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水蒸気蒸留およびHPLCによる食品中のソルビン酸分析の不確かさおよび頑健性

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Measurement uncertainty and robustness of sorbic acid determination in food samples by steam distillation and HPLC

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Abstract

Japanese official method for sorbic acid determination in food samples was evaluated for its measurement uncertainty and robustness. Smoke-dried cuttlefish was used as a model food sample. As trueness estimated in terms of recovery with spiked blank sample was within the acceptable range, within-laboratory reproducibility based on repeated independent analyses of the sample with incurred analyte at about half of the regulatory standard was thought to be the good estimate of measurement uncertainty.

Robustness of steam distillation procedure with respect to seven factors such as weight of the sample, speed of distillation, final volume of distillate was evaluated according to Plackett-Burman experimental design, while reproducibility of HPLC procedure was by analysing the data of repeated independent determinations of the extract by two-way nested ANOVA considering isolation columns and calibration curves as two factors. The distillation procedure was found to be quite robust against the chosen modifications of various factors, and most part of the HPLC procedure variability was attributable to the variation of calibration curves.

As the method is used for analysis of a range of sample matrices and analyte concentrations, data of sorbic acid determinations of spiked samples that consisted of various matrices and various concentrations were evaluated as a whole. The recovery rates were within the range of 70% to 100%, and relative standard deviations of repeatability were all within the acceptable ranges calculated from Horwitz equation. It seemed that variability of mean recovery rates would be a useful parameter to evaluate equivalence between different methods.

Keywords : 不確かさ、頑健性、ソルビン酸、枝分かれ実験計画
uncertainty, robustness, sorbic acid, nested experimental design

I　はじめに

近年、食品分析の分野でも注目されつつある「分析値の不確かさ」は、測定結果の信頼性を定量的に示すものであるが、検査方法の性能に固有の要因のみならず、検査員の熟練度や機器の管理状況なども含んだ総合的な指標であり、この値を把握しておくことは個々の試験室における検査結果の信頼性の維持、向上のために重要といえる[1]。また、検査法の頑健性とは、「同じサンプルが異なる条件で分析された時の分析法の再現性を示す性能」あるいは「通常の分析条件での変動が測定結果に影響を与えない性能」と定義される指標であり[2,3]、一連の分析工程における個々のステップの頑健性を評価しておくことは、迅速で正確な作業遂行のための重要な情報である。またこれらの項目は国際的な単一試験室における妥当性評価ガイドラインにおいても評価すべき項目として挙げられている[4]。

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