Use of Drierite Trap to Extend the Lifetimeof Vapor Generation Absorption Cell

Application Note

Atomic Absorption

Author

R. Le Houillier

Centre de Recherches minerales

Ministere de L'Energie et des

Ressources

Gouvernement du Quebec

Canada

Introduction

Vapor generation techniques, which employ continuous flow technology developed a few years ago by Agilent [1] and reported by others [2,3] are useful and sensitive for the hydride determinations of mercury, arsenic, antimony, selenium, tellurium, bismuth and tin.

Centre de Recherches minerales is involved in the determination of arsenic in various sample types such as water, rocks and sediments. More than 10,000 samples are analyzed each year. An automated vapor generation accessory for atomic absorption analysis has been bought to cater for this requirement. For our purposes, however, the lifetime of the quartz absorption cell proved to be rather short and thus increased the cost of our analyses. Devitrification and cracking of the absorption cell were the major factors. A successful attempt to overcome this problem is reported.

Instrumentation and Typical Parameters

Spectrometer	Agilent AA-475
Sample presentation	Agilent PSC-55 for automated analysis
Source lamp	Agilent hollow cathode arsenic lamp
Lamp current	10 mA
Wavelength	193.7 nm
SBW	0.2 nm
Integration period	3 seconds
Vapor generation accessory	Agilent VGA-76
Acid channel	6 M HCl
Flow rate	1 mL per minute
$NaBH_4$ channel	0.6% $NaBH_4$ 0.5% NaOH 10% w/v KI
Flow rate	1 mL per minute
As^V standard solution	7 M HCl
Flow rate	7 mL per minute

The principle of operation of the automated vapor generation accessory has been adequately described by Brodie et al [1]. Figure 1 shows a schematic diagram of this accessory. Arsine produced is transported by nitrogen from the gas/liquid separator through the transfer tube and into the quartz absorption cell. If too much alkaline substance, such as KI, is carried over with the arsine gas and nitrogen into the absorption cell, a significant decrease of cell life is observed [4]. To cure this problem, a drierite trap was inserted between the absorption cell and the output of the gas/liquid separator. Figure 2 shows a schematic diagram of the glass trap containing eight mesh drierite. The trap must be installed vertically for maximum efficiency. Aging of the drierite is detected by its color changing from blue to pink. The operator will visualize a change in color when two-thirds of the drierite has been humidified by the alkaline substance. The drierite must be changed at this point.

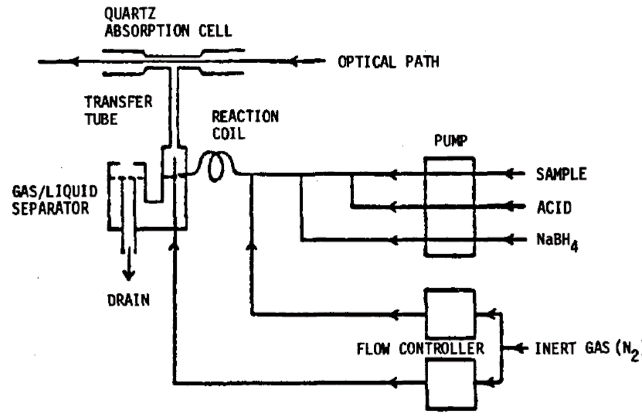

Figure 1. Schematic diagram of vapor generation accessory.

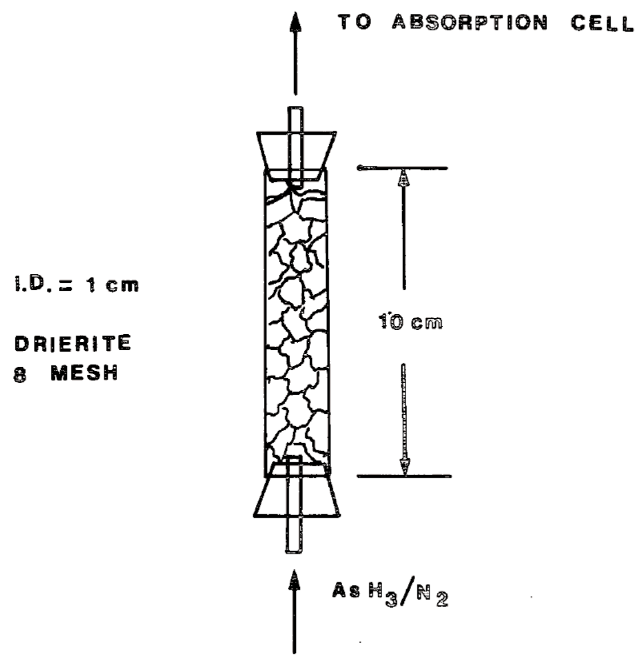

Figure 2. Glass trap with eight mesh drierite.

Results and Discussions

In routine analyses of arsenic in sediments and rocks samples, devitrification of the quartz cell and its breakage have been observed after less than a week of operation. Three quartz cells were replaced within approximately a month of operation. To solve this problem, a drierite trap was experimented with. The alkaline humidity carried over by the arsine and nitrogen gases was absorbed on the drierite trap. Without the drierite trap, devitrification of the quartz cell occurs in the region of the change in cross section of the cell. This devitrification becomes evident after about seven hours of continuous operation, which means that three litres of sample solution (400 ng/mL of As^V in 7M HCl) have been pumped into the system. With the drierite trap and after pumping a total volume of 23 L of the sample solution, no deterioration of the quartz cell was seen. From such observations it was decided to use the drierite trap and to verify its influence on the conditioning time of the system.

Figure 3 shows the profile of the relative absorbance against time without the drierite trap. It takes about 6 minutes to obtain maximum absorbance with a solution of 10 ng/ml of As^V. After the maximum absorbance is reached, a blank is pumped into the system to determine the time required to decrease to zero absorbance (approximately two minutes). The same time again is required to reach the maximum absorbance. This curve is obtained without conditioning the system.

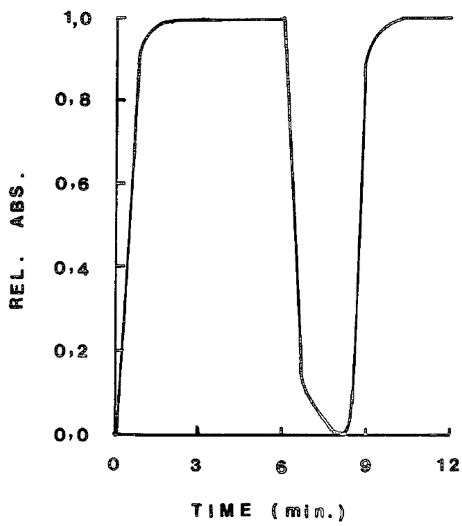

Figure 3.

The same experiment was conducted with the drierite trap and the results are shown in Figure 4. Forty minutes were required to obtain maximum absorbance, which is almost seven times the delay obtained without drierite. To reach zero absorbance with an analytical blank required eight minutes and about another eight minutes to reach the maximum absorbance. To condition the whole system in a shorter time when drierite trap is used, a 400 ng/mL solution of As^V was pumped into it. Figure 5 shows the appearance of the absorbance obtained in such conditions. The maximum absorbance was reached in approximately ten minutes. To reach zero absorbance required about five minutes and the same time to reach its maximum value. When the whole system was conditioned in this manner and the absorbance was brought back to zero, a 10 ng/mL solution of As^V was pumped into the system. The relative absorbance then obtained is shown in Figure 6. After about 1.5 minutes, the maximum absorbance was reached. It then requires about 1 minute to decrease it to zero with a blank solution. This indicated that proper conditioning of the whole system, when a drierite trap is used, does not significantly affect the number of routine analyses conducted in a given time.

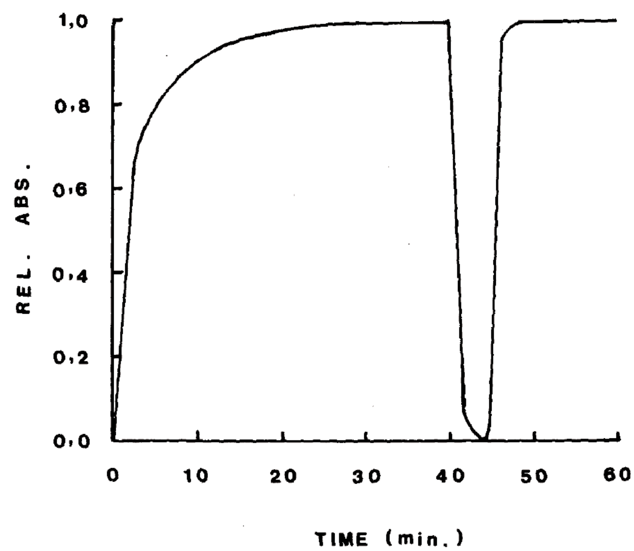

Figure 4.

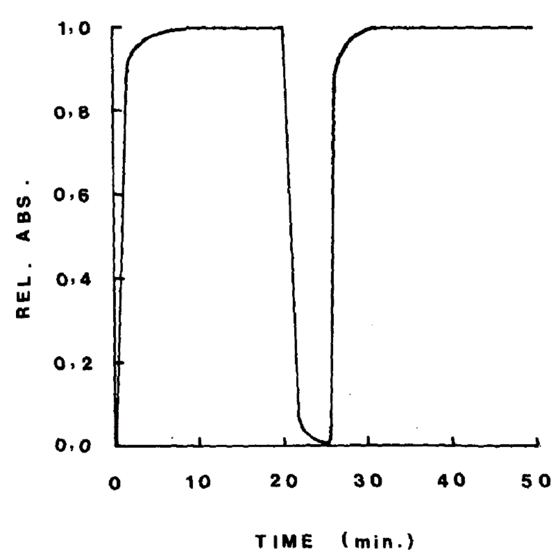

Figure 5.

The system can then be conditioned with the 400 ng/mL of As^V, and if properly purged with the analytical blank, no significant memory effects are observed. However, the use of the drierite trap may be responsible for absorbance loss in the order of 20% at the level of 10 ng/mL. Nevertheless, with increasing arsenic concentration, the absorbance loss decreases significantly to less than 10%.

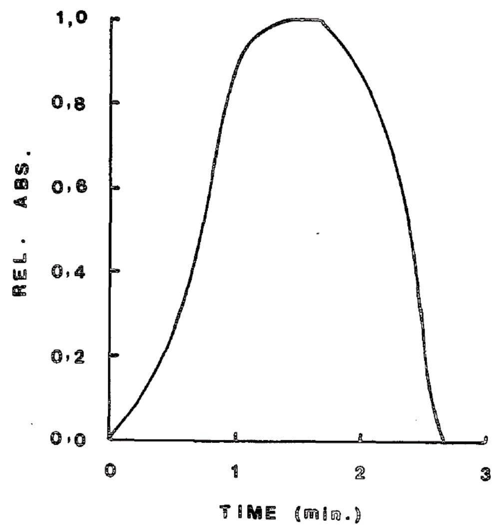

Figure 6.

Conclusion

The use of a drierite trap, when a significant number of samples are analyzed, provides a good means of protecting the absorption cell and extending its lifetime. Furthermore, the results obtained with a drierite trap are as good as those formerly produced without it.

Acknowledgment

The author wishes to thank Mr Guy Vallieres for his experimental contribution and the Centre de Recherches minerales for permission to publish this paper.

References

1. Varian Instruments at Work No AA-38 March 1983.
2. Yongming Liu, Benling Gong, Juhua Sun and Tiezheng Lin, Bunseki Kagaku, 32, E9 1983.
3. W. B. Robins and J.A. Caruso, Analyst, 104, 35 1979.
4. VGA-76 Operation Manual (Publication No 85-100577-00). Varian Techtron Pty. Limited, Mulgrave, Victoria, Australia.

For More Information

For more information on our products and services, visit our Web site at www.agilent.com/chem

www.agilent.com/chem

Printed in the USA
November 1, 2010
AA065

Agilent Technologies