

SAMPLE PREPARATION FOR FLAME ATOMIC ABSORPTION SPECTROSCOPY: AN OVERVIEW

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ABSTRACT

Sample preparation is an important step in chemical analysis, from time and reagent consuming point of view and from the probability of errors. The present article gives an overview of recently most used techniques in sample preparation for flame atomic absorption spectroscopy. Wet and dry sample decomposition techniques, separation and pre-concentration methods of the target analyte(s) have been discussed.

Keywords: Sample preparation, FAAS, decomposition, separation, pre-concentration

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INTRODUCTION

Elemental analysis of the majority of organic and inorganic matrices requires the partial or total dissolution of the sample prior to instrumental analysis.

Analysis by spectroscopic methods practically always necessitates a simple or more complex preparation of the sample. These steps are generally the most critical part of analysis because they are responsible for the most important errors.¹⁻³

Only a few direct methods allow the introduction of the sample without any preparation. In these cases the lack of reliable calibration is the major problem. On the other hand, sample preparation allows the separation and/or pre-concentration of analytes and makes possible the use of several determination methods.

Sample preparations involve digestion, extraction and preparation of the analytes before the analysis, so this step is time limiting, requiring ca. 61% of the total time to perform the complete analysis, and is responsible for 30% of the total analysis error. Nowadays the goals to be reached are the best results, in the shortest time, with minimal contamination, low reagent consumption and generation of minimal residue or waste.¹

Sample preparation was probably the single most neglected area in analytical chemistry relatively to the great interest in instruments. While the level of sophistication of the instrumentation for analysis has increased significantly, a comparatively low technical basis of sample preparation often remains.⁴

Sample preparation and development of methods have now become a growing field along with instrumental improvements. There are drastic improvements in the detection power of measurement techniques used. Consequently, the analyst also realizes that it is often no longer mandatory to resort to laborious, dubious and time-consuming separation or pre-concentration steps in the sample preparation procedures.²

It is normal in atomic spectroscopy for the sample to be found in one of two forms solid or liquid. The liquid case would seem to be the easiest form in which to handle the sample, with maybe a requirement for filtration being all that is required. However, the inherent lack of sensitivity of many spectroscopic techniques and the need to carry out determinations at lower and lower levels means that invariably some form of pre-concentrations is required. If the sample is in a solid form, the normal requirement is to convert it into the liquid form although it is possible to analyse solids directly by using the atomic spectroscopy, but this is not the preferred approach.⁴

The principal objectives of sample preparation for residue analysis are: isolation of the analytes of interest from as many interfering compounds as possible, dissolution of the analytes in a suitable solvent and pre-

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