

定量 NMR を利用した機能性関与成分の定量に用いる 市販試薬の絶対純度

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Determination of absolute purities of commercial agents used for the quantification of functional substances by quantitative NMR analysis

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Abstract

The new system of Foods with Function Claims (FFCs) have been launched in April 2015. FFCs are foods submitted to the Secretary-General of the Consumer Affairs Agency as products whose labels bear function claims based on scientific evidence on food safety and effectiveness, under the responsibility of the manufacturers. More than 1000 FFCs have been submitted as of November 2017. In a registered report to the Secretary-General, the manufacturers are required to describe in detail the analytical method for the quantitative determination of functional substances (FSs) to ensure the quality control of FFCs. Usually, the quantification of FSs are performed by HPLC analysis using commercial available laboratory grade agents as reference standards. Up to now, the purities of commercial available laboratory grade agents are also determined by an HPLC.

Lately, a quantitative NMR (qNMR) have been developed as the absolute quantification method to determine the purities of organic compounds, including pure natural compounds, which are traceable to the International System of Units (SI).

In this study, we determined the absolute purities of two commercial available laboratory grade agents, glabridin and cyanidin 3-*O*-glucoside, used for the quantification of FSs by ¹H-qNMR analysis. Glabridin is an ingredient of licorice, cyanidin 3-*O*-glucoside is one of the anthocyanins and an ingredient of bilberry and black currant etc.

The absolute purities of glabridin and cyanidin 3-*O*-glucoside were 98.02% and 87.22%, respectively. It was revealed that their absolute purities determined by ¹H-qNMR analysis were lower than their purities estimated from area percentage of main peak under HPLC analysis (>99.7% and ≥ 99%, respectively). The lower absolute purities of the two natural compounds by ¹H-qNMR analysis might be caused by contamination of impurities including water and solvents which are difficult to detect by HPLC. Therefore, the ¹H-qNMR analysis would be a reliable method for the accurate purity determination of the commercial reagents used as reference.

Keywords : 定量 NMR (qNMR)、絶対定量、グラブリジン、シアニジン-3-*O*-グルコシド、機能性関与成分
quantitative NMR (qNMR), absolute quantification, glabridin, cyanidin 3-*O*-glucoside, functional substances

I 緒言

平成 27 年 4 月に「機能性表示食品」制度が始まったが、機能性表示食品は、事業者の責任において、科学的根拠に基づいた機能性を表示した食品であり、販売前に安全性及

び機能性の根拠に関する情報などが消費者庁長官へ届け出られたものである。ただし、特定保健用食品とは異なり、消費者庁長官の個別の許可を受けたものではないと定義されている¹⁾。平成 29 年 11 月現在、既に 1000 種類以上の機能性表示食品の届け出がある。また、機能性表示食品の届け出