



## Atomic absorption spectrometry – A multi element technique

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### ABSTRACT

In the past, many types of researches have been performed to allow multi-element determinations using atomic absorption spectrometry. The first spectrometers developed for this purpose were proposed in the 1970s, using flame and furnace atomizers.

In the early 2000s, a spectrometer equipped with software that allows a fast selection of the hollow cathode lamps was introduced commercially. This equipment operates in a sequential mode, and it enables the determination of up to 16 elements. Many publications have reported its performance.

Over the years, many spectrometers were built adding new technologies, but they were discontinued due to low sensitivity, difficulty for background corrections, etc. However, all the efforts of the past have contributed to the development and consolidation of the high-resolution continuum source atomic absorption spectrometry (HR-CS AAS) using flame and furnace atomizers. This technique allows the establishment of sequential and simultaneous methods for determinations of many elements with high sensitivity and efficient background correction.

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### 1. Introduction

Atomic absorption spectrometry (AAS) is one of the most commonly used techniques for analytical purposes [1]. It has been widely used in research laboratories and also in the food, environmental, pharmaceutical, petroleum and in other sectors [2–6]. It can be employed by three different atomization processes, which are: flame atomic absorption spectrometry (FAAS), electrothermal atomization atomic absorption spectrometry (ETAAS) [5], and chemical vapor generation atomic absorption spectrometry (CVG-AAS) [7,8]. However, the choice of the ideal technique is determined by the analyte's chemical nature and its content in the sample, as well as the sample's chemical composition and its physical state. Direct determination of analytes in solid samples using atomic absorption spectrometry requires specific

instrumentations, as opposed to liquid samples [1,4]. FAAS is the simplest and most widely used approach, although the sensitivity is the smallest among all others. ETAAS also has excellent applicability, as it allows the direct analysis of solid samples, and it has a higher sensitivity than FAAS. However, it requires an efficient optimization of the temperature program and choice of the chemical modifier to obtain valid results [1,9]. CVG-AAS has excellent sensitivity, and, in the past, it has been restricted to some elements (arsenic, antimony, selenium, bismuth, cadmium, lead, etc.). Currently, many researchers have developed works to extend the applicability of this technique for the determination of other chemical elements [10]. Cold vapor-AAS has a restricted use for the determination of mercury and cadmium [11,12]. It has an excellent sensitivity and it is one of the most recommended methodologies for the determination of mercury.

Despite all of these advantages and applications, the fact that AAS was not a multi-element technique was its main limitation in the past. Simultaneous or sequential methods could not be established. Also, the internal standardization technique, which is often recommended for corrections of matrix interferences, could not be used.

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The comparison between inductively coupled plasma optical emission spectrometry (ICP OES) and FAAS reveals that: (i) the two techniques have a similar sensitivity; (ii) the FAAS spectrometer is a cheaper equipment than the ICP OES spectrometer; (iii) the costs of gas consumption to employ FAAS are lower than using ICP OES, even when utilizing nitrous oxide, which is more expensive than acetylene; and (iv) both methodologies have allowed the coupling with flow injection analysis (FIA), sequential injection analysis (SIA) and others flow techniques [13,14]. Also, the comparison between ETAAS and inductively coupled plasma mass spectrometry (ICP-MS) shows compatible results. The sensitivities are similar, but the gases consumption using ETAAS is much lower than ICP-MS, and the ETAAS spectrometer and its maintenance are cheaper than the ICP-MS spectrometer. However, the optimization step of the experimental conditions for ETAAS, in some cases, can be more complicated when the element is volatile and the matrix is complex. The multi-element characteristic of the ICP OES and ICP-MS has always been the principal advantage in relation FAAS and ETAAS, respectively. Table 1 presents the limits of detection for several elements using FAAS, ETAAS, ICP OES and ICP-MS [15].

## 2. First studies – atomic absorption spectrometry as multi-element technique [16]

One of the first works established using AAS as a multi-element technique was proposed by Salin and Ingle [17]. The equipment was developed using four hollow cathode lamps, combined with beam splitters, a carbon rod atomizer, a special monochromator with separates slits for each element, and a single photomultiplier tube. Then, the lamps of each element are turned “on” and “off” in a sequential manner at the same frequency so that only one lamp is “on” at a time. The major limitation of this equipment was the impossibility of using the hydrogen lamp for background correction since its continuum radiation passes through all slits. The absolute light levels resultant of this approach was considerably lower than the systems developed using a single lamp. Spectral overlap problems were also observed. Despite this, these differences did not significantly affect the limits of detection, sensitivity, precision and calibration curves in most cases. The authors concluded that, after an instrumental improvement, this system could allow the determination of six to eight elements routinely [17]. Harnly et al. developed the first atomic absorption spectrometer using xenon arc lamp (300 W) as a continuum source. This instrument allowed

**Table 1**  
Limits of detection ( $\mu\text{g L}^{-1}$ ) from spectrometric techniques for some elements [15].

Element	FAAS	ETAAS*	ICP OES	ICP-MS
Ag	3	0.02	0.2	0.003
Al	30	0.2	0.2	0.06
Ba	20	0.5	0.01	0.002
Ca	1	0.5	0.0001	2
Cd	1	0.02	0.07	0.003
Cr	4	0.06	0.08	0.02
Cu	2	0.1	0.04	0.003
Fe	6	0.5	0.09	0.45
K	2	0.1	75	1
Mg	0.2	0.004	0.003	0.15
Mn	2	0.02	0.01	0.6
Mo	5	1	0.2	0.003
Na	0.2	0.04	0.1	0.05
Ni	3	1	0.2	0.005
Pb	5	0.2	1	0.007
Sn	15	10	1	0.02
V	25	2	8	0.005
Zn	1	0.01	0.1	0.008

Obs: \*Values based on a 10  $\mu\text{L}$  sample volume.

the simultaneous determination of up to 16 elements using flame and electrothermal atomizers [18]. Afterward, this equipment was employed in the simultaneous determination of nine metals in manganese nodules using air-acetylene flame. The results showed efficiency in accuracy and precision comparable to other conventional spectrometric methods [19]. Harnly and Kane also optimized the simultaneous determination of nine elements using this same spectrometer with electrothermal atomization. The parameters studied were: acid matrix (nitric or hydrochloric acid solution), measurement mode (peak height or area), atomizer surface (graphite type), atomization mode (tube wall and platform), and atomization temperature [20]. Many other works were also developed using this equipment; however, several limitations were observed, and the authors concluded that the use of high-quality gratings and two-dimensional detectors would be required [21].

In the late 1980s, an atomic absorption spectrometer capable of determining four elements simultaneously with furnace or flame as the atomizer using Zeeman for background correction [22] was developed. Farah and Sneddon optimized an automated flame/furnace spectrometer with the capacity of measuring four elements simultaneously. They observed that the sensitivity of the multi-element determination is lower than the single-element determination [23]. Sneddon et al. published a review article in 1993 presenting the several attempts that were developed aiming the use of AAS as a multi-element technique [21]. In 1996, Harnly, evaluated in detail the potential of an ETAAS spectrometer (prototype) using a continuous source, which was developed with the advances in the fields of the spectrometry and detector technology. The results obtained demonstrated that the instrument has the followings characteristics: (i) capacity for multi-element determinations from 30 to 40 elements; (ii) wavelength and time-integrated absorbance measurements which are independent of the source width; (iii) detection limits comparable to line source AAS; (iv) extended calibration ranges. Figures of merit obtained for some elements are comparable with those found using inductively coupled plasma mass spectrometer [24]. An electrothermal atomization atomic absorption spectrometer with longitudinal Zeeman-effect background correction using electrodeless discharge lamps (EDL) and transversely-heated graphite tube atomizer (THGA), allowed the development of multi-element determinations. This instrument was made commercially available but was later discontinued [25].

In the early 2000s, a company introduced a flame atomic absorption spectrometer, which works in fast sequential mode, fitted with a deuterium background corrector, automatic switching of hollow-cathode lamps, and programmable gas control that enables automatic set-up and change-over of gas flow. The use of 4 multi-element lamps allows the determination of up to 16 elements. However, during the analytical measurements, the burner height is fixed. Thus, the optimization of this parameter has been made considering either the analyte of lower sensitivity in AAS, or the one with the lowest concentration in the analyzed matrix. This equipment has had an excellent commercial acceptance, and several works have been developed using this instrument [26–33]. Projahn et al. proposed the use of the internal standardization technique in flame atomic absorption spectrometry using this spectrometer [26]. Cassella et al. determined sequentially calcium and magnesium in biodiesel using a single solution of the sample [27]. Pereira determined sequentially cadmium, copper, and lead in tea leaves employing thermospray-FAAS [28]. Another method was proposed for sequential determination of arsenic and antimony, bismuth, and lead by HG AAS [29]. Ferreira et al. developed an analytical strategy using slurry sampling for the determination of copper, manganese and iron in seafood [30]. Some methods have also been established using the internal standardization [27,31–33]. Table 2 reports some analytical methods developed using this spectrometer.

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