



# Evaluation of the Mark-VI Spray Chamber for Flame Atomic Absorption Spectrometry

## Application Note

Atomic Absorption

### Author

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### Introduction

The atomization system - nebulizer, spray chamber and burner, plays an integral part in the analytical performance of a flame AA spectrometer. The spray chamber in particular plays a crucial role in promoting intimate mixing of the nebulized aerosol with the fuel. Proper spray chamber design ensures the minimization of carbon build-up, burner blockage and volatilization interferences. The spray chamber must also provide excellent drainage to reduce sample clear-out times and signal spikes caused by irregular drainage.

In this paper the performance of the Agilent Mark-VI spray chamber is evaluated. Performance aspects such as characteristic concentration, drainage, carbon build-up, burner blockage and the effect of interferences are investigated.

### Description of Design

In the Mark-VI spray chamber, Figure 1, aerosol production begins with a high efficiency pneumatic nebulizer, employing an inert platinum-iridium capillary and a tantalum venturi. The adjustable glass bead breaks up the aerosol droplets and provides the operator with control over the aerosol concentration. The acetylene is injected tangentially into the aerosol, promoting intimate mixing. As the mixture spirals toward the burner it encounters a twin-head mixing paddle which twice reverses the direction of rotation, promoting further mixing and large droplet removal.

The design incorporates a steeply angled floor and a wide-bore drainage tube to ensure liquid is rapidly drained from the spray chamber. The nebulizer bung is fitted with a ceramic face-plate. This ensures any droplets deflected from the glass bead are not re-nebulized. Re-nebulization of droplets would cause noise spikes in the analytical signal [1].



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Throughout the system high grade polypropylene is used to ensure chemical resistance. Flame safety is maintained by a comprehensive series of interlocks monitoring the nebulizer bung, the pressure relief bung, the liquid trap, the burner, the flame shield and the flame itself. The Mark-VI spray chamber is compatible with Agilent nitrous-oxide and air acetylene burners of the Mark-5A or Mark-5 types.

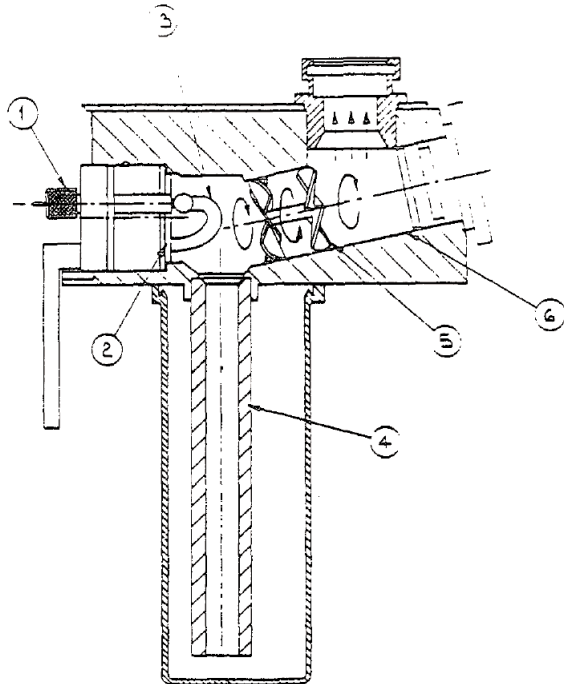


Figure 1. The Agilent Mark-VI spray chamber: (1) nebulizer, (2) ceramic face-plate, (3) adjustable glass bead, (4) drainage tube, (5) duel-head mixing paddle, (6) enhanced slope floor.

## Experimental

In the following evaluations the Mark-VI spray chamber was tested using a SpectrAA-40 atomic absorption spectrometer. Standard recommended conditions of wavelength, slit width and lamp current were used unless otherwise stated. Instrument settings such as burner position, gas flows and nebulizer setting were adjusted to give maximum sensitivity. Where stated the glass bead was then adjusted closer to the nebulizer venturi to reduce sensitivity. The spray chamber and burner were cleaned thoroughly prior to use. The ceramic face of the nebulizer bung was cleaned by immersing the nebulizer bung in a detergent solution in an ultrasonic bath for several minutes: The mixing paddle was cleaned in chromic acid in an ultrasonic cleaning bath. Care must be taken in handling chromic acid. Agilent hollow cathode lamps were used. Standard solutions were prepared in dilute nitric acid in de-ionized distilled water. Agilent Mark-5A burners were used.

## Performance Evaluations

### Drainage

A fully optimized spray chamber should be designed so that residual solution can drain quickly to waste. The system should be designed so that solution is not re-nebulized as it drains, causing noise spikes in the analytical signal. Poor spray chamber drainage will require the use of long delay times between samples to minimize carry-over of solution. An example of the drainage problems associated with poor spray chamber design is shown in Figure 2. Noise spikes characteristic of poor drainage are observed for greater than 100 seconds after the solution aspiration is stopped.

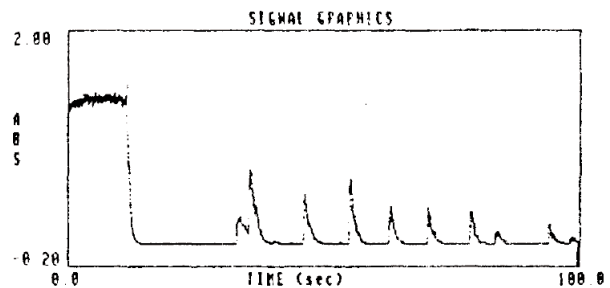


Figure 2. Signal spiking due to poor drainage of a high concentration aluminium standard using a conventional spray chamber.

The floor of the Mark-VI spray chamber slopes steeply to a recessed, wide bore drainage tube. Drainage of aerosol droplets deflected from the glass impact bead is aided by a ceramic face-plate, fitted to the nebulizer bung. This drainage plate is very easily wetted and droplets in contact with this surface drain rapidly to waste and are not re-nebulized.

Significantly the drainage tube is placed at the point of maximum drainage demand - beneath the adjustable glass bead. The shape of the glass bead arm has been optimized so that solution draining from the bead drips almost directly into the drainage hole. In a study of aerosol deposition patterns in a spray chamber O'Grady et. al. [2] showed that the majority of aerosol is deposited within the first third of the spray chamber with or without the glass bead in place. For spray chamber designs with the drainage tube placed under the burner at the rear of the spray chamber, the majority of the drained fluid must traverse the length of the spray chamber before it can be drained.

Figure 3 shows the drainage performance of the Mark-VI spray chamber for a 1000 ppm copper solution using the air-acetylene flame. After aspirating the copper solution for ten seconds, distilled water was immediately aspirated. Figure 3(a) shows that the drainage is rapid and complete - the signal returning to the baseline and showing no sign of spiking afterwards. Figure 3(b) shows this drainage on a smaller time scale indicating that drainage is complete within 5 seconds of aspirating the blank.

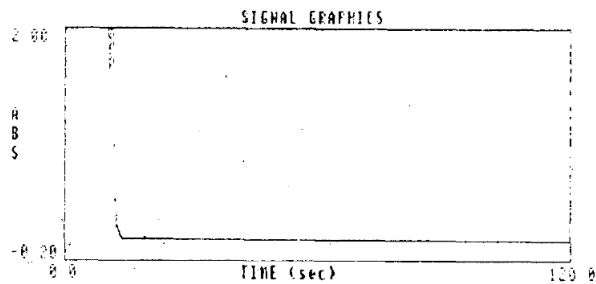


Figure 3a. Drainage of 1000 mg/L copper solution using the Mark VI spray chamber, 10 sec aspiration shown on 120 second time scale.

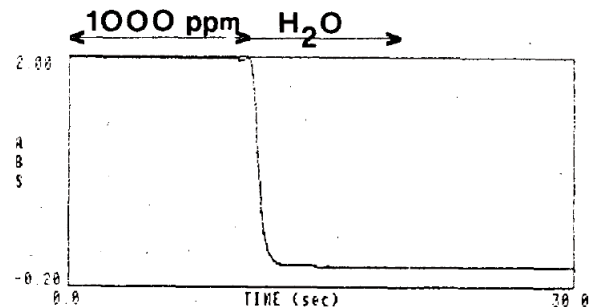


Figure 3b. Drainage of 1000 mg/L copper solution using the Mark VI spray chamber, 10 sec aspiration shown on 30 second time.

## Carbon Build-Up

Flame disruption due to carbon build-up may occur when a nitrous oxide-acetylene flame is used. Carbon build-up is caused by the thermal breakdown of acetylene molecules and is therefore aggravated by the use of acetylene rich flames.

The build-up of carbon usually occurs above the jaws of the burner but may actually block the slot if the deposit is not removed. The carbon deposits may then act as a nucleus for the precipitation of dissolved salts and solids causing further blockage.

The problem of carbon build-up can be minimized by:

- Proper burner cleaning
- Allowing the burner to warm up on a lean flame before analysis
- Using the maximum total gas flow consistent with the required flame stoichiometry
- Using the leanest possible flame

Spray chamber design can also contribute to minimizing carbon build-up. In the Mark-VI spray chamber intimate mixing between sample-oxidant aerosol and the fuel ensures that a correct and even flame stoichiometry is achieved across the burner. The occurrence of localized regions in the flame which are rich in acetylene is avoided, therefore minimizing carbon build-up. The Mark-VI spray chamber can be operated with an acetylene rich flame for longer periods than conventional systems before carbon builds up on the burner.

Further, with the Mark-VI system substantially reduced fuel flows can be used to obtain maximum sensitivity, resulting in reduced carbon build-up. This effect is shown in Table 1. The fuel and oxidant levels required to achieve maximum sensitivity for several elements using the Mark-VI and a conventional spray chamber are listed. The results indicate that a significantly lower fuel level is required when using the Mark-VI system.

Table 1. Spray Chamber Comparison: Gas Flows Required for Maximum Sensitivity.

Element	Nitrous oxide/acetylene (L/min)	
	Mark VI N <sub>2</sub> O/Acet	Conventional N <sub>2</sub> O/Acet
V	11.0/7.5	11.0/8.5
Al	11.0/7.0	11.0/7.8
Si	11.0/7.7	11.0/8.0

## Burner Blockage From High-Solids Solutions

A common problem in flame AAS is the physical obstruction of the burner slot by solids deposited from the sample aerosol. Solvent evaporates from aerosol droplets deposited inside the burner jaws leaving solid material. Such deposits act to accumulate more solids. Eventually disruption of the flame results, the noise level may worsen and the flame may blow out as the gas velocity increases through the constricted slot. Samples containing high concentrations of salt (for example, sea water) or sugar (for example, soft-drinks) are typical of samples which cause blockage.

The problem of burner blockage due to solids deposition can be minimized by optimizing spray chamber design. The action of the mixing paddle and the glass bead to promote the formation of an aerosol of a large number of small droplets ensures passage through the burner slot with minimal deposition.

Performance of the atomization system for difficult samples can be further improved by proper operation. If high solids samples are to be analyzed using a high-vac nebulizer the nebulizer should be operated in the HIGH-SOLIDS position. In this position the platinum-iridium capillary of the nebulizer is advanced through the throat of the nebulizer venturi, as shown in Figure 4.

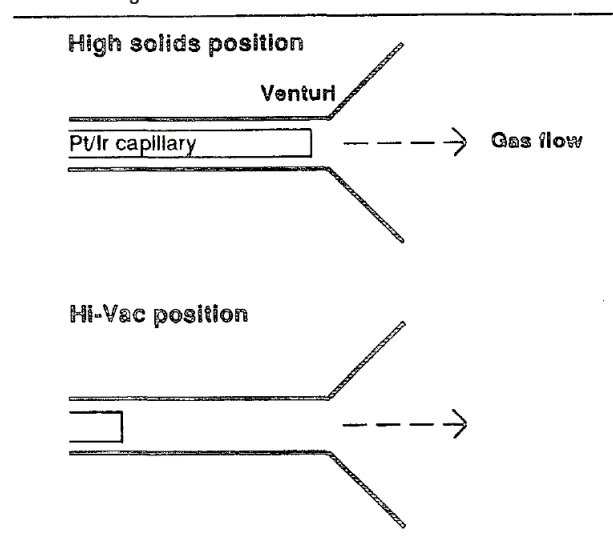


Figure 4. Adjustment of the hi-vac nebulizer.

In this position there is less opportunity for aerosol to deposit in the venturi and is therefore ideal for operation with samples containing a high percentage of dissolved solids. Adjustment to the HIGH-SOLIDS position is simply achieved by rotating the nebulizer thimble fully clockwise, and fitting the wider bore plastic sample uptake capillary to the nebulizer. The wider bore sample capillary is supplied with the nebulizer.

For solutions which do not have a high dissolved solids content the high-vac nebulizer may be operated in the HIGH-VAC position. The high-vac narrow bore plastic sample uptake capillary is fitted to the nebulizer. The nebulizer thimble is then adjusted anti-clockwise to withdraw the platinum-iridium nebulizer capillary deep into the throat of the venturi. This results in maximum suction through the venturi which minimizes the

effects of hydrostatic head on sample uptake rate. These effects occur because of the increasing distance between the sample liquid level and the nebulizer. As the sample is consumed the liquid level drops and the nebulizer is required to pump sample from an increasing distance. The use of the high-vac nebulizer in the high-vac setting greatly reduces this effect [3].

The aerosol concentration can also be regulated by adjusting the position of the glass bead. The position of the glass bead is controlled by an adjusting screw which the operator can adjust while aspirating solutions into the flame. If the glass bead is brought closer to the nebulizer venturi a greater proportion of large droplets will be removed from the aerosol resulting in an aerosol consisting of fine droplets. A fine droplet aerosol will pass through the burner jaws with minimal deposition resulting in longer operation without burner blockage for samples with a high dissolved solids content.

By adjusting the glass bead away from the nebulizer venturi more aerosol with a greater proportion of large droplets will be passed through the burner, resulting in greater sensitivity. Since a greater number of large droplets is passing through the burner slot the analytical signal will be more noisy and clogging of the burner may result when samples with a high content of dissolved solids are aspirated. Adjustment of the glass bead to the maximum sensitivity position is therefore only recommended for samples with a low content of dissolved solids. It should be noted that on any spray chamber, samples containing thermally unstable oxidizing agents such as perchloric acid may cause the nitrous-oxide acetylene flame to flash back with a loud noise. The chance of such an event is greatly increased if the system is operated at high sensitivity. If a flash back occurs using a SpectrAA instrument the pressure relief bung at the rear of the spray chamber will be ejected, automatically triggering an interlock which safely shuts down the flame.

Moving the glass bead even further away from the venturi than the maximum sensitivity position, results in a decrease in aerosol production and therefore a decrease in sensitivity. However, the proportion of large droplets remains higher than when the bead is close to the venturi and burner clogging will be worse than if the bead is close to the nebulizer venturi.

Several experiments were performed to evaluate the performance of the Mark-VI spray chamber for samples with high dissolved solids content.

## Determination of Aluminum in Steel

The determination of refractory elements in steel presents a number of problems. The high temperature nitrous oxide-acetylene flame is generally preferred, but the nitrous oxide-acetylene burner is more susceptible to slot blockage and carbon build-up. A sample of steel dissolved by sodium pyrosulfate fusion was recommended as a difficult test for the Mark-VI spray chamber. A solution simulating such a sample was prepared to test the new spray chamber. The sample consisted of:

- 50 µg/mL aluminium
- 25 g/L iron
- 20 g/L sodium pyrosulfate
- 40 mL/L conc. hydrochloric acid

The performance of the Mark-VI spray chamber was compared to a conventional spray chamber. The instrument was established to give maximum sensitivity (highest aerosol concentration) and then the glass bead was brought closer to the nebulizer venturi, to improve the high solids capability of the system. The glass bead was adjusted closer to the venturi to give approximately 20% of the maximum sensitivity for aluminum on the Mark-VI spray chamber. The conventional chamber was adjusted to give a matching absorbance, therefore matching the aerosol concentration passing through the burner slot.

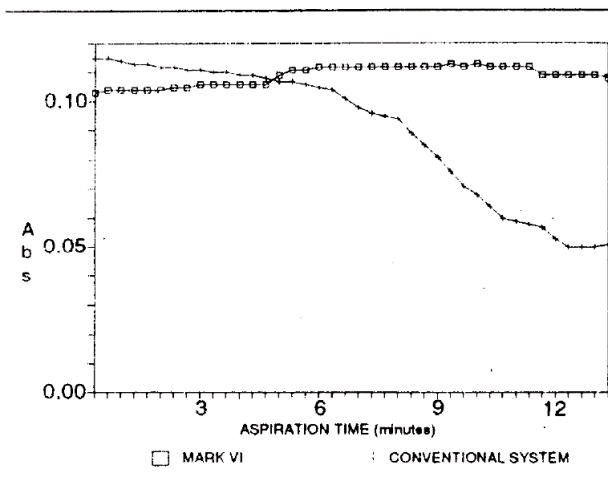


Figure 5. The measurement of aluminium in a continuously aspirated solution simulating a steel sample dissolved by sodium pyrosulfate fusion.

The results from this experiment are shown in Figure 5. It is clear from these results that the Mark-VI spray chamber showed less than a 10% variation in aluminum signal over the period of the experiment. The conventional chamber however showed a 50% deterioration in analytical signal over the same period, approximately 12 minutes.

During the experiment with the conventional spray chamber, disruption of the flame by burner blockage was observed during the first three minutes of testing and the burner was covered by carbon build-up after 11 minutes. The burner used with the Mark-VI spray chamber showed no significant signs of blockage during the experiment although the flame was more ragged after the experiment than before.

## Determination of Silicon in 5% Sodium Chloride

A 5% (wgt/vol) sodium chloride solution containing approximately 90 ppm silicon was prepared to represent a high salt sample. Typical sea water concentrations of sodium chloride are approximately 2.7% (wgt/vol). The element silicon was chosen as it requires a fuel-rich nitrous-oxide acetylene flame for maximum sensitivity. The combination of a high salt matrix with a rich flame will promote burner blockage.

The spray chambers studied were optimized to give maximum sensitivity for silicon. The glass bead of the conventional spray chamber was set closer to the nebulizer venturi to give approximately 25% of the original absorbance. The glass bead of the Mark-VI spray chamber was adjusted closer to the venturi to give a matching absorbance, and therefore an approximately equal aerosol concentration.

The results from this experiment are shown in Figure 6. The conventional spray chamber showed significant flame disruption at approximately 13 minutes, by which stage the quality of the flame and the analytical signal had begun to deteriorate. From Figure 6 it can be seen that a 50% reduction in analytical signal occurred within approximately 17 minutes.

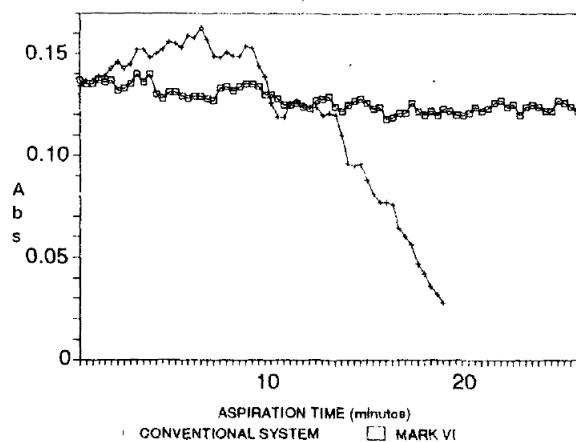


Figure 6. The measurement of silicon in a continuously aspirated solution of 5% sodium chloride.

The new Mark-VI spray chamber showed only a gradual decrease in analytical signal during the 27 minute experiment. The maximum signal deviation from the original absorbance over the time of the trial was -12%. No significant flame disruption was observed during the operation of the Mark-VI system.

### Determination of Aluminium in Soft-Drink

The determination of metals in soft-drink is complicated by the high sugar content of the samples. Deposition of the sample aerosol within the burner jaws, followed by evaporation of water, results in the build up of crystalline sugar. Thermal degradation takes place leaving a carbon residue which may block the burner, particularly when the nitrous-oxide acetylene burner is used.

In this experiment the determination of aluminium in a cola flavored soft-drink was investigated. The soft-drink sample was degassed in an ultrasonic cleaning bath and then spiked to give an aluminium concentration of approximately 50 mg/L. The instrument was set up to perform 40 replicates of 20 seconds each with continuous sample aspiration. The spray chambers studied were set to give maximum sensitivity and therefore highest aerosol concentration. Under these conditions the burner would be expected to block more rapidly than if the systems were set up for high solids operation by adjusting the nebulizer and glass bead.

The results from this experiment are detailed in Figure 7. The conventional spray chamber showed a decline in absorbance of approximately 20%, during the first 100 seconds of aspiration and a decline of 50% after approximately 230 seconds. The Mark-VI spray chamber gave a much more stable absorbance for the first 230 seconds and a decline in absorbance of 50% after approximately 340 seconds. The major cause of signal deterioration in both cases was the build-up of carbon (sugar) deposits within the burner jaws. It is evident from Figure 7 that the Mark-VI spray chamber offers a substantially longer operation time.

It should be noted that each of the experiments for burner blockage employed continuous aspiration of the sample. In practical operation rinsing between samples with distilled water is recommended. Rinsing between samples would greatly extend the period for which a spray chamber could be operated without burner blockage occurring.

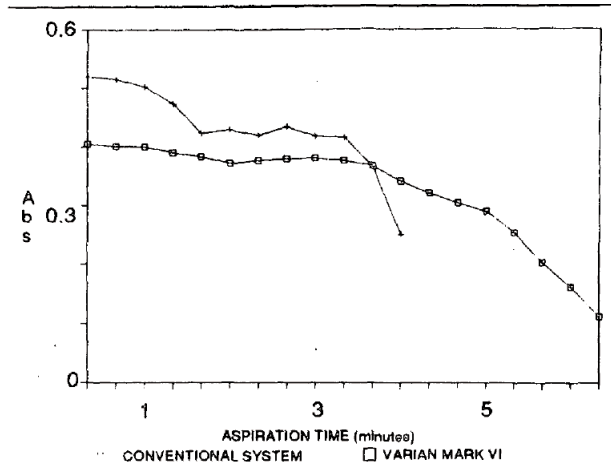


Figure 7. The measurement of aluminium in a continuously aspirated soft-drink sample.

### Interferences

There are many types of interferences that occur in flame AAS that can be minimized by effective spray chamber design. Browner et. al [4] have studied the relationship between aerosol droplet size and the atomization process. Further work [5] investigated the effect of droplet size on interferences in flame AAS. A number of interferences were investigated including the effect of phosphate interference on calcium and aluminium interference on magnesium absorbance.

Ham and Willis [6] found that many volatilization interferences could be minimized by the removal of large droplets from the aerosol. This will result in a reduction in sensitivity. The interference of aluminium on-magnesium absorbance, due to the formation of magnesium aluminate, can be minimized by reducing the average sample aerosol droplet diameter.

The performance of the Mark-VI spray chamber in minimizing the effects of such interferences has been investigated. The adjustable impact bead and the dual-head mixing paddle were used in combination to reduce aerosol droplet size and therefore minimize volatilization interferences. In the evaluation of interference effects, the flame stoichiometry and the height of the light path above the burner are important. In this work such variables were adjusted initially to give maximum sensitivity. The glass bead was then adjusted closer to the nebulizer venturi to reduce the sensitivity to the desired level.

The performance of the Mark-VI spray chamber was evaluated for the interference effect of aluminium on magnesium absorbance. A solution of 0.5 mg/L magnesium containing 50 mg/L aluminium was prepared. The magnesium absorbance of this solution was compared to a 0.5 mg/L magnesium solution not containing the aluminium interferent at various settings of the glass bead. The Mark-VI spray chamber was compared to a conventional spray chamber system.

The results of this experiment are shown in Figure 8. The bar graph shows the suppression of the magnesium signal due to the interferent (vertical axis) versus the reduction in magnesium absorbance (horizontal axis) due to the adjustment of the glass bead closer to the venturi. As the glass bead is wound in closer to the nebulizer it acts to break up the aerosol into smaller droplets therefore reducing the effect of volatilization interferences.

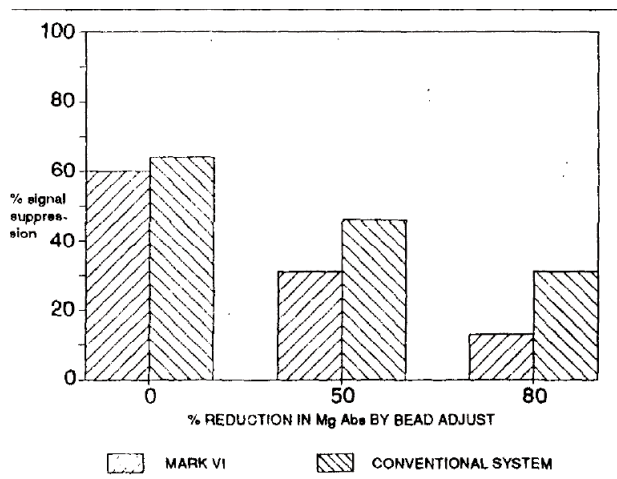


Figure 8. The effect of aluminium interferent on magnesium absorbance.

The Mark-VI spray chamber showed a slightly higher magnesium absorbance than the conventional system at maximum sensitivity, indicating that the smaller aerosol droplet diameter produced, reduced the effect of the aluminium interferent. Greater benefit was realized when the glass impact bead was brought closer to the nebulizer. After reducing the analytical signal by 80% using the glass bead the Mark-VI showed only a 13% reduction in absorbance due to interference while the conventional system showed a suppression of 32%.

In a second experiment the enhancement effect of sodium sulfate interferent on molybdenum absorbance was investigated [7]. The enhancement effect of sodium sulfate on molybdenum absorbance using the nitrous oxide-acetylene flame has been previously reported [8]. For the determination of molybdenum in samples containing sodium sulfate such as soil, standard additions may be necessary to overcome the enhancement effect when a conventional spray chamber is used.

Solutions of 10, 25 and 50 µg/mL molybdenum were prepared with and without sodium sulfate interferent. The enhancement effect was then investigated at the different molybdenum concentrations using the Mark-VI and a conventional spray chamber.

The results of this experiment are shown in Figure 9. It can be seen that the interference effect is severe at all molybdenum concentrations using the conventional spray chamber. The molybdenum absorbance obtained using the Mark-VI spray chamber is affected only minimally by the presence of the interferent.

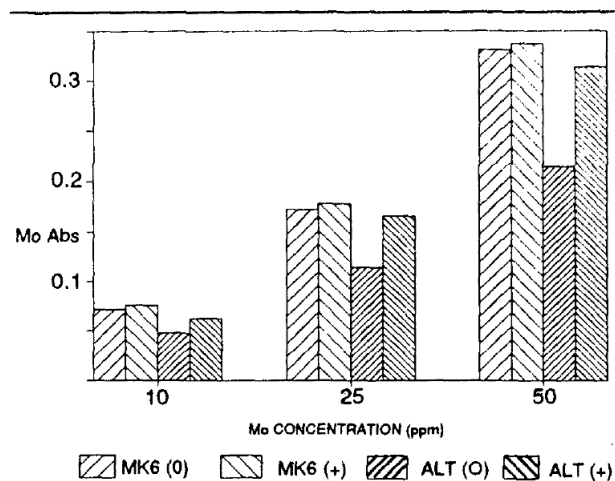


Figure 9. The effect of sodium sulfate interferent on molybdenum absorbance (7). (0) = no interferent present; (+) = with interferent present; ALT = alternative conventional spray chamber; MK6 = Agilent Mark VI spray chamber.

## Characteristic Concentration and Detection Limits

In designing a spray chamber for flame AAS a balance must be achieved between performance for difficult samples and sensitivity. It has been shown that the production of a large proportion of small aerosol droplets provides the best performance in the presence of interferents and for difficult samples. In achieving this performance poorer sensitivity may be observed as large aerosol droplets are removed from the aerosol.

For most elements the dominant source of noise at normal working levels is analyte flicker noise arising from fluctuations in the aerosol produced by the spray chamber. The production of a uniform, small droplet aerosol reduces analyte flicker noise and produces a more stable signal.

Lower detection limits can generally be achieved if the sensitivity for an element can be increased. The Mark-VI spray-chamber can be operated with the mixing paddle in place for best performance for high dissolved solids samples and for general use, and with the mixing paddle out for maximum sensitivity and detection limits. If the Mark-VI spraychamber is operated with a nitrous oxide-acetylene flame without the mixing paddle, the benefits of reduced carbon build-up and a more stable signal may not be seen.

The performance of the Mark-VI spray chamber was evaluated for a number of elements in terms of characteristic concentration, detection limit and analyte signal noise. For each of these tests the Mark-VI spraychamber was operated with the mixing paddle in place. This gives an indication of the performance that can be achieved using the Mark-VI spraychamber with the benefits of the mixing paddle. Better characteristic concentrations and detection limits could be expected with the mixing paddle removed. The performance of the new spray chamber was compared to a conventional system. Tables 2 (a) and (b) detail the results of this comparison for detection limit and characteristic concentration.

The detection limits given in Table 2 (b) are based on twice the standard deviation of 10 replicate absorbance measurements of a very low concentration standard solution of the element. Note that IUPAC recommendations state that either two or three standard deviations may be used in the calculation of detection limits but the number used must be stated end three is the recommended figure. Two standard deviations was used in this work so that detection limits could be compared to values previously published in the literature.

Table 2. Spray Chamber Comparison— Characteristic Concentration and Detection Limits

Element	Mark VI	Conventional
Cu	0.04	0.03
Pb	0.10	0.08
Al	0.73	0.74

Element	Mark VI	Conventional
Cu	0.003	0.004
Pb	0.011	0.017
Al	0.03	0.035 (9)
Al	0.05	0.04
Ca	0.001	0.006
V	0.085	0.045
V	0.074	0.073

It can be seen from the results in Table 2 (a) that the characteristic concentration figures obtained from the Mark-VI spray chamber are competitive with conventional systems even with the mixing paddle in place. A degree of variability can be expected in the detection limit figures. The ability to achieve the best results will rely on operator ability to fully optimize the system. The detection limit results for aluminium [9] and vanadium are presented in duplicate in Table 2 (b) to indicate the degree of variability that can be expected from such figures.

## Analyte Signal Noise and Stability

Several experiments were conducted to evaluate the analyte signal noise observed with different spray chamber systems for a number of elements. The same standard solutions were examined on different spray chambers using the same spectrometer, hollow cathode lamp and burner. Figure 10 shows a comparison of the signal obtained for aluminium on the Mark-VI spray chamber and a conventional system. Note that the absorbances are approximately the same indicating that the aerosol concentration passing through the burner is similar. It can be seen that a more stable and less noisy signal is obtained using the Mark-VI spray chamber compared to the conventional system. This reduction in signal noise end drift is due to the production of a more uniform, small droplet aerosol.



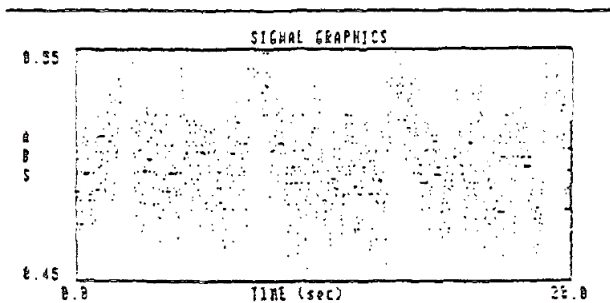


Figure 10(a)

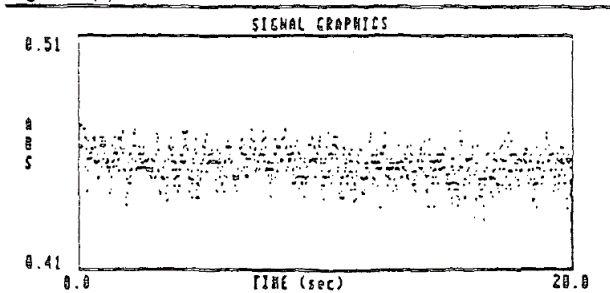


Figure 10(b)

Figure 10. Comparison of aluminum signal noise using (a) conventional spray chamber, and (b) the Agilent Mark VI spray chamber.

An example [10] of the signal stability of the Mark-VI spray chamber is shown in Figure 11 using a fuel rich air-acetylene flame for chromium. A solution of 10  $\mu\text{g}/\text{mL}$  chromium was prepared in 5% (wgt/vol) sugar solution.

The sensitivity of the system was then reduced to 30% of the maximum by adjusting the glass bead closer to the nebulizer venturi. The combination of the glass bead and the mixing paddle produce a fine aerosol and a stable analytical signal.

Thirty-one samples of the solution were aspirated with 5 seconds delay time and ten integrations of 5 seconds each. Each of 31 samples was therefore aspirated for 55 seconds each. Figure 11 (a) shows the first fourteen minutes of operation, Figure 11 (b) shows the second fourteen minutes of operation. It can be seen from Figure 11 that the signal remained completely stable over 28 minutes. The analytical signal did not deviate by more than 0.5% from the initial absorbance over this time.

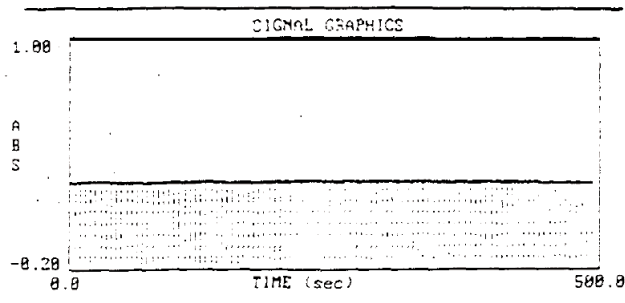


Figure 11(a)

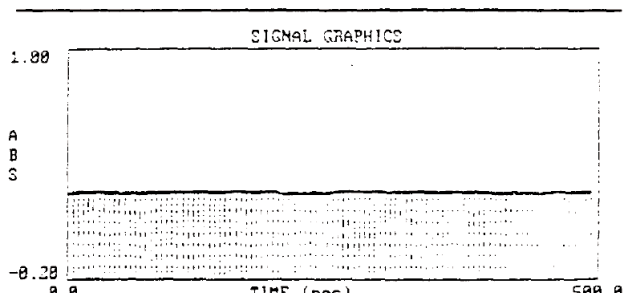


Figure 11(b)

Figure 11. Long term signal stability for chromium in 5% sugar solution, (a) 0–500 seconds, (b) 500–1000 seconds.

## Conclusion

The Agilent Mark-VI spray chamber has been evaluated in terms of drainage, carbon build-up, burner blockage and Interferences, characteristic concentration and detection limits and signal stability.

Effective drainage is achieved by a steeply angled chamber floor, a ceramic nebulizer face-plate and a recessed wide bore drainage tube. The production of a smaller drop size aerosol reduces burner blockage and reduces signal noise and improves long term signal stability. The mixing paddle promotes better fuel and oxidant mixing resulting in reduced carbon build-up. Characteristic concentrations and detection limits are competitive with alternative systems even with the mixing paddle in place and volatilization interferences are substantially reduced.

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