

## 定量 NMR (qNMR) による日本薬局方試薬の純度規格化の検討

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Trial Study to Determine Absolute Purities of Chemical Reagents Used  
as Reference Standards in the Japanese Pharmacopoeia  
by Using Quantitative NMR (qNMR)

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## Summary

In the "Crude Drugs" section of the Japanese Pharmacopoeia (JP), many chemical reagents that are commercially available and chemically specified in the section on "Reagents and Test Solutions" are used as reference standards for quantitative analyses. However, there is no information on the absolute purity of these standards because it is very difficult to obtain pure natural compounds and to determine their purity with International System of Units (SI) traceability. Therefore, in a strict sense, quantitative regulation of crude drugs and related products in JP by the use of such marker compounds is somewhat ambiguous.

Recently, quantitative NMR (qNMR) using a certified reference material as a qNMR reference has been developed. This method qualifies as an absolute quantification method and is theoretically able to determine the purity of any compound with SI traceability. Therefore, we are proposing to introduce the qNMR method to JP for the specification of reagents used as marker compounds. In this report, in order to clarify practical issues that must be solved before the adoption of qNMR by JP, we applied qNMR to reagents (and a naturally purified compound) which are known to have wide-ranging purity. We found that the selection of the specific NMR signal(s) for calculating the purity of the target compound was very important. It is our view that a simple non-exchangeable signal such as a singlet or doublet should be selected and that the numbers of selected signals should be modified depending on the level of purity of the target compound. Coexisting signals from impurities in the integration section cause integration errors, and increasing the number of the selected signals consequently enhances the likelihood of accurate integrations. Further studies and intensive discussion in the panel on crude drugs in JP are needed to reach a final consensus on this issue.

## Key words

Quantitative NMR, International System of Units traceability, the Japanese Pharmacopoeia, Chemical reagents, Marker compounds, Crude drugs