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 \([^{11}\text{C}/^{14}\text{C}/^{131}\text{I}]\text{CH}_3\text{I}\) and final \([^{11}\text{C}/^{14}\text{C}]4\text{-methylacetophenone}\). 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 \([^{11}\text{C}]\) and \([^{14}\text{C}]\text{CH}_3\text{I}\) provided information on rate of reaction and kinetic isotope effect of different reaction steps. Currently, studies with \([^{3}\text{H}]4\text{-acetylphenylboronic acid}\) are taking course to provide a view on the reaction from the standpoint of arylboronic acid.