Atomic Absorption Standards, Context and Application

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1.0 Abstract

There are two main types of *Atomic Absorption Spectrometry (AAS)*, Flame Atomic Absorption Spectrometry (FAAS) and Graphite Furnace Atomic Absorption Spectrometry (GFAAS). There are advantages both technical and commercial, attributable to each one. In this paper, we will look briefly at the underlying technology in each, how they operate and what those advantages and disadvantages are.

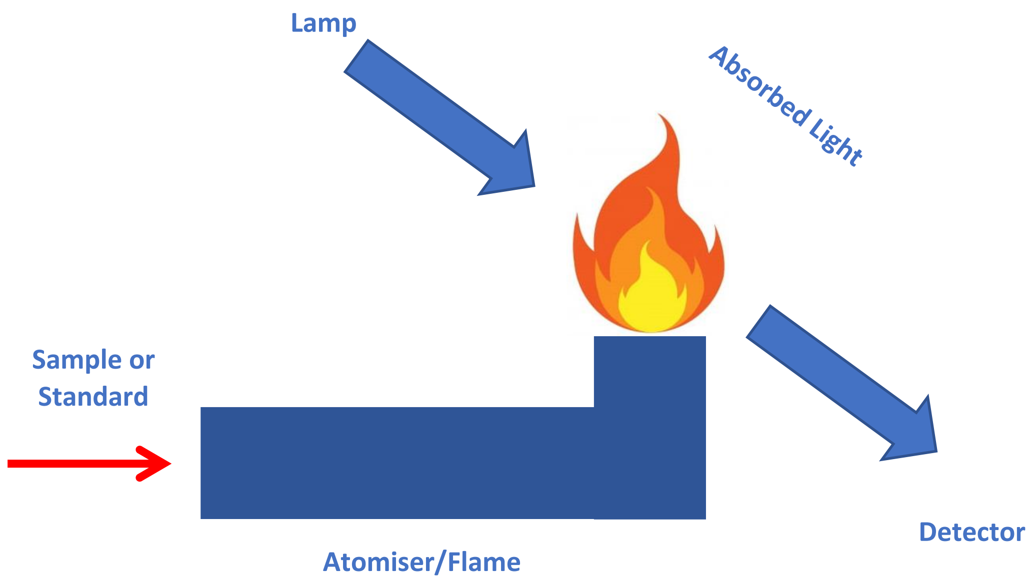
Irrespective of choice, in terms of selection of an AAS instrument the analyst will require high quality standards. AAS is a comparative analytical method, the instrument needs calibration, the whole measurement process needs controlling, methods need validating and in a regulated industry, the system needs qualification. Although, there are many good producers of AA standards, such standards have been produced commercially in this authors laboratory for over 30 years. Therefore, because of familiarity with these products and the very considerable research and development resources, expended on their development, the features and benefits of the Reagecon standards, will be described in detail. It is our belief that the features and benefits described form a template of what to look for in an ideal AAS Standard, irrespective of source. This is the primary objective of this publication. The measurement of AAS, in particular Graphite Furnace Atomic Absorption, requires some additional solutions, in the form of Releasing Reagents and Matrix Modifiers. Details of these ancillary products are also presented.

2.0 Flame Atomic Absorption Spectrometry (FAAS) – The Technology

In Flame Atomic Absorption Spectrometry (FAAS), either an air/acetylene or a nitrous oxide/acetylene flame is used to evaporate the solvent and dissociate the sample into its component atoms. When light from a hollow cathode lamp (selected based on the element to be determined) passes through the cloud of atoms, the atoms of interest absorb the light from the lamp. This is measured by a detector, and used as the basis to calculate the concentration of that element in the original sample. The use of a flame limits the excitation temperature reached by a sample to a maximum of approximately 2600°C (with the nitrous oxide/acetylene flame). For many elements this is not a problem. However, there are a number of refractory elements like Vanadium, Zirconium, Molybdenum and Boron which do not perform well with a flame source. This is because the maximum temperature reached, even with the nitrous oxide/acetylene flame, is insufficient to break down compounds of these elements. As a result, Fame AAS sensitivity for these elements is not as good as other elemental analysis techniques.

The functionality of the technique can be illustrated in a very simple graphic (Graphic 1). In its simplest form, the sample or standard is introduced into a flame, the sample is dissociated into its component atoms by the flame, light of an appropriate wavelength is passed through it, the element of interest absorbs the light and the concentration of the element is measured based on the absorption[1].

Simple Graphic to Illustrate Functionality of FAAS



Graphic 1

2.1 Advantages and Disadvantages of FAAS[2]

These can be summarised as follows:

2.1.1 Advantages

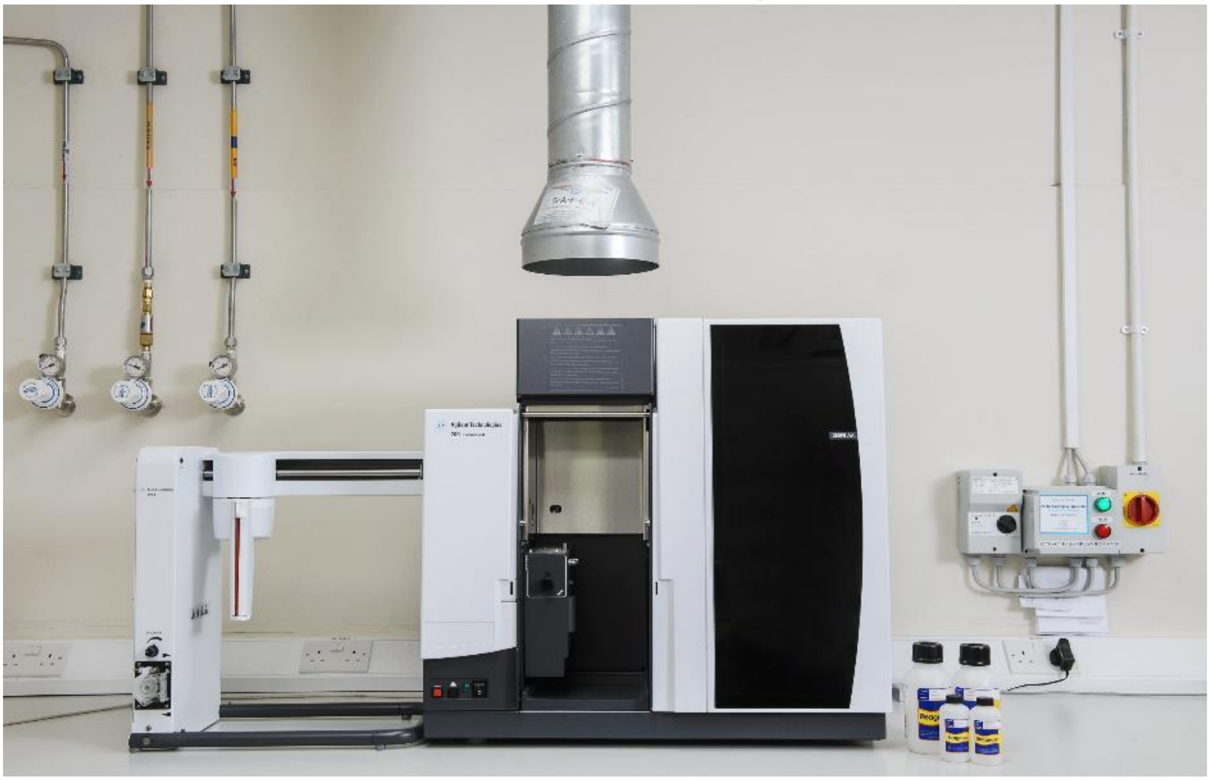
FAAS is inexpensive to run and maintain and carries a fairly low capital cost. It is rapid for selected elements. It is exceptionally accurate for alkali metals and can be used for some organic solvents. In our standards laboratory, we have been able to test and certify alkali metal standards to a specification of ± 0.2% using modified FAAS techniques.

2.1.2 Disadvantages

It has poor sensitivity (high limits of detection). Only single element determination at a time is facilitated and it requires a large amount of sample. It has a narrow linear range, lower sensitivity than GFAAS and is not as efficient in terms of sample throughput. The excitation temperature of 2600°C renders it unsuitable for elements that require a higher excitation temperature or it requires a higher temperature to breakdown the compounds containing the relevant element.

The modified FAAS instrument used in the authors cation and anion laboratory is presented in Photograph 1.

FAAS Instrument Used in Reagecon



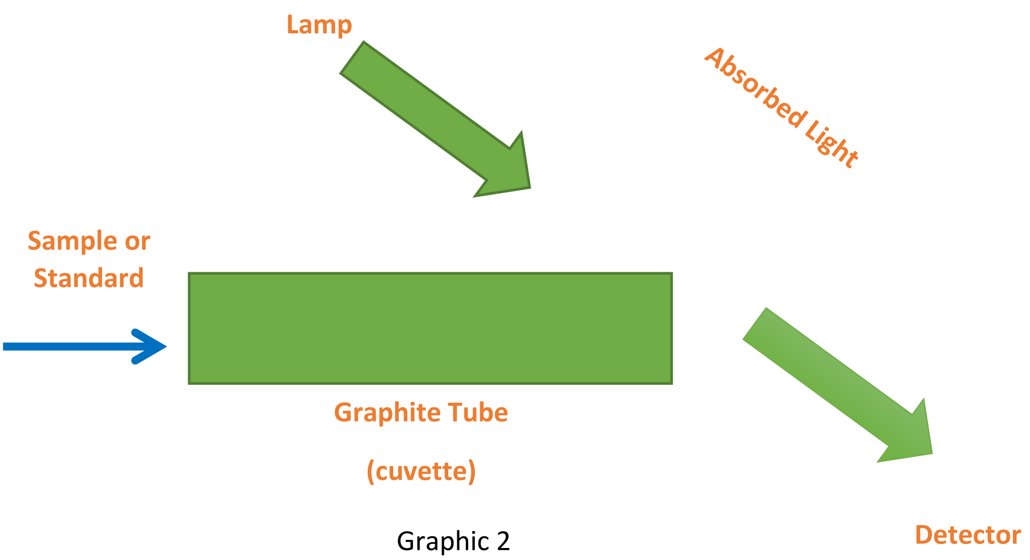
Photograph 1

3.0 Graphite Furnace Atomic Absorption Spectrometry (GFAAS)

In the case of Graphite Furnace the technique is essentially the same as flame AA, except the flame is replaced by a small, electrically heated graphite tube, or cuvette, which is heated to a temperature of up to 3000°C to generate the cloud of atoms. The higher atom density and longer retention time in the tube improve GFAAS detection limits by a factor of up to 1000x compared to FAAS, down to the sub-ppb range. However, because of the temperature limitation and the use of graphite cuvettes, refractory element performance is still somewhat limited.

Again, the technique can be illustrated by a simple graphic (see graphic 2).

Simple Graphic to Illustrate Functionality of GFAAS



Graphic 2

3.1 Advantages and Disadvantages of GFAAS

These can be summarised as follows:

3.1.1 Advantages

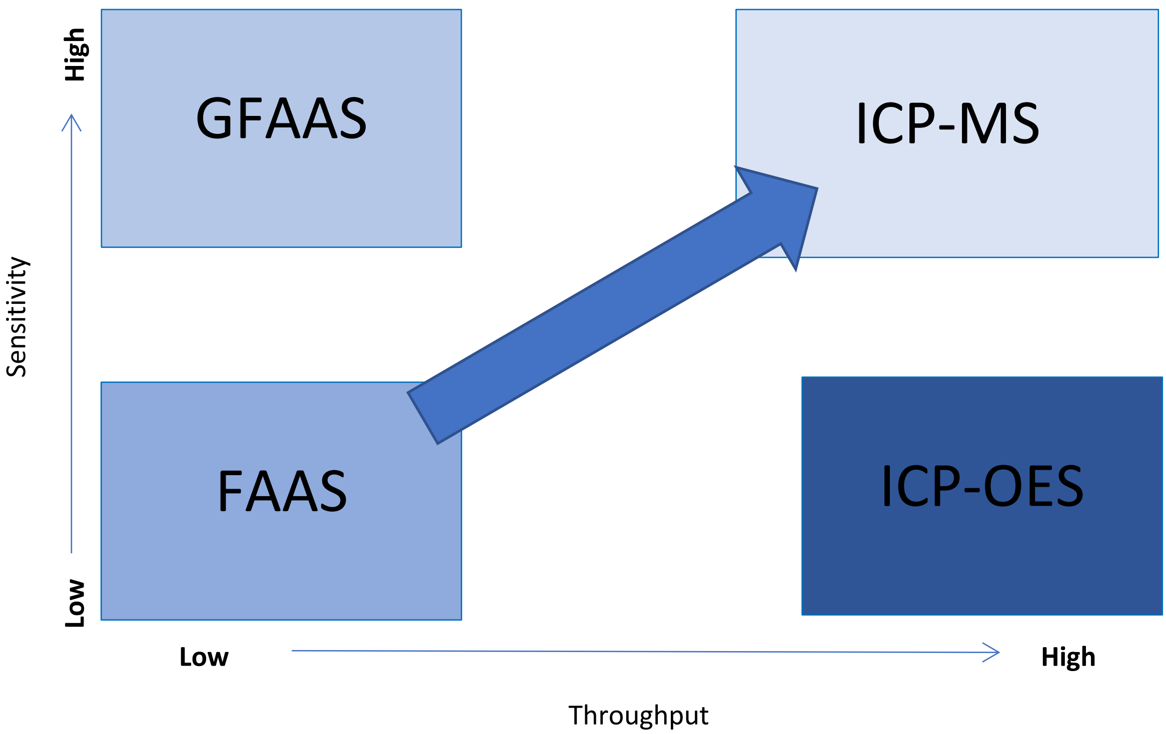
GFAAS is a relatively inexpensive technique which requires small sample volume and has excellent sensitivity. It facilitates a higher throughput of samples than FAAS.

3.1.2 Disadvantages

It only does a single element determination at a time and has high operating costs. It has very narrow linear range, it is cumbersome and time consuming, and is not suitable for organic solvents. It also requires Matrix Modifiers.

4.0 Comparison of Elemental Techniques

If we compare FAAS and GFAAS with Induced Coupling Plasma (ICP), we see in graphic 3 that GFAAS is a significantly more sensitive technique. ICP-OES on the other hand facilitates a significantly greater throughput than AA, but to combine throughput and sensitivity, ICP-MS is the ultimate method of choice.



Graphic 3

However, apart from throughput and sensitivity, other parameters also have to be taken into account when determining overall fitness for purpose. Table 1 presents a number of additional parameters that must be taken into account when comparing GFAAS, which is the most popular AA technique, when compared to various ICP techniques.

Fitness for Purpose, Comparison Between GFAAS and ICP Iterations[3]

		Sequential	Simultaneous	
Criteria	**GFAAS**	**ICP-OES**	**ICP-OES**	**ICP-MS**
Detection Limits	ppt	ppb	ppb	ppb-ppt
Linear Range	2-3	4-6	4-6	9*
Interferences	Moderate	Many	Many	Few
Speed	Slow	Slow	Fast	Fast
Elemental coverage	Poor	Good	Good	Excellent
Multi-element	No	Yes	Yes	Yes
Simultaneous	No	No	Yes	Yes
Sample size	µL	mL	mL	µL or mL
Capital cost	Low	Low	Moderate	High
Operating cost	High	Moderate	Moderate	Low

Table 1

5.0 Atomic Absorption Standards, Matrix Modifiers and Releasing Agents

Reagecon manufactures an extensive range of AA Standards, all of which are applicable to both FAAS and GFAAS. Table 2 provides a detailed summary of the features and benefits of the Reagecon range of standards[1], which is the widest range available on the market.

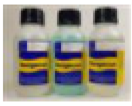
Features and Benefits of the Reagecon Range	
1	Use similar raw materials to those used in the manufacture of Reagecon's ICP-MS/ICP-OES Standards. This means using pure metals where possible of greater than 99.999% purity, or where not possible using metals of 99.995% purity or where not available, using metals or salts of 99.9% purity.
2	All products for Atomic Absorption are produced in class ISO7 (10,000) clean room conditions.
3	All Raw materials are assayed for purity by ICP-MS and titration prior to manufacture. This provides two layers of traceability.
4	Products are certified gravimetrically, Reagecon holds ISO 17025 accreditation for the calibration of laboratory balances.
5	All final products are assayed by titimetry and verified using an ICP-MS instrument or in the case of alkali metals by an AAS instrument.
6	All products traceable to a primary standard or certified reference material or in a lot of instances both of the above.
7	A wide range of customised options are available.
8	More element mixtures can be formulated and certified upon request.
9	Several matrix modifiers (10) and releasing agents (4) formulations available.
10	All standards are available at 1000 ppm and 10,000 ppm plus several additional concentrations.
11	Widest range available from any supplier.

Table 2

The quality and purity of all of these solutions, have a very significant impact on the quality of the analytical result. Reagecon's Releasing Agents and Matrix Modifiers are made from the same high purity raw materials that are used for standards manufacture, the range is substantial and the company manufacture customised mixes upon request.

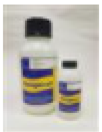
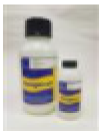
Detailed technical specifications on all Reagecon Standards, Matrix Modifiers and Releasing Agents, shipping details, availability, pricing and a whole host of other information is available at www.reagecon.com. Examples of the Reagecon AAS Standards, Matrix Modifiers and Releasing Agents, as presented on the Reagecon ecommerce facility can be seen in graphics 4, 5 and 6.

Atomic Absorption Standards[4]

	AA-GLO-B-500	**Reagecon Boron Standard for Atomic Absorption (AAS) 1000 µg/mL (1000 ppm) in Water (H_2O)**	Contact us
	AA-GLO-BA-500	**Reagecon Barium Standard for Atomic Absorption (AAS) 1000 µg/mL (1000 ppm) in 0.5M Nitric Acid (HNO_3)**	Contact us
	AA-GLO-SIL-100	**Reagecon Silicon Standard for Atomic Absorption (AAS) 1000 µg/mL (1000 ppm) in Water (H_2O)**	Contact us

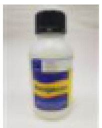
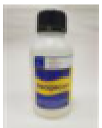
Graphic 4

Atomic Absorption Standards – Matrix Modifiers[4]

	MMS1001	**Reagecon Palladium Nitrate 2% Matrix Modifier for Graphite Furnace Atomic Absorption Spectroscopy (GFAAS) in 10% Nitric Acid (HNO_3)**	Contact us
	MMS1005	**Reagecon Palladium Nitrate 2% Matrix Modifier for Graphite Furnace Atomic Absorption Spectroscopy (GFAAS) in 10% Nitric Acid (HNO_3)**	Contact us
	MMS101	**Reagecon Ammonium Dihydrogen Phosphate 40% Matrix Modifier for Graphite Furnace Atomic Absorption Spectroscopy (GFAAS) in Water (H_2O)**	Contact us

Graphic 5

Atomic Absorption Standards – Releasing Agents[4]

	RA1CO5	**Reagecon Release Agent 1.0% Lanthanum for Atomic Absorption (AAS) in Hydrochloric Acid (HCl)**	Contact us
	RA1N05	**Reagecon Release Agent 1.0% Lanthanum for Atomic Absorption (AAS) in Nitric Acid (HNO_3)**	Contact us
	RA5C05	**Reagecon Release Agent 5.0% Lanthanum for Atomic Absorption (AAS) in Hydrochloric Acid (HCl)**	Contact us

Graphic 6

6.0 Summary

The quality of any analytical result, is entirely dependent on the standards used. The AAS technique is no different in this respect, and in addition to high quality standards, the quality of the other reagents as outlined above, are also very important. There are many good producers of these materials and the information provided in this publication, provides the analyst, with a template upon which to base their decision on where to obtain these materials. AAS is still a widely used and universal technique, although to some extent, now superceded by various ICP technologies. Ultimately the decision of which technique to use, is governed by the basic principle of fitness for purpose. It is hoped that if AAS, happens to be the chosen technique, then the information provided here, will bring value, efficiency, enable the analyst to obtain the correct result, and prove the correctness of that result.

7.0 Bibliography

1. Barron, John. (2016)., Physical and Chemical Standards Compendium, p 275. www.reagecon.com

2. Barron, John., (2017) Atomic Absorption Standards, Sales Presenter No. 2, Internal Reagecon Communication.

3. Barron, John., (May 2019) Cation and Anion Webinar, presented at the Reagecon Sales Convention, Shannon, County Clare.

4. Reagecon (2020), Ecommerce Facility