

## **SPECTRAL INTERFERENCES IN ICP-OES**

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### **Introduction**

In atomic emission and atomic absorption spectrophotometry, the sample is heated to a high temperature and thereby decomposed into atoms and ions that absorb or emit visible or ultraviolet electromagnetic radiation at energies characteristic of the elements involved. The flame test is a very simple form of this technique. The yellowing of a flame by the addition of salt, for example, occurs because the sodium in salt emits strongly in the yellow portion of the visible spectrum. These methods are especially useful for low concentrations of metallic elements in both qualitative and quantitative analysis.

Interference on ICP-OES has not always been considered a serious problem, especially in the beginning, when compared with Atomic Absorption Spectroscopy. This was due to the euphoria created by the new technique. It meant that criticism was subdued.

The ICP was vastly superior to the Atomic Absorption technique, that the interferences suffered by the Atomic Absorption technique dwarfed the little interferences suffered by the ICP-OES technique. This is how ICP-OES has had a reputation as an interference free technique.

### **Discussion**

This work is concerned with spectral interference on the ICP-OES.

The ICP-OES technique is a widely used analytical tool in the analysis of a wide variety of samples, from exploration samples, to mining and metallurgical samples. The reasons why it is so popular are not hard to find. Among them are the following:

- Long dynamic range
- Few interferences
- User friendly nature of the instruments
- Simultaneous nature of the instrument (Ability to produce results for 60+ elements in the time it takes to produce one result). This reduces Turn around Times of the analyses.
- Reasonable stability of instruments
- Ability to tolerate high dissolved solids

While the ICP-OES was assumed by many to have been free of interference in the beginning, (this was in comparison to the Atomic Absorption technique) the reality is

that it suffers from interference like all other instrument techniques, although the actual effects on the results may differ from other techniques.

The interferences it suffers from are as follows:

- Spectral interference includes the following: stray radiation, overlapping of lines, poor resolution of lines and overlapping of a line by another. Stray light is light that does not originate from the analyte but is recorded in one of the analyte channels. It can originate from many sources, including reflection inside or outside the spectrometer. A strong line near the analyte line, gives rise to what is called near stray light, while far stray light arises from the grating. The calcium channel in a polychromator is a known source of stray light.
- Matrix effects include the following: atomization and volatilization interference, nebulisation interference, transfer and desolvation interferences, chemical interference and ionization interference.

By definition, an interferent enhances or depresses the intensity of the analyte, resulting in an incorrect result recorded.

There are a number of ways to deal with spectral interferences in ICP-OES:

1. Use the technique, but use standard addition to calibrate and analyze samples or
2. Use the calibration curve method with matrix matching for standards and samples or
3. Use the calibration curve method and do corrections for spectral interferences, using either the spectral corrections graph or the easier PC based approach..

### **The standard addition method**

Here a known concentration of the analyte is spiked into both calibration standards and samples alike. The samples are sprayed on ICP-OES instrument, and the results are obtained. The procedure gives reliable results. It is however time consuming and tedious. It increases Turn Around Times unreasonably.

It is thus not a recommended approach to doing analyses by ICP-OES where time is of the essence.

### **Matrix matching**

Matrix matching is a difficult way to do analysis. This is due to the fact that one can never matrix match standards to samples exactly all the time. Many resources are needed to do multi-element analysis using this approach. It is also time consuming, and thus increases turn around times like the standard addition approach.

### **Corrections for spectral interferences using an interference correction graph**

Here, a number of known concentration standards of the interferent are prepared and sprayed into the ICP as normal samples. The instrument response is measured on the wavelength of the element against which the interferent interferes. The results are plotted on a graph, with the known concentration of the interferent on the Y-axis and the apparent analyte concentrations plotted on the X-axis. When samples are analyzed for the element that is interfered with, the results will be more than the actual analyte concentration. The correction is effected by determining the actual concentration of

the interferent. The correction is then effected by reading off the apparent concentration of the analyte from the graph and then subtracting this value from the analyte concentration. Figure 1 is an example of a spectral interference correction graph.

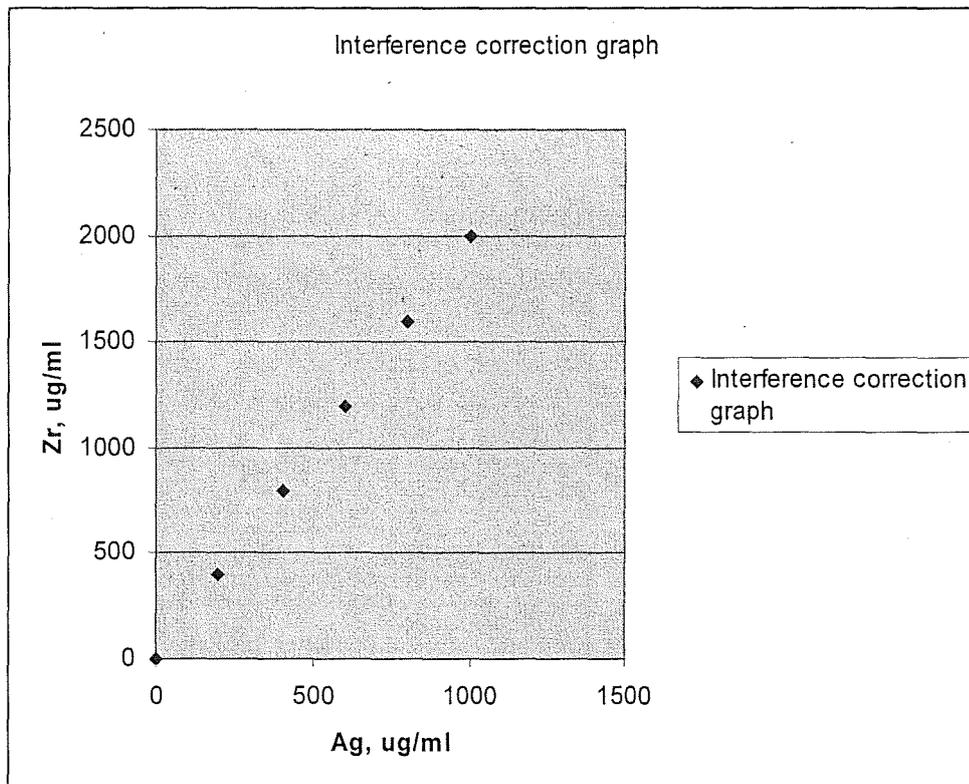


Figure 1. Interference correction graph

### Corrections for Spectral interferences using a computer

Modern spectrometers are equipped with sophisticated software, which comes handy when doing spectral corrections. There is a need to be aware that the computer as is currently used to do corrections, assumes that the relationship between intereferent and analyte is proportional.

This is the same principle that the correction graph approach takes.

It is very difficult to do matrix matching with all samples that one analyses by ICP-OES.

- Select all available lines for each element in the suit of elements that will become the method. The only criterion used to select the lines should be that they are sufficiently sensitive; otherwise they are of no use as they will have high detection limits on the method.
- Prepare typical samples, which will routinely be analyzed on the method being set up. This is to ensure that the method is set up using typical samples, which will be practical as they will have all the typical interferences experienced under normal operations.
- Spray the samples on the instrument and store the spectra from the excercise. The stored data is recalled and the spectra are then examined using the computer. The required elements whose spectra consist of more than one emission lines are

then scrutinized and the lines with interferences are rejected and only those without are selected for use.

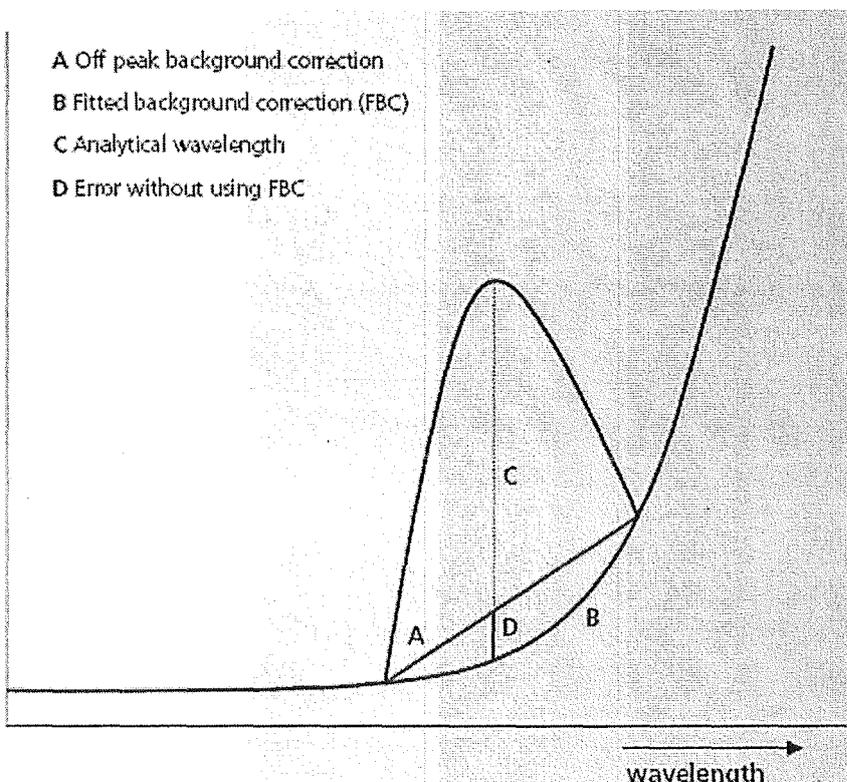
- Where there are no interference free lines, the lines with interference is used, depending on the type of interference. Continuum overlap is extremely difficult to correct for. AI tends to give continuum overlap, which affects lines between 190nm and 220nm.

Direct spectral overlap can be corrected where greater resolution to the peaks may reduce interference.

These days it is a little different, when one considers the extent of technological development.

These days, spectral corrections can be achieved for the most part, by spectral manipulations on a PC. This means that while it was necessary to draw up a spectral correction curve in the past, it is no longer necessary, as long one is using the latest instruments.

Figure1. below is an example of how the new instruments do spectral corrections and background correction in one step. The figure shows how the new Varian ICP Expert II software allows the user to do both actions together without problems. It also shows in D the effect of the interference, if no corrections were done.



The software contains wizards to help the user to master the ins and outs of the software with ease. This allows for fast method development and thus fast analysis after one starts to use the software.

The software, which is called Fast Automated Curve-fitting technique, solves interference related problems easily and fast, without the use of the interference correction graph.

### Conclusion

Spectral interferences are the most serious shortcoming of ICP-OES. Spectral interferences are difficult to correct for. This is due to the fact that a number of approaches exist to do corrections. Where old instruments are used, the spectral correction graph is useful. Where a new instrument with new software is used, the computer can be used to do corrections effectively. Effective mastery of spectral corrections can lead to full mastery of the ICP-OES instrument and its diverse applications.

### References:

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