Fe-Co nanoparticles were prepared from the double Fe- and Co- oxalate. The formation of FeCo metallic phase (solid solution) begun at 250 °C as determined from the X-ray diffraction spectra recorded during heating of the oxalate sample in reducing atmosphere. The reduction finished at 500 °C by formation of \( \alpha \)-FeCo phase with the mean particle size of about 17 nm. The final product was investigated using Mössbauer spectroscopy, transmission electron microscopy (TEM), and magnetic measurements. Analysis of Mössbauer spectra shows the ferromagnetic \( \alpha \)-FeCo phase represented by two six-line patterns corresponding to interiors and surfaces of the nanoparticles. The hyperfine inductions of these components are 34.06 for the first one and 35.03 T with hypefine field distribution 34.22 T for the second one. In addition to a weak paramagnetic component represented by the doublet with isomer shift 0.14 mm/s and quadrupole splitting 0.49 mm/s was found. The doublet was ascribed to fine particles in superparamagnetic state. The TEM has shown conglomerates of particles with composition about 50 at. % Fe and 50 at. % Co.