Main complications connected with detection, identification and determination of trace organic constituents in complex matrix samples

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A R T I C L E   I N F O

A B S T R A C T

It is well known that some problems with the determination of organic analytes at trace level can occur. This issue is connected with contamination during each stage of the analytical procedure from sampling to sample preparation up to chromatographic analysis, which often leads to false-positive or overestimated results. Another problem associated with determination of analytes occurs at trace- and ultra-trace level is a background problem which is mainly dictated by techniques, glassware and solvents. This review provides information on main complications connected with determination of trace organic constituents in complex matrix samples. Error sources in the field of determination of trace analytes are described in detail. In addition, the type of the background in each of the stapes of analytical procedure is summarized.

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1. Introduction

In the end of XIX century, Wilhelm Ostwald, defined analytical chemistry as the art of separating, recognizing different substances and determining the constituents of a sample [1]. Since this time, analytical chemistry evolved from art to a branch of chemical science of greatest theoretical and practical utility for medicine, industry and in general for all applied sciences [2]. Over the years, applications of analytical chemistry in many different areas such as food science, pharmaceutical science, wastewater technology, etc. led to the development of analytical approaches aimed to the global characterization of a thousand type of samples to define their physico-chemical and sensory characteristics as well as to ensure their quality and safety for consumers, supporting and implementing the laws and regulations of the many fields.

Nowadays it can be stated that analytical chemists are philosophizing and moreover, they are using many new logical terms and ideas to describe population distributions at near zero concentrations, where the quantized nature of matter dominates [3]. These new definitions and concepts are required to estimate the limits of detection and quantification and the capability of an analytical method, equipment, and connect them with its selectivity, sensitivity and optimization [3,4]. The evolution and development of separation techniques like extraction, cloud point extraction, solid and liquid phase microextraction techniques were also of high importance [5].

In recent years, design and discover completely new analytical methodologies and reference materials that will constitute the tools for obtaining reliable analytical information become of major importance. This is due to the fact that analytical chemistry frequently requires the determination of sub-ultra-trace xenobiotics in samples, which are very often characterised by a complex matrix composition (Fig. 1).

Without a doubt it need to be stated that in the last 7 decades, analytical chemists succeeded to introduce many improvements in their work as well as cost and time saving procedures. And so, several classical methods such as gravimetric and titrimetric analyses are now in most cases replaced by instrumental techniques which can produces two- and even multi-dimensional information [6]. Moreover, several new approaches with advance analytical features have been introduced. Old applications which let the determination of a single or at most two analytes have been improved or replaced by others which allow to simultaneous determination of dozens of analytes at quantities of a few nanograms, picograms or even femtograms [6].

This improvement is of high importance as the application area of trace- and ultra-trace analysis is extensive (Fig. 2). In addition,

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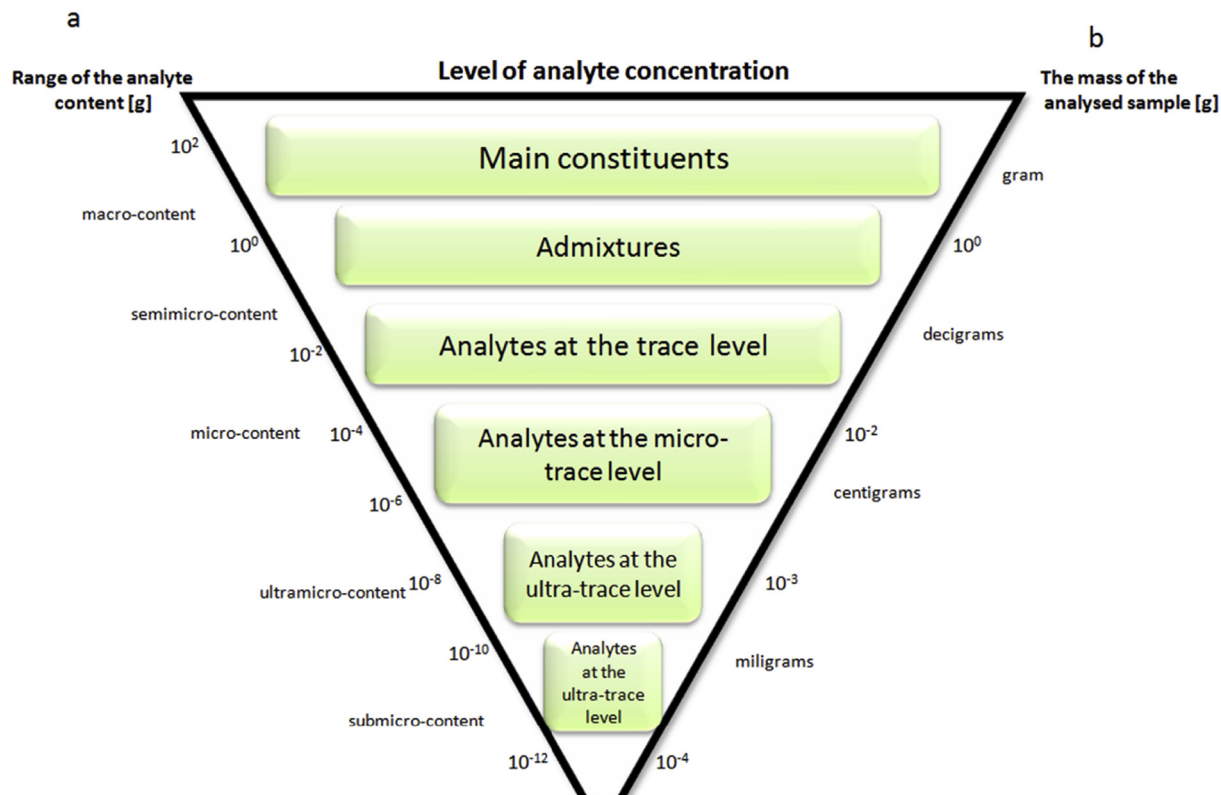
Fig. 1. Schematic classification of different analytical approaches from the point of view of: a) range of analyte content in different types of sample of representation of directions in the development of analytical chemistry; b) size (mass or volume) of sample for analysis.

the consideration of noises and interferences of the analytical signal, the strategies of their removal as well as the concepts of sensitivity, limit of detection (LOD), limit of decision, response time, etc. are treated in some cases mainly by applying logical ideas, for example different tools of chemometric analysis [7].

It is well known that some problems with determination of analytes that occur in a sample at trace-level of concentration exist. The various operations carried out during sample preparation procedure which is very often required prior to final determination, can be a source of many errors crucially affecting the final result of analysis [7,8].

Another problem associated with determination of analytes occurs at trace- and ultra-trace level is a background problem. Two types of background problem are known: an instrumental or constants background, and a sample- or volume-dependent background [9]. The later is the most important and problematic.

This review provides information on an important topic which is the problem associated with determination of organic compounds occur in the sample at trace- and ultra-trace level of concentration. Error sources in the field of determination of trace analytes are described in detail. In addition, the type of the background in each of the stages of analytical procedure is summarized.

To the best of our knowledge, no review article related to problem of background and its value estimation in determination of trace organic analytes published in last few years exist. It will be useful for the readers not only because it presents the problem of background but mainly, it shows how to solve this problem.

2. Type of the background in each of the stage of analytical procedure

It is well known that different types of blanks exist and can be distinguished on every stage of analytical method existed [5]. These are presented on Fig. 3. A proper preparation and analysis of these blanks may provide valuable information on characteristics of overall background noise and interferents. Field blank and trip blank contribute to evaluating contamination connected to sampling technique, preservatives applied and transportation. Field blank should be prepared on matrix matched to the sample type, without addition of analytes standard solution. Trip blanks may be prepared in the field or in laboratory and then transported along with samples.

Method blanks provide information on contamination that may occur during sample preparation techniques under laboratory conditions. These kind of blanks are prepared in the same way as actual samples, without analyte addition. Reagent blanks are useful for determination of analytes concentration in specific reagents used at sample preparation stage. Finally, instrument blank is used to identify contamination that origins from system components. General characteristics of samples used in the stage quality control of analytical results are presented in Fig. 4. More detailed description of each blank and background problems are discussed in the next paragraphs of this paper.

It also needs to be mentioned that the important issue that impact on the background error is the location of the analytical instrument in relation to the tested object/sample. The following