A combination of organo-analytical methods allows for determination of kinetics of simple reactions, but a formation of complex mixture during a multistep, intermediate-involving reactions, such as cross-couplings, remains challenging. To avoid interference of side products and synthetically unessential intermediates with analysis, we designed a method to track substrates that participate in these reactions. In this work, we present the use of multiple-radioisotope labelled reagents approach that allows for a better distinction of synthetically relevant intermediates using radioactive detector-coupled HPLC (radio-HPLC). Palladium catalyzed methylation of 4-acetylphenylboronic acid was used as a model reaction. Multiple radioactively labelled molecules have been observed in HPLC chromatogram when using carbon-11 ($^{11}$C), carbon-14 ($^{14}$C), and iodine-131 ($^{131}$I) labelled methyl iodide, including starting $[^{11}C/^{14}C/^{131}I]CH_3I$ and final $[^{11}C/^{14}C]4$-methylacetophenone. Identities of intermediates were indirectly determined by radio-HPLC and the presence of proposed compounds confirmed by HRMS studies of reaction mixtures. Kinetic study with $[^{11}C]$- and $[^{14}C]CH_3I$ provided information on rate of reaction and kinetic isotope effect of different reaction steps. Currently, studies with $[^3]H4$-acetylphenylboronic acid are taking course to provide a view on the reaction from the standpoint of arylboronic acid.