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Stoichiometry of Fe₃O₄ Nanoparticles Determined by Mössbauer Spectroscopy HIEN-YOONG HAH, MICHAEL COLE, SHARON GRAY, CHARLES JOHNSON, JACQUELINE JOHNSON, The University of Tennessee Space Institute, VLADIMIR KOLESNICHENKO, PAVEL KUCHERYAVY, GALINA GOLOVERDA, Xavier University of Louisiana — The detailed composition of nanoparticles is dependent on the method of preparation. Previous Mössbauer studies of Fe_3O_4 nanoparticles have shown that the samples were not pure Fe_3O_4 . In most cases they were γ - Fe_2O_3 , which has the same spinel structure as Fe_3O_4 but with the Fe^{2+} ions oxidized to Fe^{3+} and vacancies to ensure charge neutrality. In other cases the oxidation was incomplete and the structure contained defects on the octahedral (B) sites. In this work the samples of diameters 5.3 and 10.6 nm were prepared from colloidal magnetite in diethylene glycol under an inert atmosphere. The Mössbauer spectra showed that they are superparamagnetic with blocking temperatures, T_B , of 50 K and 120 K respectively. The spectra at 6 K, the lowest temperature studied, were close to those of bulk crystalline Fe_3O_4 , in particular showing lines due to Fe^{2+} which were absent in many previous studies.

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