TECHNICAL NOTE

Atomic Absorption

Analytical Lifetime of PerkinElmer Graphite Tubes – A Checklist

At PerkinElmer, the improvement of graphite tube technology is given the same importance as continuous improvement of instrument hardware and software. This is a major reason why interferences in graphite furnace atomic absorption (AA) are now under much better control. If the PerkinElmer® Stabilized Temperature Platform Furnace™ (STPF) concept is completely and correctly applied, spectral and non-spectral interferences are virtually eliminated in graphite furnace AA.

In addition to reduced interference effects, improvements have also been realized in increasing average tube lifetime. These improvements have resulted from a number of enhancements, including the use of advanced graphite materials developed by experts in graphite technology exclusively for PerkinElmer and in cooperation with PerkinElmer scientists. Another factor is the use of lower atomization temperatures. The use of integrated absorbance measurement (peak area, a part of the STPF concept) significantly lowers the atomization temperatures required without sacrificing performance. Use of automatic sampling devices for sample dispensing not only automates and improves analyses, it also provides longer tube life. Atomization from a platform prevents direct contact of strongly acidified samples with the tube walls and prolongs tube lifetime as well.

Other features of PerkinElmer graphite tubes include the addition of a mark on the tubes in the form of a dot. This marking indicates the orientation of the platform and guides the user on the proper installation into the graphite furnace. Consistent installation of graphite tubes facilitates accurate and precise analytical results.

In practical application of the graphite furnace, there are a number of techniques which can be used to ensure that you obtain maximum analytical and mechanical graphite tube lifetime, including:

- Cleaning and Inspection of the Graphite Contacts
- Thermal Conditioning
- Thermal Pretreatment
- Atomization Temperature
- Atomization Time
- Influence of the Matrix
- Cooling
- Inert Gas
- Lifetime of the Graphite Contacts

Cleaning and Inspection of the Graphite Contacts

When replacing a graphite tube, the inner surfaces of the graphite contacts should always be cleaned. A cotton-tipped applicator can be used for this cleaning. After cleaning, the contacts should be carefully inspected, especially after a mechanical breakage of a tube, and replaced if damaged.

Syngistix™ for AA software features a tool for counting the number of firings on a tube and the contacts. A reminder may also be set to pop up when the tube or contacts should be replaced to minimize the risk of tube breakage.

Thermal Conditioning

After insertion, every new tube should first be thermally conditioned. It is recommended to utilize the "Condition Tube" feature in Syngistix for AA software to automate the graphite tube conditioning process.

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Thermal Pretreatment

It is very important for good analytical results and for long tube life to use a ramp time for thermal pretreatment. Even for very simple sample matrices, a maximum temperature increase of approximately 50 °C/s is recommended. For more complicated sample matrices, a ramp of 20 to 50 °C/s should be applied. Above all, conditions should be avoided where very sudden fume development occurs. If necessary, the thermal pretreatment should be separated into two or more steps for better control of the matrix decomposition.

Atomization Temperature

For optimum performance, atomization temperatures higher than 2600 °C should not be used because the vaporization rate of carbon is accelerated at higher temperatures, leading to premature tube degradation. For a number of difficult-to-atomize (refractory) elements, application of a higher atomization temperature initially increases the signal heights, but it drastically reduces tube lifetime. Higher atomization temperatures are not analytically justified.

Atomization Time

When using integrated absorbance measurements (peak area), atomization time has to be selected to be sufficiently long for the signal to return to the baseline value. High atomization and heating temperatures should be applied only for the minimum time necessary to obtain a suitable reading. Any longer period can significantly reduce tube life.

Influence of the Matrix

The sample matrix itself may have a strong negative influence on tube lifetime. Strong acids or oxidizing agents, such as HNO_3, $HClO_4$ and H_2O_2, are particularly bad in this regard. For example, if nitric acid penetrates into the graphite tube walls, a sudden temperature increase can create gaseous decomposition products inside the graphite lattice. The graphite lattice can be partially damaged and become even more porous, amplifying the effect during subsequent measurements until the tube breaks.

To avoid this type of problem, always minimize the acid concentration used, avoid strong oxidizing agents if possible, use a slow ramp for thermal pretreatment and use platform atomization.

Cooling

PerkinElmer graphite furnaces are designed to provide an automatic cool-down step. To cool the tube from the applied atomization or heating temperature to ambient temperature usually takes less than 20 seconds. If required, an additional cooling down time step may be added to the method.

The use of a PerkinElmer circulatory cooling unit is always recommended. This unit always provides the same water flow, and cooling is efficiently set to reach a stable temperature of about 40 °C. When using a circulatory cooling unit, a longer tube life is generally observed.

When the circulatory cooling unit is not available, tap water may be used for furnace cooling. The recommended cooling water temperature is 20-40 °C with a flow rate of 2 L/min ±0.5 L/min. However, if the temperature of the tap water is too low and/or the flow rate of the water is too high, there is a possibility that condensation of atmospheric humidity can occur on or around the graphite contacts or on the furnace windows. Moisture on the contacts causes rapid attrition of the graphite surface affecting the tube's lifetime, while moisture on the furnace windows leads to attenuation of the energy.

Inert Gas

If nitrogen is used as the inert gas, lower peak sensitivity may be obtained for some elements. In addition, at temperatures above 2300 °C, cyanogen (C_2N_2, a toxic gas) may be generated, making continuous use of nitrogen in a small, poorly ventilated room potentially dangerous. For these reasons, it is recommended that only argon be used. Argon purity should be at least 99.996% with no more than 5 ppm oxygen and 4 ppm water. Higher oxygen and/or water concentrations may decrease the useful lifetime of the graphite tubes.

An exhaust vent is very often used to remove fumes and unpleasant odors generated during thermal pre-treatment or atomization. For the graphite furnace technique, it is recommended to have an exhaust rate approximately 200 cfm, however the vent should not have an excessively high draft. An excessively high draft may also remove much of the argon used to shield the tubes and contacts and reduce their useful lifetimes.

A suitable exhaust vent system is offered by PerkinElmer.

Graphite Contact Lifetime

The graphite contacts also have a limited lifetime and have to be replaced as required. This will depend strongly upon the temperature and time programs applied and the sample matrix. When replacing graphite contacts, they must be properly inserted.

Worn or corroded graphite contacts seldom directly reduce tube lifetime. However, they have a strong influence on the furnace temperature accuracy. The condition of the contacts should be checked regularly.

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