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Review

High-resolution continuum source atomic absorption spectrometry for the simultaneous or sequential monitoring of multiple lines. A critical review of current possibilities

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ABSTRACT

This work examines the capabilities and limitations of commercially available high-resolution continuum source atomic absorption spectrometry instrumentation for multi-line monitoring, discussing in detail the possible strategies to develop multi-element methodologies that are truly simultaneous, or else sequential, but from the same sample aliquot.

Moreover, the simultaneous monitoring of various atomic or molecular lines may bring other important analytical advantages, such as: i) expansion of the linear range by monitoring multiplets; ii) improvements in the limit of detection and in precision by summing the signals from different lines of the same element or molecule; iii) simple correction for matrix-effects by selecting a suitable internal standard; or iv) accurate mathematical correction of spectral overlaps by simultaneous monitoring of free lines of the interfering molecule or element. This work discusses how authors have made use of these strategies to develop analytical methodologies that permit the straightforward analysis of complex samples.

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1. Introduction

The introduction of high-resolution continuum source atomic absorption spectrometers (HR CS AAS) represents the most significant advance experienced by this technique in various decades [1]. This instrumentation became commercially available in 2003, equipped with a flame atomizer only, while in 2007 a spectrometer featuring

both a graphite furnace and a flame atomizer in two separate sampling compartments was released, although the research groups of Becker-Ross and Welz already published several works using prototypes of similar characteristics before those dates [2,3]. As can be seen in Fig. 1, the number of articles that evaluate the use of continuum source instruments for atomic absorption is finally growing, which is particularly obvious after the introduction of the commercial device equipped with the graphite furnace. Moreover, there is also a clear tendency to publish more applied work, even outside atomic spectroscopy journals, which demonstrates that the technique is maturing very fast and it is ready to be used in routine labs.

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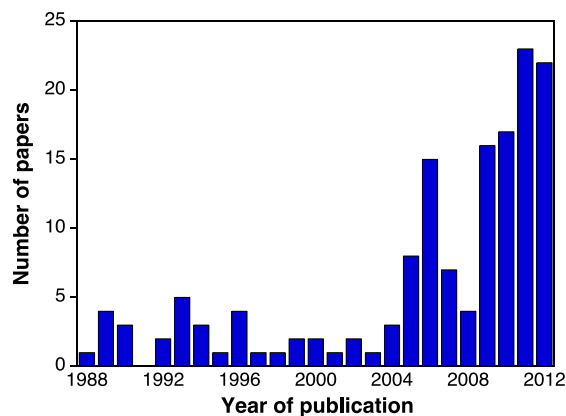


Fig. 1. Number of publications that have reported the use of continuum source atomic absorption spectrometry during the last 25 years. Papers that appeared in proceedings and book chapters are not included. Source: ISI Web of Science.

Use of continuum source-based atomic absorption equipment is not really a novel topic in the scientific literature. However, during the last five decades line source AAS was the technique of choice, following the basic idea proposed by Walsh [4]. This situation is logical because the correspondence between atomic emission and absorption lines permitted great selectivity to be attained even for instruments with limited resolution (nm level), while for successfully using a continuum source device a much better resolution was required. However, owing to the possible advantages derived from the use of a continuum source (e.g., higher multi-element capabilities), this idea was never abandoned, and several authors (notably J. Harnly) developed their own prototypes as the technical capabilities of spectrometers were improving [5–11]. These prototypes showed some limitations, and, in particular, it was difficult to find a suitable lamp with sufficient intensity in the far UV.

Becker-Ross and co-workers [12–15] presented a device based on (1) a high-pressure xenon short-arc lamp operating at brightness temperatures of approximately 10,000 °C, capable of providing a high intensity in the visible and (far) UV region, (2) an optical system based on an echelle monochromator dispersing radiation in two steps (by using a prism first and an echelle grating afterwards), and (3) a linear CCD array for detection. This configuration proved successful and the spectrometers currently commercialized by Analytik Jena are based on it, even though their characteristics are not totally identical. Fig. 2 shows the schematics of this instrument. In this regard, this review will focus on discussing the characteristics of the commercially available instrumentation.

This type of instrument provides significant advantages in comparison to traditional line source devices, such as improved signal stability, superior background correction potential, capabilities to monitor narrow molecular “lines” (the term transition is actually more correct when referring to molecular spectra, and will be used from now on), which permit the determination of non-metals, and improved linearity by making use of side pixels [16–19].

In addition, there is potential to simultaneously monitor several atomic lines, thus permitting multi-element determinations to be carried out. However, this potential is actually rather limited. The reason is that the instrumentation currently available only allows for the simultaneous monitoring of a small portion of the spectrum (0.2–0.3 nm in the UV region). This aspect does not seem to be always well understood by prospective new users of the technique that perhaps have other expectations (maybe also because some of the previous prototypes discussed before showed higher multi-element possibilities [7–11]). Thus, one of the main goals of this paper will be to further clarify this point and to discuss in detail under which circumstances it is possible to perform simultaneous multi-element

determinations with the current instrumentation commercially available.

However, there are other important advantages derived from the simultaneous monitoring of multiple atomic lines and/or molecular transitions. This review will examine them and highlight how different authors have made use of them to develop novel methodologies.

The review will be mostly focused on work carried out using a graphite furnace as atomizer, owing to its superior possibilities. Nevertheless, a specific section devoted to the peculiarities of flame (F) AAS in the context of multi-line monitoring will be presented as well.

2. Simultaneous multi-element determinations using high-resolution continuum source graphite furnace atomic absorption spectrometry

2.1. Requirements, limitations and strategies to develop multi-element methods

As discussed before, the potential of the current instrumentation to perform truly simultaneous multi-element determinations is limited. The main reason for this is that this instrumentation features a detector with 588 pixels of which only 200 are used for analytical purposes, while the remainder are used for internal corrections. This instrumentation provides an exceptional resolution, as every one of those pixels monitors a range of a few picometers only (approx. 1 pm at 200 nm), but that also means that such an instrument only allows for the simultaneous monitoring of a very small part of the spectrum: 0.2–0.3 nm in the UV region, where the most useful atomic lines are typically found, increasing in the visible region up to approx. 1.0 nm at 800 nm. Further improvements in this aspect would certainly be welcome, although they probably require the development of a suitable type of detector for this instrument (which represents a considerable investment) as the requirements of a GFAAS device (transient signals) are not the same as those for other types of optical instruments (e.g. emission devices), as discussed in various reviews [10,18,20].

Despite this limitation, there are still some possibilities to develop truly simultaneous methods. Table 1 shows a summary of the articles that have reported applications in this regard.

As discussed in these articles, there are a number of prerequisites to develop these multi-element methods. First of all, it is necessary to find atomic lines of the target elements that are sufficiently close (within 0.2–1.0 nm, depending on the wavelength). The difficulty in finding these lines depends on the target elements. There are some elements with hundreds of usable lines available in the UV–vis area, such as Co, Cr, Fe, Ni or Ti [18], and others that show at least tens of lines (e.g., Cu, Mn, Pb or Pd). Obviously, if your target analytes show many lines, chances to find some that are close enough increase.

However, not all the lines show the same sensitivity, and the lines finally selected must be suitable for the expected contents of all the target analytes. This is a severe requirement, because some solutions frequently used to adapt the sensitivity when performing mono-element determinations (e.g., diluting the sample or keeping the Ar flow during the atomization step) may not be appropriate for a simultaneous multi-element approach, as it is evident that they will have an effect on all the analytes.

Finally, the furnace conditions need to be considered. If the target elements show similar thermochemical behavior, it may be simple to develop a temperature program and use a chemical modifier that is adequate for all of them. However, if their behavior is very different (e.g., simultaneous determination of very volatile and refractory elements), compromise conditions need to be used. This last requirement does not typically represent an insurmountable problem to develop truly simultaneous methods. Such methods require the use of a chemical modifier in sufficient amount to properly stabilize the most volatile analytes (while preventing over-stabilization of the most refractories), a pyrolysis temperature low enough to avoid

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