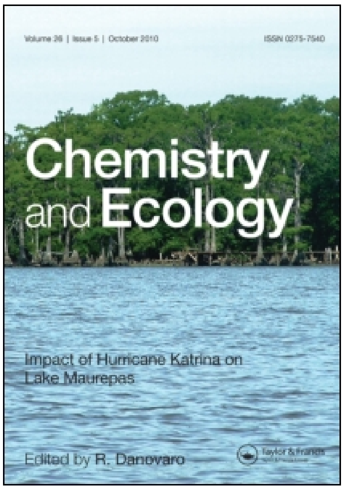

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Recent Instrumental Developments in the Background Correction Systems for Flame and Furnace Atomic Absorption†

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One of the problems that makes the use of the AA technique less frequent in some analytical fields is the interference of non-specific absorptions, particularly high in the furnace atomization. Traditional systems (used for several years for background correction with a deuterium arc lamp) are not applicable to some analyses, even if they meet most of the analytical needs. Some of the limits of this type of background correction are:

a) Incorrect results in the presence of "structured" background. In this case, since the instrument carries on background determination to be substracted from the analytical wavelength measurement taking into account the average of the values within the monochromator bandpass, it can sometimes happen that the "background" to be substracted is over- or under-estimated.

b) Difficulty in the compensation at wavelengths over 420 nm. For some instruments, the problem is overtaken by mounting a

† Presented at the Symposium on Analytical Problems in the Marine Environment, Genoa, 23–24 May, 1983).

tungsten lamp, that has the maximm emission energy in the visible region.

The possibility of using this lamp is anyway limited to few applications (Ba in seawater) as most of the non-specific absorbances are in the UV region.

c) Difficulty in the alignment of the two beams (specific and continuous) due to the different geometries of the two lamps.

d) Possibility of correcting no more than 1.5 abs.

e) Loss of energy due to the use of a "beam splitter."

f) Increase of background signals due to the electronics of the instrument that elaborates a number of signals twice the measure without the correction.

g) No possibility of compensating spectral interferences.

The late 1970s saw the introduction of a new background correction technique based on the Zeeman effect. A properly designed Zeeman system overcomes many of the above mentioned weaknesses but it is not free from problems of its own, particularly of a phenomenon known as "roll-over."

Calibration curves, in fact, tend to reverse their direction over a determined absorbance value (0.5 abs) so that to the same absorbance correspond very different concentration values. A new concept in the background correction was shown for the first time at the 1982 Pittsburgh Conference. Its basis is surprisingly simple. The method, called Smith–Hieftje, is based on the self-reversal effects of the emission lines of the hollow cathode source run at very high current.

It has been known for more than 20 years that, when an excessive current is passed through a hollow cathode lamp, neutral atoms are formed around the cathode; these atoms absorb the emitted energy changing the emission line profile (fig. 1). Excessively increasing the current, a complete self-reversal of the line can be obtained with a subsequent reduction of the atomic absorption to zero. To obtain the correction of non-specific absorption, it is enough to run the source (lamp) at a very low current, obtaining an absorbance value comprehensive of the background, then a brief pulse of a much higher current is passed through the lamp, obtaining only the absorbance of the background; therefore, background correction is obtained by taking the difference of the two signals.

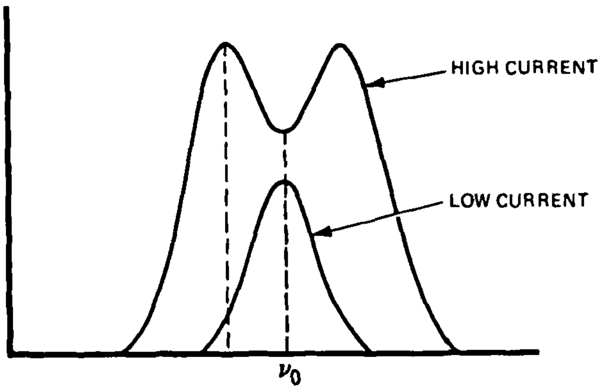

FIGURE 1

The realisation of the system has only required slight modification of the hollow cathode lamps to guarantee their lifetime, as per the traditional model ones.

In comparison to conventional deuterium background correction, the Smith–Hieftje system has all the advantages of the Zeeman:

a) Background correction can be effected in the visible as well as in the ultraviolet region of the spectrum.

b) Accurate correction for structured background.

c) Possibility of compensating even high background levels (abt 2–3 abs).

d) Correction of many spectral interferences.

Smith–Hieftje has also some advantages over Zeeman:

a) Possibility of being applied to both flame and flameless systems.

b) No loss of light due to polarizers.

c) In the absence of magnetic fields, their possible effects on structured background need not be considered.

Compared to the deuterium arc system, both the Smith–Hieftje and Zeeman systems share one disadvantage, i.e., reduced sensitivity

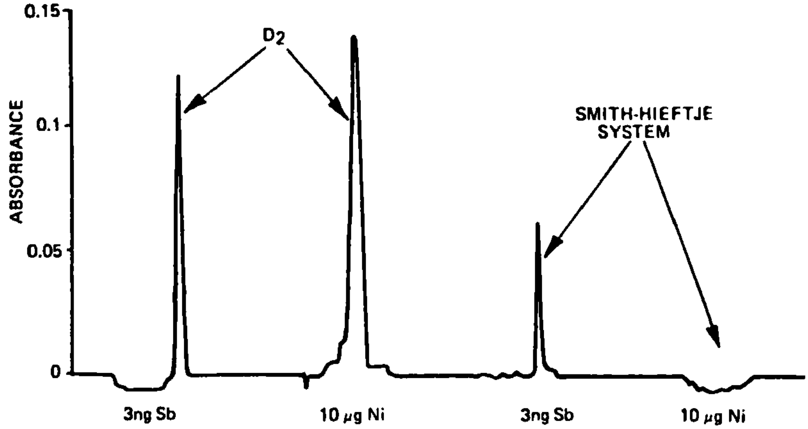

FIGURE 2

which can be compensated for in Smith–Hieftje, by a better signal/noise ratio, due to a better light efficiency of the system that allows a more stable base line.

In Figure 2, it is shown the determination of 10 μg of Ni at the 231.09 nm antimony line, illustrating that a nickel spectral interference exists with deuterium arc background correction while the next two peaks show that with Smith–Hieftje the spectral interference from nickel has all but been eliminated.

The new system for background correction has been incorporated into the last-born AA spectrophotometer of the Instrumentation Laboratory Inc. (IL Video 11, Video 12 and Video 22), complete with a video display as the preceding models (IL 451, 551 and 951).