In this work, the crystallographic structure and its changes under thermal treatment of different systems consisting of metal oxide nanoparticles is studied. The principal method used throughout the thesis is x-ray powder diffraction enriched with grazing incidence small angle x-ray scattering when the nanoparticles form an ordered structure or with x-ray absorption spectroscopy when additional information on local crystallographic structure is required. For all the systems the preparation conditions were optimized according to the crystallographic data for further applications.