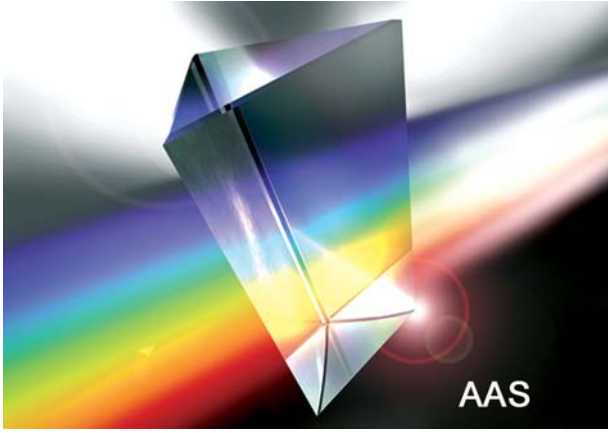
Application Note



Improvements in Detection Limits in Atomic Absorption Spectroscopy using the new digitally controlled Graphite Furnace - GFA-7000

With increasing environmental awareness, the requirement for greater quality control in our daily routines has led to the need for more precise analytical measurements such as Pb in drinking water. The analysis of heavy metals is one of the most routine tasks in every AAS laboratory. Using systems like the AA-7000 with a graphite furnace, large multi-element sequences allow the analysis of trace heavy metals like Arsenic, Copper and Lead to be successfully determined.

The Environmental Council of the European Commission; (11th March 1999) has decided to lower the tolerance level for heavy metals in drinking water, the value of 50 µg/l for Lead will be decreased to 10 µg/l because of the potentially harmful effects of Lead to the nervous systems of children and pregnant women. This decision to lower the allowed limit for Lead and other toxic trace elements was one of the major reasons for the development of the new graphite furnace GFA-7000.

The concept was to maximise sensitivity whilst maintaining a long graphite tube lifetime (2000 injections; tested using 5 ppb of Chromium at 2800 °C). The GFA-7000 digitally controls the temperature and gas flows, and automatically optimises the instrument parameters for each target element. This digital control of the heating rates and actual temperatures is important for all the drying, ashing and atomisation stages Figure 1 shows the effect of the heating rate on the absorbance signal.

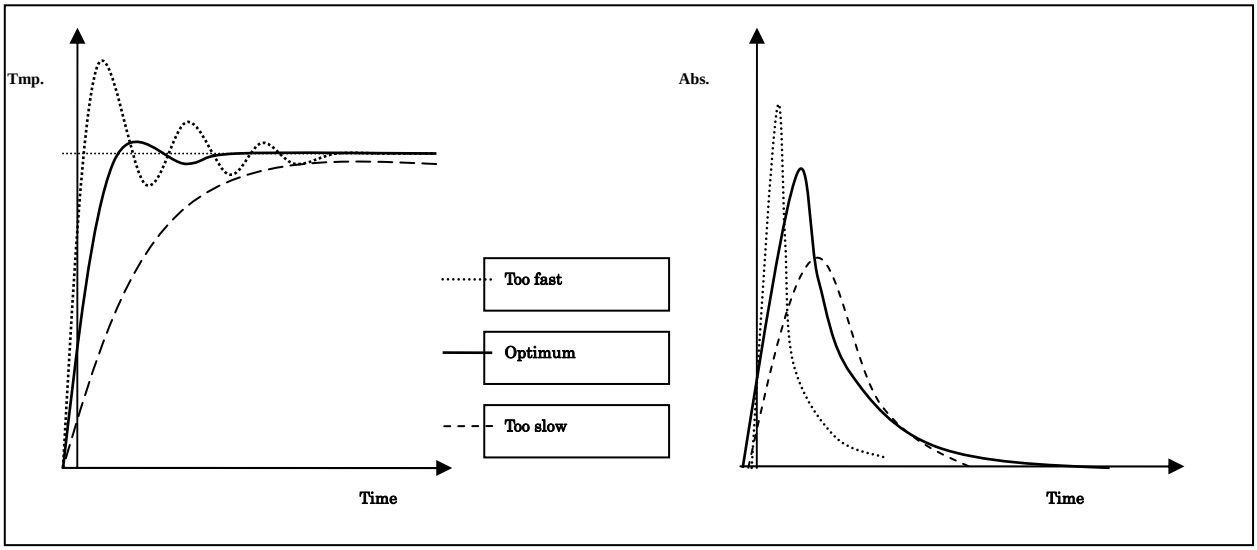


Fig. 1: Effect of heating rate on the absorbance signal

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A fast heating rate causes a high absorbance signal but during this heating rate the target temperature may be over shot resulting in poor repeatability, especially if we consider that these so called transient signals are generated and calculated within a fraction of a second.

Conversely a slow heating rate results in good repeatability but with lower absorption signals. Digital temperature control optimises the characteristics of the system, producing high absorbance values with very good repeatability. Furthermore, the system is intelligent enough to differentiate the heating rates for each pre-set temperature; for example, slow heating to approximately 1000 °C and then fast heating to 2500 °C, in order to achieve the pre-set atomisation temperature in a fraction of a second. This repeatability data is shown in Figure 2; the results are for 700 injections of a 5 ppb Pb solution resulting in an average value of 0.5 % RSD. The temperature step between the ashing and atomisation stages should be as small as possible in order to achieve the target atomisation temperature quickly and to avoid excessive expansion of the inert gas (Ar). During atomisation the electronic flow controllers achieve a precise flow of inert gas through the graphite furnace tube, stopping the gas flow in the last 3-5 seconds of ashing, in order to prevent the atom cloud being expelled from the tube. This improves the residence time and repeatability of the target element (as shown in Figure 3).

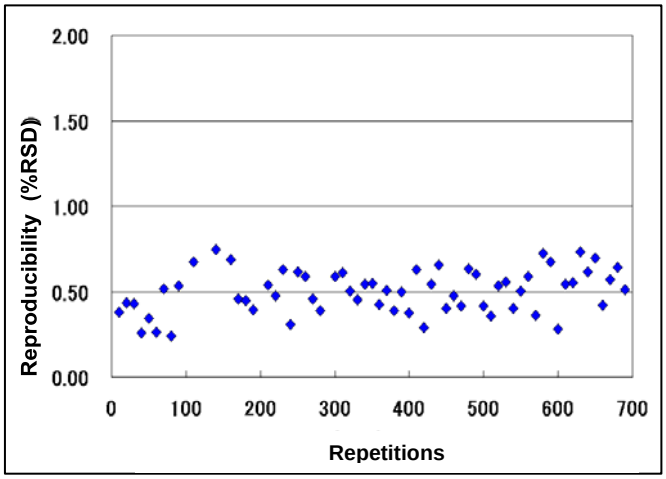

Fig. 2: Result of 700 injections of Standard Pb Solution

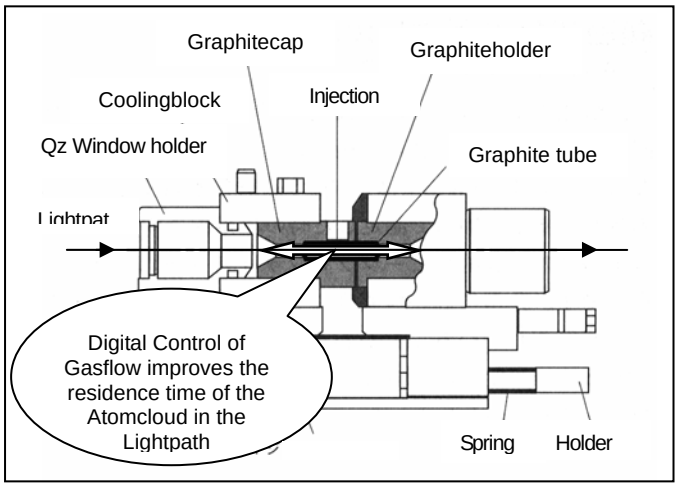

Fig. 3: Principle of the GFA-7000 graphite furnace

System Configuration

Atomic Absorption Spectrometer:	AA-7000F
Auto Sampler:	ASC-7000
Graphite Furnace:	GFA-7000

Tab. 1: Detection Limits (20 µl injection, Pd Modifier *)

Element	GFA-EX7	GFA-7000
Pb *	0.085 µg/l	0.05 µg/l
Mn	0.015 µg/l	0.008 µg/l

An important criteria of performance for graphite furnaces is the detection limit, this can be judged according to DIN regulation - 51461 Part 1. This states that the detection limit is a measure in an analytical method and can be defined as the amount or mass of the detectable element. This amount can be determined using a predefined statistical limit and the detection limit can be calculated from the measured absolute standard deviation of the blank solution and the reciprocal of the sensitivity for the target element.
For the determination of Pb it is possible to calculate the standard deviation by measuring a blank solution ten times and then multiplying this by the reciprocal of the sensitivity (calculated from the calibration curve). Additionally a statistical factor of 3σ should be used and needs to be taken into consideration during the comparison of data from the GFA-EX7 and GFA-7000.
A comparison of this performance data shows a 30 to 50 % improvement in the detection limits using the GFA-7000 compared to the GFA-EX7. This improvement in sensitivity makes the GFA-7000 even more flexible, being able to produce precise and reliable results even if further reduction of the tolerance levels continues in future.