



Quantification of analytes affected by relevant interfering signals under quality controlled conditions

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Abstract

The analysis of organic contaminants or residues in biological samples is frequently affected by the presence of compounds producing interfering instrumental signals. This feature is responsible for the higher complexity and cost of these analyses and/or by a significant reduction of the number of studied analytes in a multi-analyte method.

This work presents a methodology to estimate the impact of the interfering compounds on the quality of the analysis of complex samples, based on separative instrumental methods of analysis, aiming at supporting the inclusion of analytes affected by interfering compounds in the list of compounds analysed in the studied samples.

The proposed methodology involves the study of the magnitude of the signal produced by the interfering compounds in the analysed matrix, and is applicable to analytical systems affected by interfering compounds with varying concentration in the studied matrix. The proposed methodology is based on the comparison of the signals from a representative number of examples of the studied matrix, in order to estimate the impact of the presence of such compounds on the measurement quality. The treatment of the chromatographic signals necessary to collect these data can be easily performed considering algorithms of subtraction of chromatographic signals available in most of the analytical instrumentation software. The subtraction of the interfering compounds signal from the sample signal allows the compensation of the interfering effect irrespective of the relative magnitude of the interfering and analyte signals, supporting the applicability of the same model of the method performance for a broader concentration range. The quantification of the measurement uncertainty was performed using the differential approach, which allows the estimation of the contribution of the presence of the interfering compounds to the quality of the measurement.

The proposed methodology was successfully applied to the analysis of pesticide residues in spiked oranges considering the quantification of the oranges ethyl acetate extract by gas-chromatography with electron capture detector. The application of the proposed methodology to the analysis of this fruit using the studied chromatographic system, allowed the quantification of an increased number of analytes in the samples.

The magnitude of the measurement uncertainty estimated by the proposed methodology is fit for the purpose of monitoring pesticide residues in oranges since, frequently, the difference between the maximum residue level and the best estimation of the sample content is larger than the respective uncertainty.

The proposed methodology can be useful in other analytical fields and/or instrumental methods of analysis.

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Abbreviations: GC-ECD, gas-chromatography with electron capture detector; GPC, gel-permeation-chromatography; $IP_{[IPs]}$, estimation of the interpolation uncertainty based on a study of the interpolation performance conducted in intermediate precision conditions; $IP_{[RM]}$, estimation of the interpolation uncertainty based on the regression model; MRL, maximum residue level; Spk–Blk, subtraction of the unspiked sample signal from the respective spiked sample signal; Spk–Blk_{Dif}, subtraction of an unspiked sample signal from the spiked sample signal considering oranges of different origins

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