Agilent

Trusted Answers

Analysis of Foods and Beverages Using Atomic Absorption Spectrometry

A Q&A guide to novel techniques

Contents

Introduction

Atomic absorption (AA) spectroscopy may not be a new analytical technique, but that doesn't mean it is any less important than other newer tools. Many analytical methods and regulations have "grown up" with AA spectrometry, and it remains a center point of sample analysis at many labs to this day.

AA instruments continue to be appealing for several reasons such as its lower purchase price and operational costs. AA technology is also developing in ways that both keep these tools relevant and may ease the transition to other options when labs are ready to invest in other techniques.

In this e-book, we'll explain some important ways that the AA technique meets the increasingly stringent demands of modern food analysis including:

- Historical and modern uses of AA spectrometry,
- Regulatory issues,
- The use of flame versus graphite AA spectrometry,
- Quality control for milk products,
- Automation,
- Heavy metals analyses, and
- Challenging matrices such as wine.

Flame AA Method Development Primer

This easy-reference e-Primer helps you set up instrumentation and operating conditions for more than 60 elements, with tips for optimal performance. It also includes application guidance for sample types in areas such as food and agriculture, environmental, chemicals, minerals, and more.

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Agilent 200 AAS Instruments Video

The Agilent AAS instruments are productive, user-friendly, and reliable. Watch this video and discover how an Agilent AA instrument can help you handle your analytical challenges.

Watch video

Novel Atomic Absorption Spectrometry Techniques for Analysis of Foods and Beverages

Though atomic absorption (AA) spectrometry has been routinely used by analytical laboratories for decades, it is certainly no less important and relevant for modern analyses. As regulations become more stringent (making analytical work more complex), AA instruments have kept pace and remain a go-to analytical technique for food analysis. Innovations in flame and graphite furnace AA spectrometry for analyzing complex matrices like wine and milk are just part of the ongoing development of the technique.

Q. How widely used is AA spectrometry in food testing labs?

A. For decades, AA spectrometry has been a well-established technique for food analysis and for many other applications. With this technique, samples are atomized in a flame or a graphite furnace. AA spectrometry techniques can analyze more than 60 elements and cover a wide analytical range, from % levels down to subparts per billion.

The technique is best suited to applications with up to eight elements, and sample quantities ranging from a handful to 100 per day.

Flame AA spectrometry can quantify several nutritional elements such as sodium, potassium, calcium, magnesium, and iron in food samples. Elemental analysis is important for product labeling and quality control.

Quantification of heavy metals like lead, cadmium, and arsenic is mainly done by graphite furnace AA spectrometry.

The past decade has seen a significant increase in the use of AA spectrometry in the food and beverage industry for many good reasons. This mature technique is robust yet simple to use. The sensitivity and accuracy of an AA method is sufficient for most food products. Importantly, AA spectrometry is relatively low cost to run. In fact, the price of an AA instrument is the lowest of all comparable technologies suitable for food analysis such as inductively coupled plasma–mass spectrometry (ICP-MS), or inductively coupled plasma–optical emission spectrometry (ICP-OES) instruments. There are also few interferences in AA spectrometry compared with ICP-OES or ICP-MS techniques, which makes analysis much simpler.

There's also the convenience factor. If you purchase an AA instrument, you can access well-established methods to run food samples. Instead of spending time on method development and optimization, you can install an AA spectrometer, use the template methods available, verify the method, and start running samples for routine analysis soon after.

Q. AA spectrometry is a mature technique. How has the technique kept up with the increasingly stringent regulations?

A. Flame and graphite furnace AA have been well established since the 1980s. Comparatively, ICP-OES and ICP-MS techniques have been in routine use for around 20 years. So, the regulations were developed with flame AA and graphite furnace AA spectrometry in mind. Over the years, food safety has become an increasingly important topic in the world of consumer safety. Foodstuff legislation has increased, particularly relating to trace elemental analysis of toxic metals. If new regulations and standards are based on ICP-OES and ICP-MS techniques, older AA regulations continue to be revised and used.

It's important to note that international or national standards involving flame AA mainly cover one or two elements at a time. For example, there's one standard for the analysis of zinc in milk. You'll find other standards for iron and copper in wine for example. A separate one covers calcium, magnesium, and potassium in wine, and so forth.

So, to analyze the full range of sodium, potassium, calcium, magnesium, iron, copper, and zinc, you will find different methods available for flame AA. For graphite furnace AA, there are separate standards for cadmium, for lead, and for arsenic quantification.

In contrast, just one standard is used to analyze all elements in wine with ICP-OES and one for ICP-MS. We have approximately 15 standards for milk and wine analysis for AA, but only two for ICP-ES and two for ICP-MS.

Q. How can AA be used for the quality control of milk products?

A. The elemental analysis of milk is important for both quality control and product labeling requirements. Many such elements can be analyzed in food products by the simple flame AA spectrometry technique.

As more food products are imported and exported around the globe, greater quality control methods are needed. Some laboratories are looking for optimized AA systems that can efficiently and accurately analyze as many as 100 milk samples per day. Such optimization is essential because it is inefficient to dedicate one analyst to manually running large numbers of samples every day.

Remeasurement often occurs in food labs when results show errors. Errors caused by incorrectly prepared standards, sample preparation errors including dilution, and incorrect addition of ionization buffer can all force sample remeasurement.

If your lab analyzes 100 samples per day, you would need to prepare up to 10 calibration standards, dilute samples and/or add ionization buffer. These steps would be typical in a food lab and can introduce contamination and error into the analysis.

Flame AA analysis steps such as instrument calibration and sample preparation can be automated to avoid potential sources of error. Automation has the added benefit of improving the analysis workflow and reducing costs.

The Agilent Sample Introduction Pump System (SIPS 20) is an accessory for flame AA. The SIPS 20 uses dual, autoclamping, peristaltic pumps to automatically prepare up to 10 standards from a single bulk standard. The accessory eliminates the manual preparation of calibration standards, provides fast, accurate online dilution of overrange samples, and provides online addition of ionization buffer during analysis. The SIPS 20 can automate the analysis of food samples, such as milk products, where Na and K are often present at high concentrations. The high concentrations often require the addition of an ionization buffer and the dilution of overrange samples.

Agilent SIPS 20 Accessory

Learn more about the Sample Introduction Pump System (SIPS 20) accessory in this technical overview.

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Imagine doing elemental analysis using air

The Agilent 4210 MP-AES is the ideal alternative to AAS for elemental analysis. The technique uses air instead of flammable gases and:

- Saves time and reduces operating costs.
- Improves lab safety
- Offers better analytical performance, compared to flame AA
- Enables reliable unattended multi-element overnight operation

Download handbook to learn more

Q. What are the benefits of using an automated AA spectrometry technique over another newer method?

A. AA spectrometry is the simplest technique, especially when using an autosampler and an accessory like the SIPS 20 to create fully automated analysis. You could use an ICP-OES or even an ICP-MS instrument, but they cost significantly more than an AA instrument. The purchase price of a flame AA system is less than half that of an ICP-OES and at least four or five times less expensive than an ICP-MS.

The other big difference between ICP-OES and ICP-MS, compared to AA spectrometry, is the way samples are vaporized and atomized. ICP techniques use an argon plasma to atomize/ionize samples. Argon is significantly more expensive than the air and acetylene used with the automated flame AA technique.

Flame AA does have a draw back. It is dangerous to leave the instrument running and unattended in the laboratory—especially overnight. By contrast, it is not a problem to leave ICP instruments running unattended.

The most recent elemental analysis technique is the microwave plasma atomic emission spectrometer (MP-AES) developed by Agilent. The microwave plasma technology runs on air, using nitrogen extracted from the ambient environment. As no flammable gases are needed, this instrument can run unattended. The price of an MP-AES is between that of a flame AA instrument and an ICP-OES. The price, detection limits, and improved dynamic range of MP-AES make it an excellent alternative for food analytical laboratories, compared to flame AA. In the future, we believe that many flame AA users will transition to microwave plasma instruments rather than argon plasma systems purely due to cost considerations.

Q. How about beverage samples like wine? How would you analyze this type of sample?

A. Wine is a complex matrix. Samples include ethanol, sugar, tannin, and other organic substances. One of the biggest factors in the quality control of wine is measuring concentrations of metals like iron and copper. These metals must be controlled to ensure they do not exceed certain concentrations that could affect the quality and taste of the wine. For similar reasons, calcium and potassium levels are important information for the winemaker to have before bottling.

For wine analysis, you can use methods developed by International Organization of Vine and Wine (OIV). These methods cover the analysis of certain elements (e.g. sodium, potassium, calcium, magnesium, iron, copper, zinc, and manganese) by flame AA spectroscopy, ICP-OES, or ICP-MS. Again, the flame AA is the simplest to use while offering good sensitivity and accuracy, without the interferences that can be present in ICP techniques.

Wine and beverage require determination of multiple elements. Flame AA typical involves the measurement of one element at a time, which requires a sample to be presented to the spectrometer multiple times for each sample to analyze all the required elements.

Agilent Flame AA instruments include a function called Fast Sequential (FS) mode. Using this mode, multiple elements are measured each time a sample solution is presented. FS mode turns on all the instrument's hollow cathode lamps simultaneously and all eight elements in wine are measured in a single aspiration of a sample. Using an AA in this mode allows the instrument to operate in a similar way to MP-AES, ICP-OES and ICP-MS instruments.

The SIPS 20 accessory simplifies wine analysis by automatically calibrating the instrument from a single bulk standard. It also dilutes the sample if necessary and introduces the ionization buffer. FS mode analyzes all eight elements from one aspiration, reducing sample mix-up errors and simplifying the analysis workflow.

FS mode offers several benefits such as dramatically reduced analysis time and smaller sample volume. The feature also reduces the amount of gas consumed by the instrument reducing operational costs. Using multi-elements lamps, even a four-lamp position instrument can analyze 14 elements from a single solution.

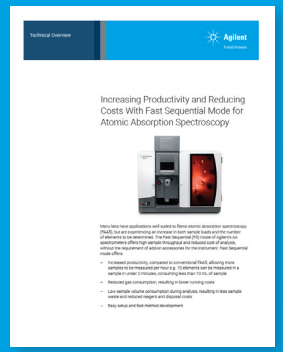
Agilent instruments also include a software function called PROMT (Precision Optimized Measurement Time). This function allows you to set acceptance criteria for precision, to optimize and reduce the measurement time. These features together—FS mode, SIPS 20, and PROMT—offer considerable time savings without compromising precision.

An ionization buffer is needed for sodium and potassium, per the OIV OENO 18/2003 standards. Again, it is possible to add the ionization buffer automatically during analysis with the Agilent SIPS 20 accessory, reducing the manual preparation required.

The Agilent SIPS 20 features can also be used to avoid manually matrix matching. Normally, when measuring wine samples, it would be necessary to spike your standards with approximately 12% ethanol to match the normal concentration of ethanol in wine. The SIPS20 can automatically perform matrix spikes, minimizing any matrix effect. In cases where its not possible to matrix match the SIPS 20 can also automatically perform method of standard addition from a single stock solution.

Internal standard correction is another useful capability, which is only possible using Agilent flame AA's FS mode. Matrix effects are controlled without the need to matrix match, allowing for samples to be analyzed directly. Using FS mode and a SIPS 20 accessory, sample analysis is simplified by letting the internal standard control the matrix effect, like an MP-AES, ICP-OES, or an ICP-MS system.

You can also use a specific template developed for 240FS, SIPS 20 and PROMT mode. The template includes 14 elements and 18 wavelengths and is precalibrated to run a semiquantitative analysis of your sample in less than 2 minutes in flame AA to gain more insights into your samples.



Learn more about Fast Sequential (FS) mode in this technical overview

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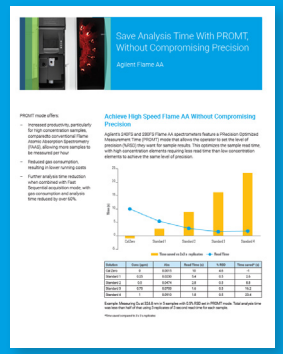

Learn how the Precision Optimized Measurement Time (PROMT) mode allows you to set the level of precision you want for sample results.

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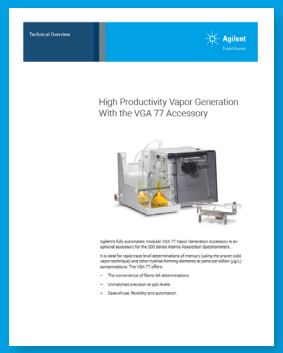

Learn how the Agilent VGA 77 Vapor Generation Accessory offers the convenience of flame AA operation with low ppb detection and automation

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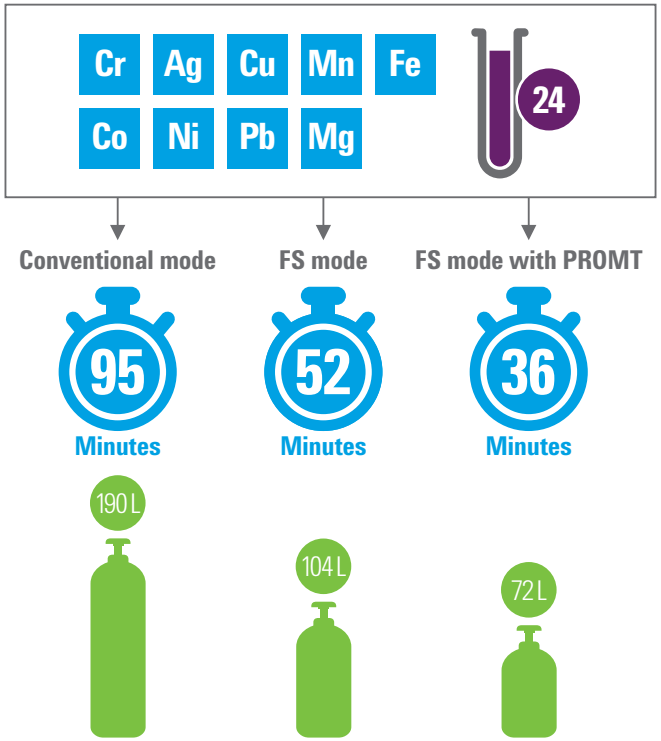
Q. How much time is saved using the Fast Sequential technique with AA spectrometry?

A. FS mode offers approximately a 35–50% improvement in workflow efficiency, compared to traditional AA measurements. The total time saved depends on the number of elements and replicates. An example of the time and gas savings offered by FS mode and FS mode combined with the PROMT software function is shown below.



Nine elements in 24 samples were quantified in three different ways: Conventional FAAS mode (3 integrations of 3 seconds for each element), Fast Sequential mode, and Fast Sequential mode with PROMT acquisition. The analysis used an autosampler, included a Calibration Zero and three standards. A 5 s rinse was performed every 10 samples.

Q. Can low concentrations of heavy metals like arsenic or mercury be analyzed on flame AA instruments?

A. Regulations obligate food producers to strictly control heavy metals, like arsenic and mercury. Flame AA instruments can be used to analyze hydride elements like arsenic and mercury at ppb or even sub-ppb levels, with use of the Agilent VGA 77 vapor generation accessory.

Using vapor generation, the sample goes through a prereduction process, and is then reacted with sodium borohydride to form a volatile metal hydride. Mercury is turned into an atomic vapor, where other hydride forming elements like arsenic require heating via the flame or through electrothermal heating. The sample is then passed through to the spectrometer to measure the element of interest. The main advantage of the hydride generation is the separation of the analyte from the matrix. This separation enables the reduction or even elimination of interferences and increases sensitivity. But, it's important to note that this technique of hydride generation can only be used for a few elements such as arsenic, selenium, or mercury.

Q. How can I determine low concentrations of other heavy metals such as lead and cadmium?

A. Flame AA will not be able to reach the required quantification limits for lead and cadmium in most regulated environments. The hydride generation technique also won't help. So, we must use a different technique: graphite furnace AA spectrometry.

Graphite furnace AA spectrometry (GFAAS) is an extremely sensitive technique. GFAAS can be used to reach the same ppb or sub-ppb levels as those achieved for arsenic and mercury hydride generation. It can offer similar sensitivity to ICP-MS, at a significantly cheaper price, making it much more accessible for food labs.

Graphite furnace AA spectrometry is much slower than flame AA spectrometry. Each replicate takes about two minutes. It would take six minutes to complete three replicates, whereas flame AA spectrometry would take six to nine seconds for three replicates.

Most labs using a graphite furnace instrument use an autosampler to automate common standard and sample preparation tasks. The PSD 120 autosampler, a standard feature on all Agilent GFAAS instruments, can accurately prepare calibration standards and perform sample preparations including modifier addition and dilution.

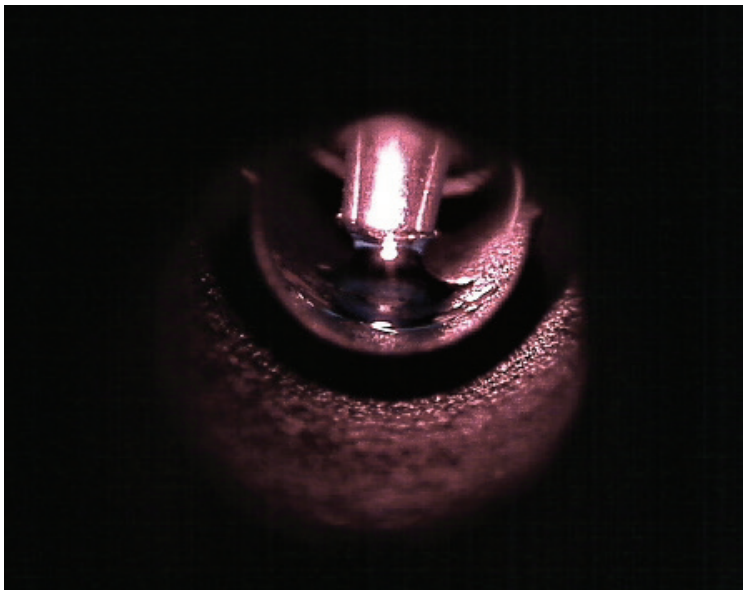
Zeeman background correction is the most effective background correction to ensure accurate quantification of analyte elements in the most challenging matrices. It is a must for food samples, like milk and wine, which have complex matrices made up highly variable concentrations of sugars, salts, and other organic compounds.

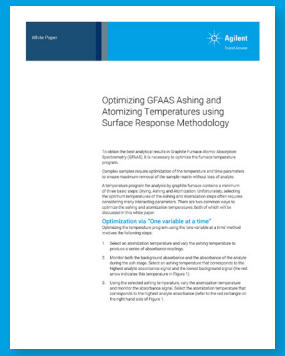
Graphite furnace analysis can prove to be challenging as an understanding of analyte chemistry is often required. Agilent's chemometric software, the Surface Response Methodology (SRM), can help you determine the optimal relationship between the three critical parameters for graphite furnace analysis:

- ashing temperature,
- atomization temperatures, and
- analyte absorbance

The SRM Wizard automates method development using 12 measurements on a representative sample, aiming to optimize sensitivity and accuracy. The Wizard ensures high performance and low-level quantification of controlled elements, like cadmium and lead.

Also, the included high definition color camera, 'Tube-Cam' shows real time viewing of the interior of the graphite. You can use this tool to monitor the critical drying steps to see if the sample is dispensing correctly, and boiling or drying well.





White paper: Optimizing GFAAS Ashing and Atomizing Temperatures using Surface Response Methodology

Download this whitepaper to discover how graphite furnace temperatures can be optimized using Surface Response Methodology.

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AA Troubleshooting and Maintenance Guide

Learn more about our best tips for optimizing your AA instrument, and how you can ensure your methods are robust and reliable.

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Q. What about optimization of the flame AA system? Can you give me some tips to help me with that?

A. The Agilent Mark 7 flame atomization system provides the capability to routinely handle high matrix samples, such as food and beverage samples with the flexibility to achieve high sensitivity.

First check that the mixing paddles are fitted inside the spray chamber. They are recommended for most applications.

Use the Agilent burner aligning and cleaning strips to ensure the burner is correctly aligned with respect to the light from the hollow cathode lamp. It's also good practice to check the nebulizer uptake rate. Ideally it should be close to 5 mL/min. Adjust if necessary.

Then aspirate a calibration standard and optimize the impact bead position so that you're getting the best combination of sensitivity and precision.

These adjustments will also help you reduce the chance of blockage of the sample introduction system. And, of course, that means you can get better long-term performance and stability.

Remember too that at the end of analysis, you should flush out the sample introduction system with an extended rinse. This means less chance of blockage the next time you need to do your analysis.

Q. Can you give me an overview of what the typical maintenance schedule for a flame AAS instrument would be?

A. These are the recommended maintenance schedules for flame AA – the timing may vary depending on your application and workload. Adjust these as necessary.

There are a number of checks you should complete before using the instrument.

- Check that there is enough gas in the cylinders at the start of the run, especially important for the acetylene to make sure that there is no carryover of acetone into the gas box of the instrument
- Check that the exhaust is working correctly
- Check the nebulizer uptake rate and inspect the burner to make sure it's clean (no carbon or matrix build-up)

Then after analysis is complete, follow the recommended shut down procedure to wash out the sample introduction system, and then empty the waste container. It's also good practice to wipe down the exterior surfaces of your atomic absorption system especially in the sample compartment to remove any acid residue that may have built up on those surfaces.

On a weekly basis or perhaps as necessary, clean both the burner and the flame atomization system. The burner should be cleaned inside and on the outside of the burner slot using a metal polish like "Brasso". The Agilent burner cleaning strips make it easy to polish the inside of the burner slot. A well polished burner will give you much better performance and much better resistance to blockage.

Cleaning the spray chamber is straightforward—disassemble and wash in a detergent solution. While doing that, inspect all the components, particularly the impact bead and the condition of all the O-rings, to make sure they're in good condition. If the O-rings have nicks or are damaged or stretched, replace them immediately to ensure a good seal.

Periodically check the optical windows on the instrument and clean as necessary.

If you follow these procedures, you should be able to achieve good and consistent performance from your instrumentation.

Q. Is the optimization and maintenance schedule for a graphite furnace AAS instrument different?

A. The same optimization and start-up principles we outlined for the flame AA instrument can be applied to a GFAAS instrument.

There are a number of checks you should complete before using the GFAAS instrument.

- Check that there is enough inert gas in the cylinder at the start of the run
- Check that the exhaust is working correctly
- Check that there is sufficient solution in the rinse bottle and that this has been acidified with a few drops of detergent added (improves dispensing and keeps the capillary tip clean)
- Check the condition of the graphite tube. Use the firing counter to monitor how long the tube has been in service and replace when necessary (replacing the tube regularly will help you achieve the best performance)
- Ensure the workhead position has been optimized for maximum light throughput
- Verify that the dispensing capillary of the autosampler is aligned to dispense sample into the graphite tube. Use the Tube-Cam to check the dispensing height is correct

Then check that the temperatures and times programmed in your furnace program have been optimized, particularly the drying conditions. Use the SRM Wizard and Tube-Cam to assist.

Then introduce a calibration standard and verify that you're getting the expected sensitivity and precision. The system is then ready for analysis.

Maintenance on the GFAAS is mostly focused on the workhead and the autosampler. When you replace the graphite tube, check the condition of the graphite electrodes and wipe them to remove any residue. Typically, the electrodes will last at least for one year.

When analysis is complete, it's good practice to remove any residue built up around the sample injection hole. Then remember to empty the waste container.

Periodically check if there are any leaking seals on the dispensing syringe of the autosampler and check the optical windows on the instrument and clean as necessary.

Q. What supplies should I keep on hand to ensure I can maintain operation of my AA instrument?

A. All instruments will need hollow cathode lamps as well as the standards (CRMs) that are used to prepare your calibration standards and any ionization suppressants or modifiers you're using.

For a flame AA system, it's recommended to have parts for the sample introduction system, such as spare impact beads, burner cleaning strips, nebulizer components, capillary tubing etc. That ensures that if you suffer a blockage or a breakage, you can disassemble the nebulizer, replace the component, and continue the analysis.

For graphite furnace AA systems, you should have spare graphite tubes, sample vials, dispensing capillaries and a spare syringe for the autosampler.

For vapor generation AA systems, you should have spare quartz atomization cells, peristaltic pump tubing, and connecting tubing.

Agilent does offer a range of operating supplies kits that bundle all the supplies you need in a single kit. They're available for flame AA, graphite furnace AA and vapor generation AA. Each of these kits includes all the components typically required to support operation for up to one year of routine operation. This is a convenient way of ensuring you've got the spares that you need at the time that you need them.

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