

Benefits of transitioning from Flame AAS to the 4210 MP-AES

Technical Overview



Introduction

Reducing ongoing operating costs, increasing safety, improving analytical performance and ease of use are some of the key challenges facing current users of Flame Atomic Absorption Spectroscopy (FAAS). With the introduction of the Agilent Microwave Plasma-Atomic Emission Spectrometer (MP-AES), these challenges have been overcome, making it the ideal instrument for laboratories looking to transition away from FAAS to a more powerful, less expensive and safer technique. Additionally, with the extra performance of the MP-AES, the sample preparation process can also be significantly simplified, saving time and money.

The Agilent 4210 MP-AES features a waveguide design and torch that is capable of running samples with high total dissolved solids without compromising detection limits.



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Reduced running costs

The largest contributor to ongoing running costs for entry level spectroscopy is gases. FAAS uses a combination of air and acetylene, or nitrous oxide and acetylene for the flame. While air can be provided by an air compressor, the acetylene and nitrous oxide is supplied in cylinders and regularly needs to be replenished as it is consumed.

The 4210 MP-AES uses nitrogen extracted straight from air to sustain the plasma. The Agilent 4107 Nitrogen Generator coupled to an air compressor supplies all the free nitrogen required at greater than 99.5% purity. This leads to dramatic reductions in operating costs over the life of the instrument.

The potential cost saving of using the 4210 MP-AES for the determination of Ca, Mg, Na and K in fruit juice is illustrated by comparing a FAAS purchased with an air compressor and 1 year of consumables to an MP-AES purchased with air compressor, nitrogen generator, SPS 4 autosampler, and 1 year of consumables (Figure 1).

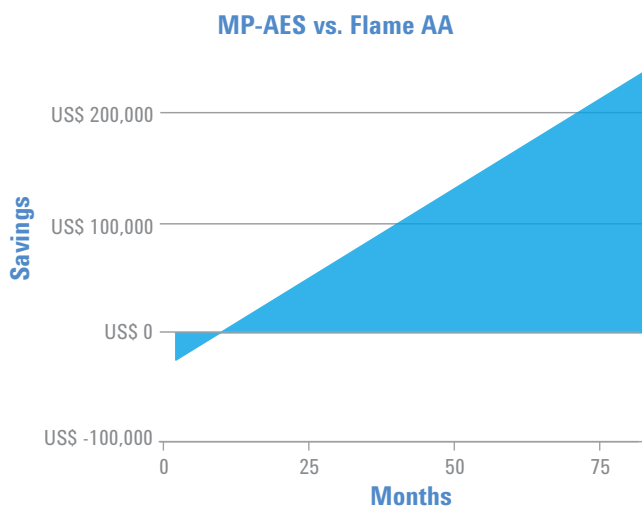


Figure 1. Online cost estimator showing the cost savings of using the MP-AES for the determination of Ca, Mg, Na and K in fruit juice.

¹ This example is intended to help you compare the running costs and savings of the MP-AES vs. flame AA. The applied formulas and parameters are correct to the best of our knowledge, but we cannot guarantee the results. Savings may vary depending on factors such as local gas and electricity costs, operator costs, number and types of elements. For this calculation operator labor costs were set to US\$25/hour and electricity costs were set to US\$0.18 per kW.

The analysis requirements were assumed to be 500 samples per week and 4 elements per sample.

The calculation assumes that the FAAS is run without an autosampler and that 3 elements are determined with air/acetylene and 1 element with nitrous oxide/acetylene. In this example the results show an estimated cost saving of greater than US \$220,000 over a 7 year evaluation period¹. A global average gas cost was used in this calculation and results will vary from country to country.

Improved safety

Another major concern for FAAS users is the safety aspects related to the use of acetylene and nitrous oxide, from the storage and handling of cylinders, to the use of a flame in the instrument. Presence of a naked flame is of concern in all labs, particularly those that handle organic solvents, and for this reason it is not possible to run FAAS unattended.

It is also common to have to change burners to determine the full range of elements when running FAAS. While Agilent's FAAS instruments are fully interlocked to ensure the correct burners are used with the correct method, care must be taken when handling burners which can remain hot after use.

These issues are eliminated with the 4210 MP-AES. The requirements for acetylene and nitrous oxide can be avoided, along with the storage and handling concerns, and there is no need for burner change-over because of the increased performance of the higher temperature nitrogen plasma.

Improved analytical performance

The plasma of the 4210 MP-AES operates at around 5000 K which results in improved detection limits when compared to FAAS. The improvements in detection limit means that it's possible to analyse elements like phosphorus, which have very high detection limits on FAAS.

Table 1 shows the instrument detection limits (IDL) on MP-AES and FAAS for elements determined in a rice flour sample. The lower detection limits for phosphorus, copper and iron allow the major, minor and trace elements to be determined in one sample measurement.

Table 1. Comparison of typical instrument detection limits for the 4210 MP-AES and FAAS

Element	4210 Typical IDL 10 sec read $\mu\text{g/L}$	FAAS Typical IDL $\mu\text{g/L}$
Ca	0.04	0.4
Mg	0.1	0.27
Na	0.1	0.26
K	0.6	0.76
P	66	26000
Fe	1.7	7.3
Pb	2.5	14
Cu	0.5	1.2
Mn	0.2	1.0

The design of the waveguide and torch in the 4210 MP-AES, combined with the mass flow control and humidification of the nebulizer gas line, gives excellent long term stability in samples with a complex matrix common in mining and environmental samples. The introduction of elevated salt solutions into an air acetylene FAAS burner over an extended period, such as an 8 hr work day, will require maintenance to avoid blockage. If this routine maintenance is not performed, it can lead to signal drift.

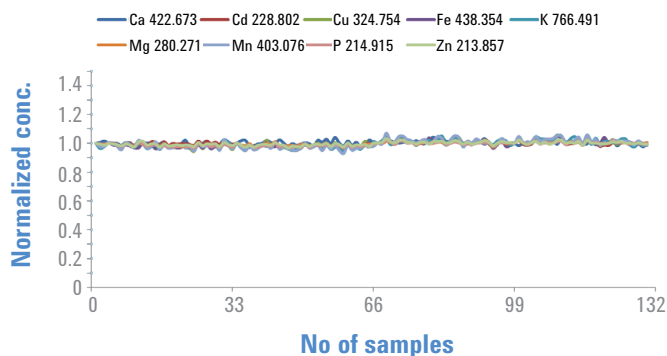


Figure 2. Digested rice flour with 2% TDS analyzed over 8 hrs. Recalibration was performed each 2 hrs and the resulting stability was <3% RSD for all elements. Default multi purpose sample introduction was used.

Long term stability of the 4210 MP-AES was tested with a 2% TDS solution of digested rice flour. The results are shown in Figure 2.

The 4210 MP-AES also features a greater linear dynamic range than FAAS. Table 2 gives the linear calibration range and correlation coefficient for major elements in a fruit juice sample for the 4210 MP-AES. Also shown is the optimum working range on FAAS for the same elements. The calibrations for FAAS used the default New Rational model. The greater linear range of the MP-AES compared to FAAS reduces the need to perform dilutions on over range samples which simplifies the analysis. Reducing the dilutions also means that if there are trace contaminants that need to be determined, it may still be possible to detect them. Furthermore, the improved linearity means that fewer calibration standards are required for an accurate calibration curve.

Table 2. Linear concentration range of the 4210 MP-AES and optimum concentration range of FAAS

Element	4210 MP-AES linear concentration range (mg/L)	Linear correlation coefficient on MP-AES calibration	FAAS optimum working range (mg/L)
Ca 422.673	0 – 20	0.9999	0.01 - 10
Mg 518.360	0 – 100	0.99988	0.15 - 20 (for Mg 202.6)
Na 589.592	0 – 20	0.99996	0.01 – 2.0
K 769.897	0 – 100	0.99968	1 – 6.0

Simplifying sample preparation

A factor which greatly influences sample preparation procedures on FAAS is the presence of interferences. Presence of compounds that cannot be broken down in the low temperature flame lead to chemical interference and elements like Na and K can suffer from ionization interference.

Various strategies for dealing with these interferences are well established. It is common to add releasing agents such as strontium or lanthanum to overcome chemical interferences, or alternatively the hotter nitrous oxide flame can be used. Ionization effects

are usually overcome by adding an ionization buffer to the solution, such as sodium, potassium or cesium. Another strategy is to extract the elements of interest into an organic phase in order to remove the interfering elements. As a result the sample must be individually prepared for each element in the sample.

With the hotter plasma source of the 4210 MP-AES these chemical interferences are eliminated. This means that the element specific sample preparation required on FAAS is not needed which greatly simplifies the sample preparation process. As an example the elements covered in a fruit juice analysis are shown below with a comparison of the sample preparation required for each element (Tables 3 and 4).

Table 3. Typical sample preparation requirements for FAAS and MP-AES

Element	Possible chemical interferences	FAAS specific sample preparation	MP-AES specific sample preparation
Ca	Refractory compounds Ionization effects	Lanthanum releasing agent Cesium ionization buffer	None
Mg	Ionization effects	Cesium ionization buffer	None
Na	Refractory compounds Ionization effects	Lanthanum releasing agent Cesium ionization buffer	None
K	Ionization effects	Cesium ionization buffer	None

Table 4. Certified Reference Material (CRM) recoveries of major elements in grapefruit juice, analyzed by MP-AES. No ionization suppressant was required and excellent accuracy for K was achieved. Additionally, no lanthanum nitrate was added and excellent recoveries for Ca was achieved

Grapefruit Juice T0842QC	Certified Value (mg/L)		Found (mg/L)	%Recovery
	Assigned Value	Range		
Calcium	145.6	123.6 – 167.6	158.3 ± 3.2	108.7
Magnesium	92.5	77.5 – 107.4	91.1 ± 0.6	98.5
Potassium	1102	979 – 1225	1100 ± 14.7	99.8

Conclusion

Agilent's 4210 MP-AES is the ideal instrument for customers looking to transition from Flame Atomic Absorption Spectroscopy (FAAS) to another technique. By using nitrogen as the source gas for the plasma, running costs are greatly reduced, and by removing the requirement for hazardous nitrous oxide and acetylene safety is greatly increased. Additionally the higher temperature nitrogen plasma atomization/ionization source improves detection limits, linear range, and long term stability, and allows the sample preparation process to be greatly simplified.

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