An analytical method for the determination of Ni-63 and Fe-55 in nuclear waste samples such as graphite, heavy concrete, aluminium and lead was developed. Different decomposition methods (i.e. ashing, acid digestion and alkali fusion) were investigated for the decomposition of the samples and for the separation of Fe and Ni from the matrix. Hydroxide precipitation was used to separate Fe-55 and Ni-63 from the matrix elements and ion exchange chromatography was used to separate Fe-55 and Ni-63 from the interfering radionuclides as well as from each other. The separated Ni-63 was further purified by extraction chromatography. The purified Ni-63 and Fe-55 was then measured by liquid scintillation counting. The chemical yields of the separation procedures for Fe-55 and Ni-63 are above 90% and the decontamination factors for all interfering radionuclides are more than 10(5). The detection limits of the analytical method for Fe-55 and Ni-63 are 0.018 and 0.014 Bq, respectively. The methods developed were used in the analysis of Fe-55 and Ni-63 in heavy concrete, aluminium and lead from two concrete cores and graphite from thermal column in the Danish research reactor DR-2 and the results are presented in this paper. (c) 2004 Elsevier B.V. All rights reserved.