Supplementary Information

## Doping of Inorganic Materials in Microreactors - Preparation of Zn doped Fe<sub>3</sub>O<sub>4</sub> Nanoparticles

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## **Supplementary Figures**

**Supplementary Figure 1**: The unit cell of  $ZnFe_2O_4$  with the blue sphere representing the  $O^{2-}$  anions, the grey and brown polyhedra the Zn-O tetrahedra and the Fe-O octahedra respectively.



**Supplementary Figure 2**: The equipment used for the preparation of  $Fe_3O_4$  and the  $Fe_{3-x}Zn_xO_4$  (x = 0.27, 0.37, 0.48) series. a) the microreactor, pumps and thermal controller; b) the reactor (Type 3025); c) the microreactor holder.

a)



b)



c)



**Supplementary Figure 3**: Mössbauer spectrum of pure Fe<sub>3</sub>O<sub>4</sub> recorded at 80K



**Supplementary Figure 4**: Mössbauer spectra recorded at T = 4.2 K for Fe<sub>3</sub>O<sub>4</sub> and the  $Zn_xFe_{3-x}O_4$  (x = 0.27, 0.37, 0.48) series.



Supplementary Table 1: Mössbauer parameters from spectra recorded at 4.2 K for Fe<sub>3</sub>O<sub>4</sub> and the Fe<sub>3-x</sub>Zn<sub>x</sub>O<sub>4</sub> (x = 0.27, 0.37, 0.48) series.

Sample	Hyperfine field (T)	Isomer shift (mm/s)	Quadrupole shift (mm/s)	Line width (mm/s)
Fe <sub>3</sub> O <sub>4</sub>	$49.8 \pm 0.11$	$0.334 \pm 0.001$	$-0.023 \pm 0.001$	$0.439 \pm 0.002$
$Zn_{0.27}Fe_{2.73}O_4$	$50.33 \pm 0.25$	$0.348 \pm 0.002$	$-0.018 \pm 0.002$	$0.801 \pm 0.001$
$Zn_{0.37}Fe_{2.63}O_4$	$50.49 \pm 0.11$	$0.347 \pm 0.001$	$-0.018 \pm 0.002$	$0.514 \pm 0.002$
Zn <sub>0.48</sub> Fe <sub>2.52</sub> O <sub>4</sub>	$49.9 \pm 0.18$	$0.340 \pm 0.002$	$-0.022 \pm 0.003$	$0.437 \pm 0.003$

## Supplementary Information

The spectra were least-squares fitted using the program package "Pi" developed by Ulrich von Hörsten.<sup>1</sup> Different fitting procedures were applied to the spectra, a free distribution of hyperfine fields ( $B_{hf}$ ) with 52 equidistantly spaced subspectra, and also a relaxation model, to test whether any relaxation effects are still detectable in parts of the spectra. As no satisfactory fit could be obtained via the relaxation model, it is safe to assume that no superparamagnetic relaxation is taking place at this temperature, with all samples being below their characteristic blocking temperature for this measurement method.

1 https://www.uni-due.de/~hm236ap/hoersten/home.html (accessed 18.12.2014)