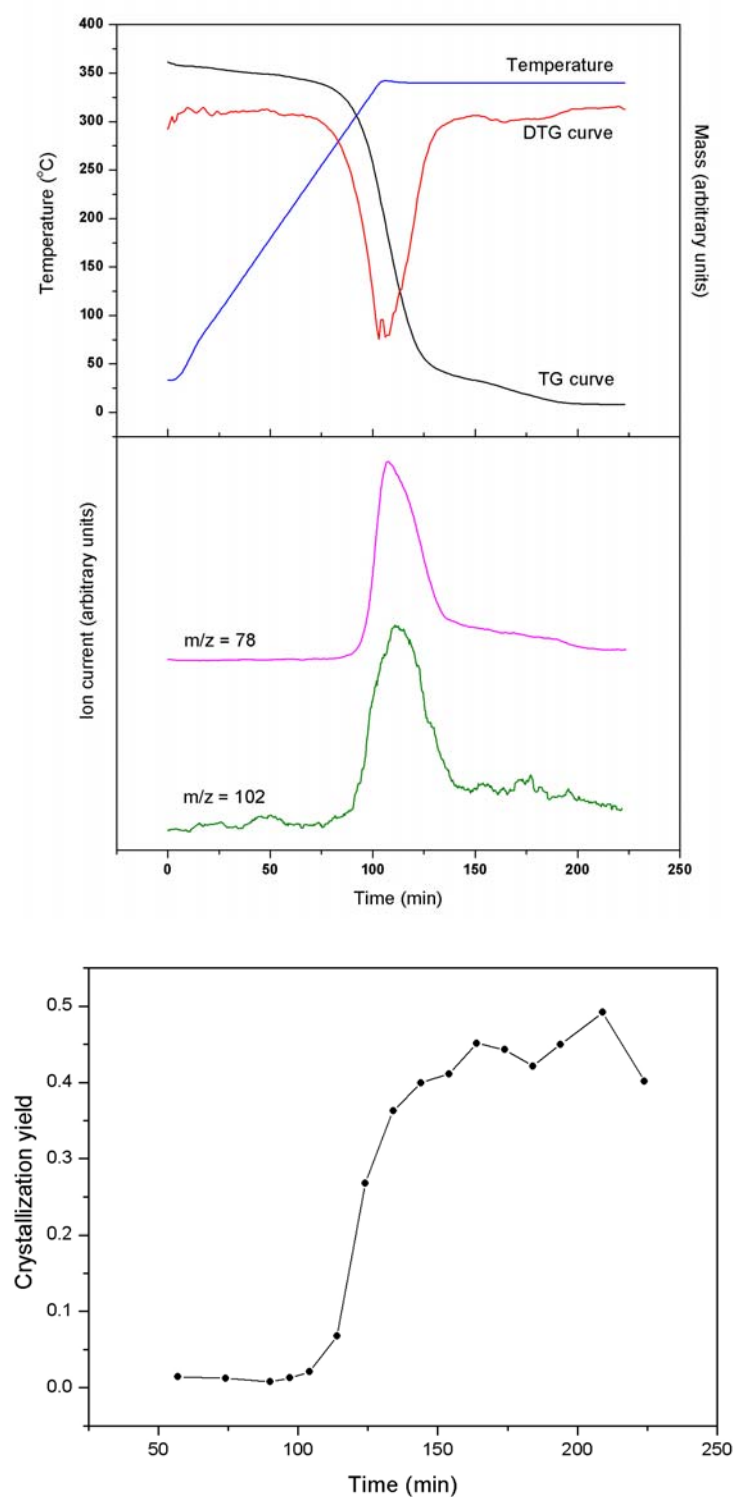


Figure S11. TG-MS and the crystallization yield data for the synthesis of zirconia nanocrystals. The synthetic procedure was the same as described in ref. 7. $m/z = 78$ and 102 are assigned to isopropyl chloride and isopropyl ether, respectively. They are the byproducts of the non-hydrolytic sol-gel reaction between zirconium(IV) isopropoxide and zirconium(IV) chloride. As a result, the evolution of those two

species indicates the formation of Zr-O-Zr link, which leads to the nucleation and growth of zirconia nanocrystals. Detailed discussion on the reaction mechanism is also presented in ref. 7. The heating procedures in TG-MS and the crystallization yield measurement are the same. From the data shown in this figure, it can be seen that the kinetics of the formation of zirconia nanocrystals is very similar to “heating-up” process of iron oxide nanocrystals.

References

- (1) Park, J.; Lee, E.; Hwang, N.-M.; Kang, M.; Kim, S. C.; Hwang, Y.; Park, J.-G.; Noh, H.-J.; Kim, J.-Y.; Park, J.-H.; Hyeon, T. *Angew. Chem. Int. Ed.* **2005**, *44*, 2872.
- (2) Lee, Y.; Lee, J.; Bae, C. J.; Park, J.-G.; Noh, H.-J.; Park, J.-H.; Hyeon, T. *Adv. Funct. Mater.* **2005**, *15*, 503.
- (3) Roca, A. G.; Morales, M. P.; O'Grady, K.; Serna, C. J. *Nanotechnology* **2006**, *17*, 2783.
- (4) Lin, C.-R.; Chiang, R.-K.; Wang, J.-S.; Sung, T.-W. *J. Appl. Phys.* **2006**, *99*, 08N710.
- (5) Wan, J.; Tang, G.; Qian, Y. *Appl. Phys. A: Mater.* **2007**, *86*, 261.
- (6) Thakur, M.; De, K.; Giri, S.; Si, S.; Kotal, A.; Mandal, T. K. *J. Phys.: Condens. Matter* **2006**, *18*, 9093.
- (7) Joo, J.; Yu, T.; Kim, Y. W.; Park, H. M.; Wu, F.; Zhang, J. Z.; Hyeon, T. *J. Am. Chem. Soc.* **2003**, *125*, 6553.