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Extended Internal Standard Method for Quantitative ^1H NMR Assisted by Chromatography (EIC) for Analyte Overlapping Impurity on ^1H NMR Spectra

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Abstract

We devised a novel extended internal standard method of quantitative ^1H NMR (qNMR) assisted by chromatography (EIC) that accurately quantifies ^1H signal areas of analytes, even when the chemical shifts of the impurity and analyte signals overlap completely. When impurity and analyte signals overlap in the ^1H NMR spectrum but can be separated in a chromatogram, the response ratio of the impurity and an internal standard (IS) can be obtained from the chromatogram. If the response ratio can be converted into the ^1H signal area ratio of the impurity and the IS, the ^1H signal area of the analyte can be evaluated accurately by mathematically correcting the contributions of the ^1H signal area of the impurity overlapping the analyte in the ^1H NMR spectrum. In this study, gas chromatography and liquid chromatography were used. We used 2-chlorophenol and 4-chlorophenol containing phenol as an impurity as examples in which impurity and analyte signals overlap to validate and demonstrate the EIC, respectively. Because the ^1H signals of 2-chlorophenol and phenol can be separated in specific alkaline

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