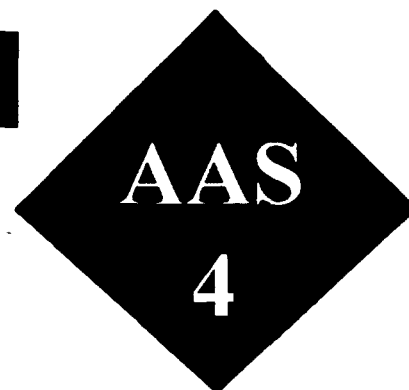


APPLICATION



HIGH SENSITIVE AA-6501S GRAPHITE TUBE LIFETIME TEST

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The AA-6501 is a fully automatic atomic absorption flame emission spectrophotometer with extraordinary performance capabilities. Flame and furnace operation as well as hydride generation techniques are controlled via user-friendly software.

A special feature of this system is the combination of atomization by flame and furnace on one platform in the instrument. Switching from flame to furnace or vice-versa is simply done by a single software command without any further hardware manipulation. In flame operation mode, automatic optimisation of burner height and combustion conditions are stored with each method. For electrothermal atomization, optimal positioning of the graphite furnace is performed automatically. In addition to optimum positioning, the graphite tube deterioration is monitored after each analysis and an automatic correction of the drying temperature is performed based on the detected condition of the graphite tube, this increases reliability of each analysis.

A high sensitive optical sensor combined with a special control system allows accurate temperature at the ashing as well as the atomising stage. All these features result in high efficiency during daily work. Further technical modifications realised in the AA 6501S result in improved sensitivity and prolonged lifetime of

the graphite tube. Continuous advancement in graphite furnace design and techniques since 1970 has resulted in the development of a highly sensitive system with longitudinal heating.

The concept of this new graphite furnace is shown in figure 1.

It was the aim of this development to extend the dwell time of the atomic cloud in the graphite tube during atomization. To achieve this a magnetic valve unit is applied to control the internal Argon gas flow with high efficiency. The total volume of the furnace was enlarged and a PTFE seal placed between the graphite tube holder, providing a semi-closed system.

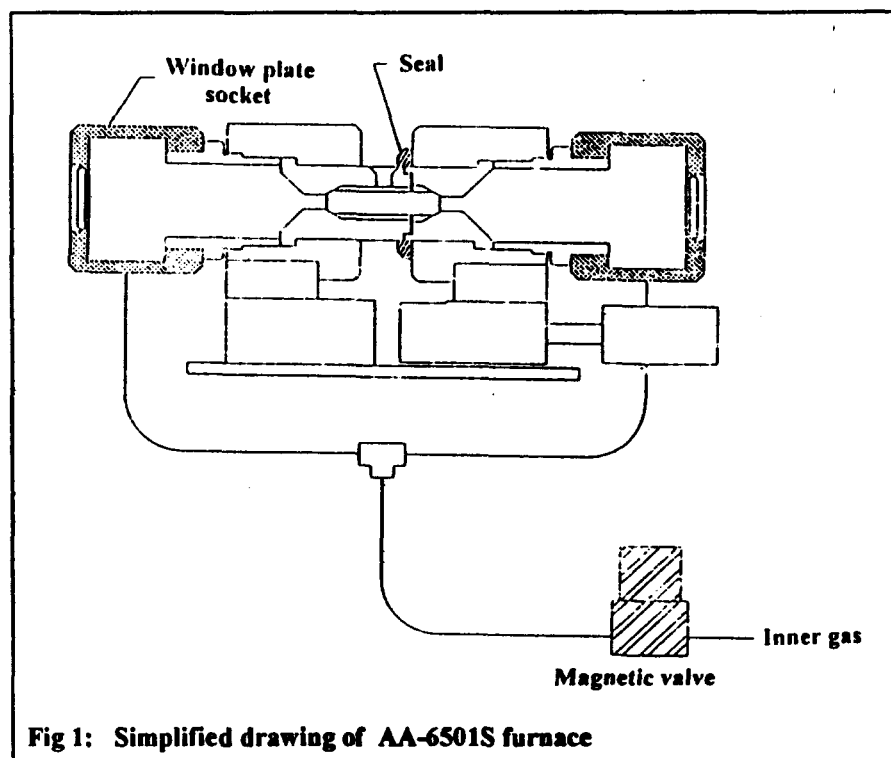


Fig 1: Simplified drawing of AA-6501S furnace

Table 1 shows some data of selected metals.

Element	Wavelength [nm]	Slit width [nm]	char.mass [pg]
Al	309.3	0.5	2.7
As	193.7	0.5	4.4
Cd	228.8	0.2	0.1
Cr	357.9	0.5	1.0
Cu	324.8	0.5	1.0
Fe	248.3	0.2	2.4
Mn	279.5	0.5	0.4
Ni	232.0	0.2	3.3
Pb	283.3	1.0	2.6
Sb	217.6	0.2	3.4
Se	196.0	0.5	5.6

Tab. 1: Characteristic mass in pg calculated from 1% Abs (correspond to 0.0044 AU), 20 µl Injection

The technical improvement does not only effect positively in high sensitive measurements but also, due to perfect temperature control from GFA-6500 furnace control unit, in prolongation of the graphite tube lifetime.

As an example, Fig's 2 and 3 show the excellent reproducibility of over 2,000 measurements for determination of chromium at an atomising temperature of 2800 °C from one single tube. A combination of the ASC-6000 and

long lifetime graphite tubes has the advantage of fully automatic multi-element analysis on a large number of samples, thus reducing the running costs of the system.

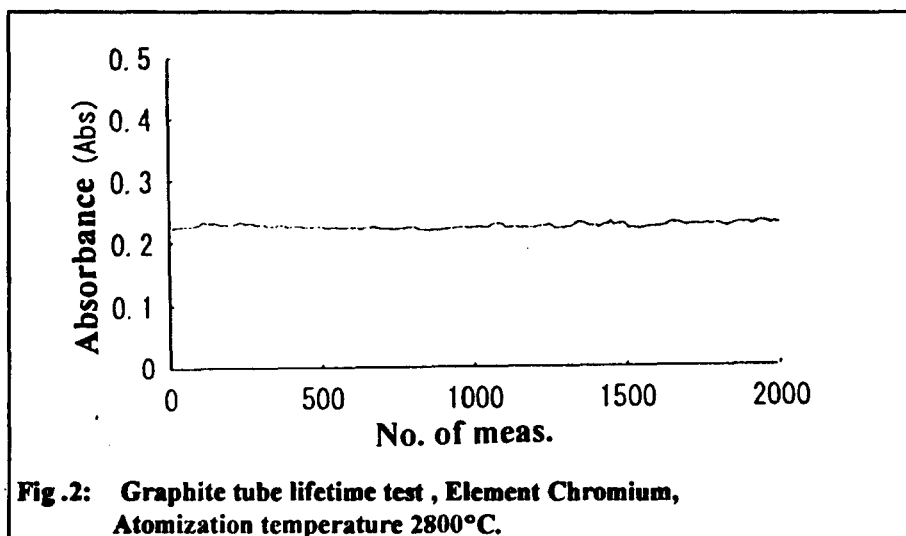


Fig.2: Graphite tube lifetime test , Element Chromium, Atomization temperature 2800°C.

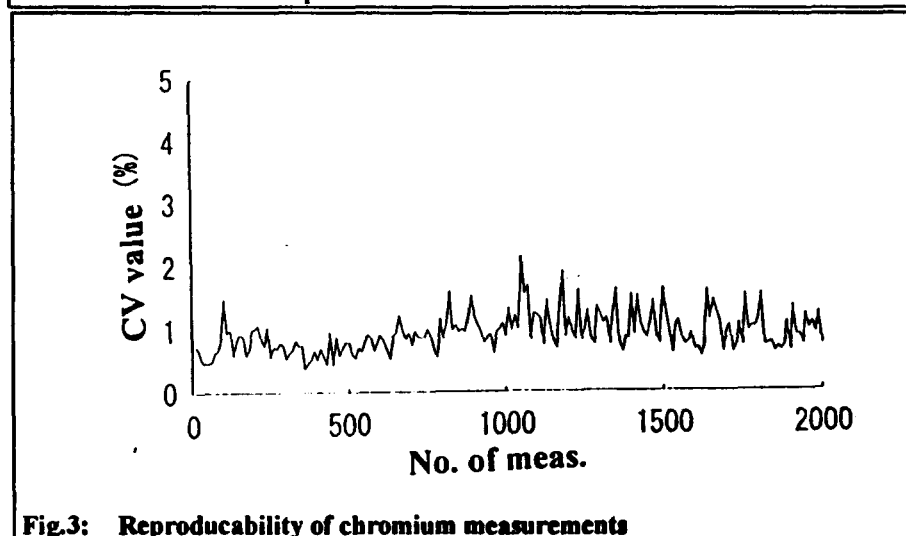


Fig.3: Reproducibility of chromium measurements

**** Furnace Meas. Conditions ***** 1993/11/05 12:54:15 ****

Instrument : Lamp : Zn #1
 Current : 8 mA / 0 mA
 λ : 307.6 nm
 Slit Width : 0.5 nm
 Mode : BGC-D2
 Analyst : Steinkrauss
 Comment : Zn bei 307.6

Measurement Parameters :
 Conc. Conversion Mode : Calibration No. of Standards : 3
 Signal Processing : Peak Height Calib. Curve Order : 1st
 Shift to Origin : ON
 Response : DIRECT Conc. Unit : ppm

Repeat Conditions : No. of Repts. Max. No. of Repts. CV Limit
 Blank Measurement : 2 2 99.9 %
 Standards (STD Addition) : 2 3 5
 Sample (Simple STD Add.) : 2 3 5
 Sens. Correction : 2 3 5

Furnace Program :
 Conc. Boost Cycle : 1~ 1
 Number of Cycles : 1

Stage#	Temp.(°C)	Time(sec)	Gas (l/min)	Heat	Sampling	PrePEAK
1	120	30	#1 1.0	RAMP	OFF	
2	250	10	#1 1.0	RAMP	OFF	
3	300	20	#1 1.0	STEP	OFF	
4	300	5	#1 0.0H	STEP	OFF	
5	2800	3	#1 0.0H	STEP	ON	2
6	3000	3	#1 1.0	STEP	OFF	

ASC Parameters :
 Auto Dilute & Re-Analyze : ON
 No. of Blank Pre-Rinse : 0
 1) 1st Reagent : R1
 2) 2nd Reagent :
 3) 3rd Reagent :
 4) 4th Reagent :
 Mixing : ON
 Injection Volume : 20 µl
 Injection Speed : 25 µl/sec
 No. of Mixing Cycle : 5
 Intake Speed : 130 µl/sec
 Discharge Speed : 330 µl/sec

STD No.	Conc. ppm	Position R1~R8/1~60	Volume (µl)	R1 (µl)	Total (µl)
1	0.1000	R2	40	360	400
2	0.5000	R2	200	200	400
3	1.0000	R2	400	0	400

	Position R1~R8/1~60	Volume (µl)	R1 (µl)	Total (µl)
Blank	R1	100	100	200
Corr.	R2	400	0	400
Sample	--	200	200	400

The outstanding performance and flexibility of this new high sensitivity graphite furnace is demonstrated using the determination of Zinc.

Zinc has a ubiquitous distribution in the environment, furnace analysis can be difficult due to the floating high level blank signals on the analytical line at 213.9 nm. The high sensitivity furnace enables Zinc to be determined on a secondary element line at 307.6 nm.

In comparison to the normally used analytical line at 213.9 nm, this secondary line shows reduced intensity by a factor of 10,000, therefore the high and fluctuating blank signals can be eliminated. The high sensitivity furnace can still determine zinc concentrations below 0.1 ppm.

The high sensitivity mode is activated and indicated by the "H" setting in the furnace program. In addition, an autosampler was used for precise standard and sample injection and, for preparation of the calibration solutions diluting from a top standard of 1 ppm Zn. Dilutions were performed by mixing defined volumes of stock standard solution (R₂) with ultrapure water (R₁). The injection volume was 20 µl.

Fig.4: Parameter set for furnace program and ASC-6000 routines for the determination of Zinc.

APPLICATION

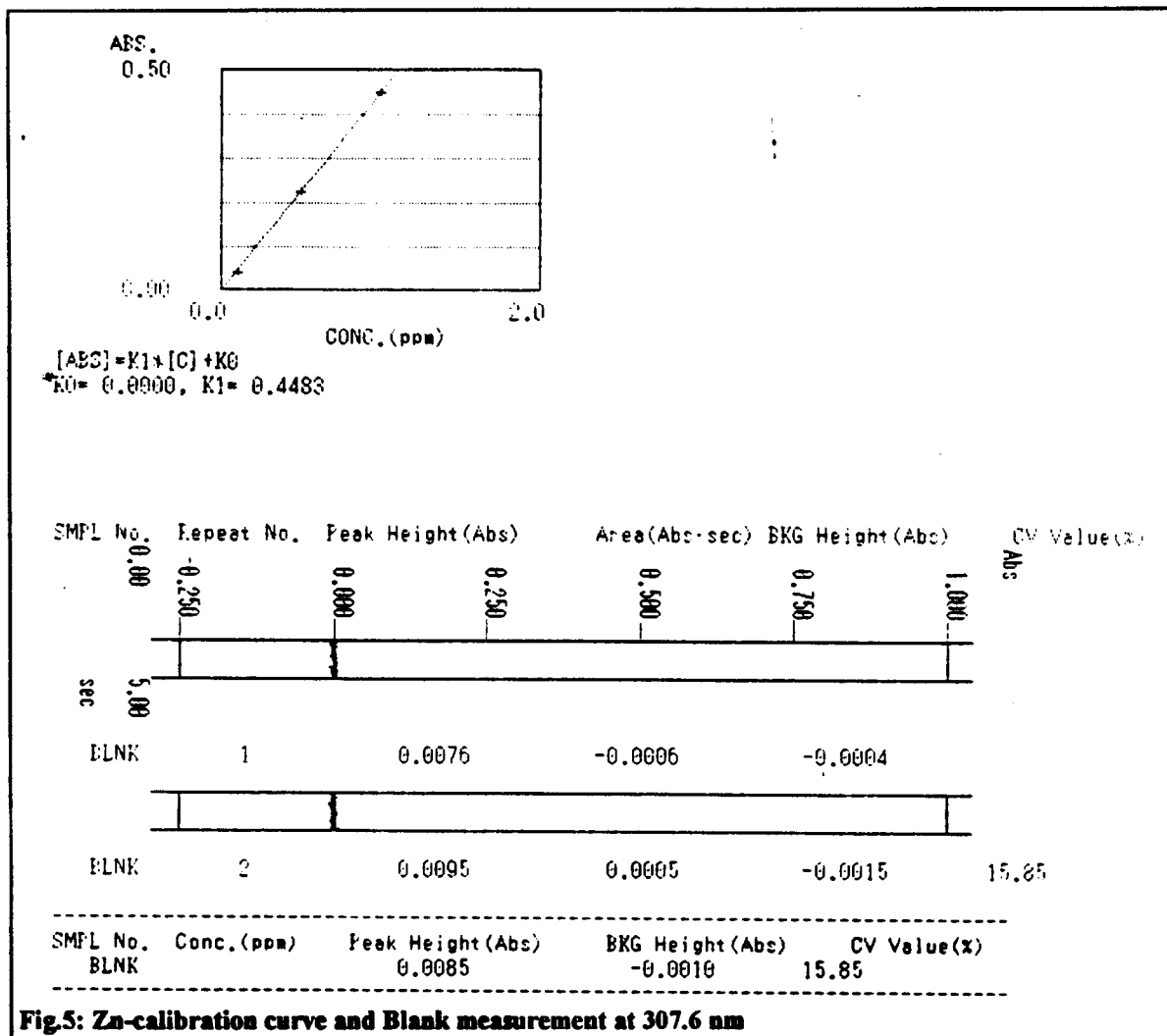


Fig.5: Zn-calibration curve and Blank measurement at 307.6 nm

The results presented in Fig. 5. show the elimination of the effects normally associated with high blank values for Zn determined at 213.9 nm and the reliable analyte values determined at 307.6 nm. Moreover, this example

shows that new ideas in trace analysis can be developed. To overcome measurement problems arising from matrix effects, the easiest method is to dilute the sample. Here traditional furnace systems have difficulty with

sensitivity. With the high sensitivity furnace, high dilution ratio's can be used and accurate analysis under such conditions is possible.