The experimental determination of the tracer or self-diffusion coefficient as a function of composition can be quite burdensome in alloys due to the use of isotopes and the number of experiments required to assess composition and temperature dependence. A new formalism recently developed by I.V. Belova et al., based on linear response theory combined with the Boltzmann–Matano method, and proposed to be used for obtaining the composition dependent tracer and interdiffusion coefficients simultaneously from a single, isotope free sandwich type diffusion experiment. In this study, experimental demonstration of this new formalism is carried out. Thin film of pure Cu is deposited by e-beam evaporation technique on a selected alloy (e.g., Cu-50Ni), sandwiched between two other alloys (e.g., Cu-75Ni) and annealed isothermally at 900 and 1000°C. After annealing, the couples were water quenched, cross-sectioned, and prepared for compositional characterization. Scanning Electron Microscopy was employed to examine the interdiffusion zone. Energy Dispersive X-ray Analysis was conducted to obtain concentration profiles for quantitative analysis. The self- and inter-diffusion coefficients simultaneously determined using the experimental methodology based on the new formalism produced results consistent with previously reported values determined independently by radiotracer and interdiffusion experiment.