Performance Evaluation of a Cyclonic Spray Chamber for ICP-MS

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ABSTRACT

The performance characteristics of a cyclonic spray chamber have been evaluated for ICP-MS. This new spray chamber provides an increase in analyte sensitivity without any increases in the levels of oxides and doubly-charged background ions.

INTRODUCTION

The conventional Scott-type spray chamber used with pneumatic nebulization for inductively coupled plasma (ICP) spectroscopy has an efficiency of transporting aerosol into the ICP torch of about 1-2%. More than 98% of the sample delivered to the nebulizer never makes it into the torch. Alternative sample introduction systems have been developed to increase the efficiency of introducing sample vapor into the ICP, including the ultrasonic nebulizer and the direct injection nebulizer, often providing improved sensitivity and detection limits. However, these devices are more costly than conventional pneumatic nebulizer and spray chamber combinations. Because of the complexity of their operation and their high sensitivity, contamination can become significant, and the ability to handle complex sample matrices can be limited. As a result, the application of these alternative nebulization systems is not universal.

A new spray chamber designed for use with conventional concentric nebulizers has recently been made available as an alternative to the conventional Scott design. The novel design of the spray chamber results in a high aerosol transport efficiency into the ICP, while maintaining low cost and simple operation. The application of this new spray chamber to ICP-mass spectrometry (MS) has been evaluated and is described in this paper.

EXPERIMENTAL

The cyclonic spray chamber (Glass Expansion Pty., Hawthorn, Australia) evaluated is shown in Figure 1. The system is designed for tangential introduction of the aerosol generated with a conventional concentric nebulizer, and operates based on centrifugal action. Aerosol droplets are discriminated according to size by means of the resulting vortex gas flow about the conical axis within the chamber. Smaller aerosol droplets are carried with the gas stream up into the ICP, while larger droplets impinge on the walls and drain down for collection to waste.

All measurements were made on the Perkin-Elmer® Sciex ELAN™ 5000 ICP-MS (Perkin-Elmer SCIEX, Thornhill, Canada), using the standard operating conditions shown in Table I. For comparison, two different concentric nebulizers, a type-C3 and a type-K, were tested (J.E. Meinhard and Associates, Santa Ana, CA). The cyclonic spray chamber was compared with the Ryton Scott-design spray chamber of the ELAN, fitted with both the standard crossflow nebulizer and the type-C3 nebulizer. All nebulizers were fed using a peristaltic pump to maintain a constant liquid delivery rate.

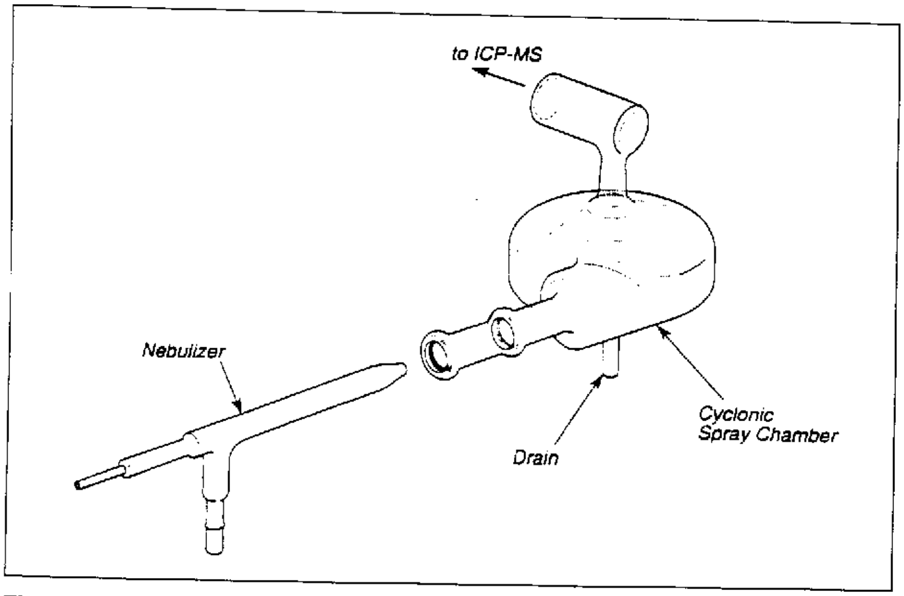

Fig. 1. *Diagram of Cyclonic spray chamber.*

TABLE I
ELAN 5000 Instrument Settings

Parameter	Setting
RF power	1170 Watts
Plasma gas flow	15 L/min
Auxiliary gas flow	0.69 L/min
Nebulizer gas flow	0.84 L/min
Dwell time	200 ms
Sweeps/replicate	5
Total integration time per isotope	1000 ms
Pump speed	25

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Nebulizer argon flow rate was optimized for each individual nebulizer. For a given nebulizer, the optimum argon nebulizer flow rate for the two spray chambers was the same. Standard solutions were prepared using PE Pure multielement calibration stock solutions (The Perkin-Elmer Corporation, Norwalk, CT), and high-purity nitric acid (SeaStar Inc., Seattle, WA).

RESULTS AND DISCUSSION

Effect of Positioning

The cyclonic spray chamber mounts to the base of the torch of the ELAN as a direct replacement for the standard spray chamber. The angle of tilt of the spray chamber was found to have little effect on sensitivity. However, a small tilt appeared to improve short-term precision slightly, presumably due to improved draining (see Figures 2 and 3). All further measurements were performed at a 17-degree tilt.

The position of the nebulizer in the spray chamber has a small effect on sensitivity and precision. The results in Figure 4 show that both sensitivity and precision can be optimized by displacing the nebulizer approximately 3 mm from the full-in, or stop position. All further measurements were made using a 3-mm nebulizer displacement.

Evaluation of Sensitivity and Precision

The sensitivity and precision of the cyclonic spray chamber used with the concentric nebulizers was compared to that obtained using the standard Ryton Scott spray chamber. For the comparison, the standard Ryton Scott-type spray chamber was fitted with the standard crossflow nebulizer as well as both types of concentric nebulizers. The cyclonic spray chamber was fitted with the two types of concentric nebulizers. Included in the study were three individual cyclonic spray chambers, two individual type-C concentric nebulizers, a single type-K nebulizer, and a single Ryton Scott-type spray chamber.

Compared to the standard Scott system, an average sensitivity enhancement of over 50% was observed for the cyclonic spray chamber system, regardless of the nebulizer, as is shown in Figure 5. Overall, this translated into a proportional improvement in detection limit for most elements tested. However, precision obtained using the cyclonic spray chamber appears to be slightly poorer, although still well under 2% (RSD). This is shown in Figure 6. It was also observed that the use of a small amount of surfactant (e.g., <0.1% Triton X-100) improved precision using the cyclonic nebulizer. This is of interest so long as the surfactant used does not contaminate the sample with the analyte(s) of interest.

Background Spectral Characteristics

Even though the efficiency of aerosol transport to the plasma is higher with the cyclonic spray chamber, the oxide level and the doubly-charged ions obtained were

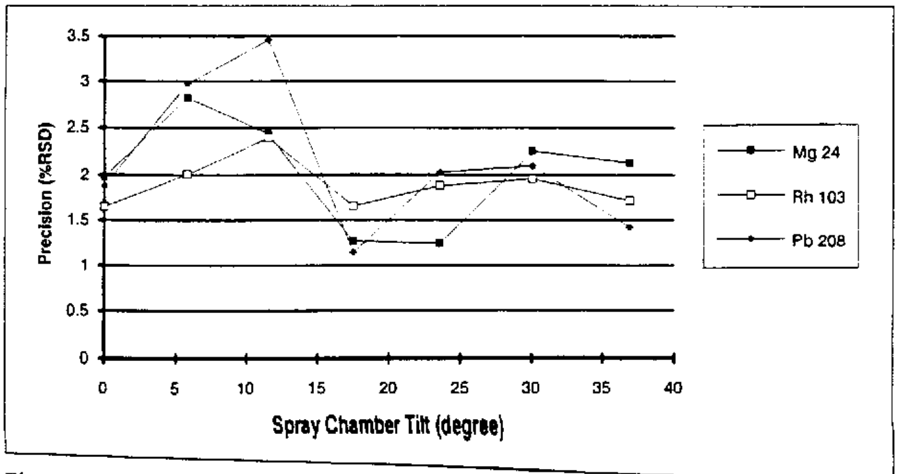

Fig. 2. *Effect of spray chamber tile angle on precision.*

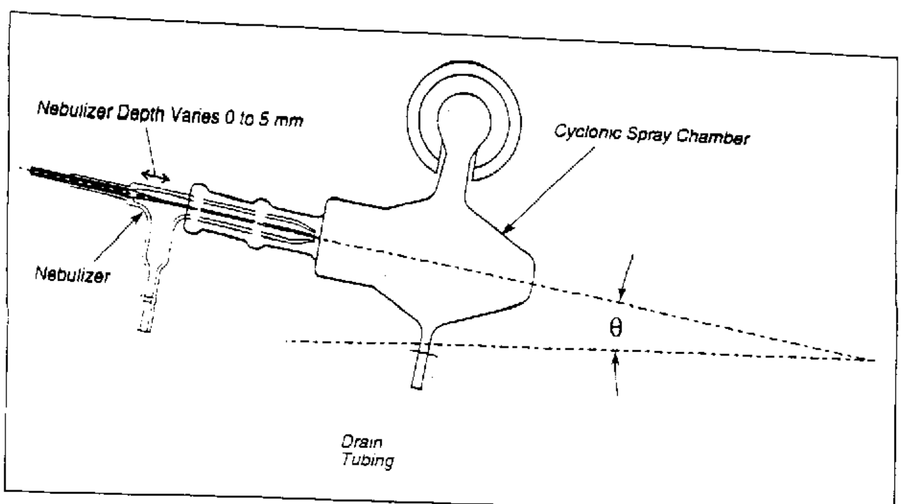

Fig. 3. *Mounting configuration for cyclonic spray chamber and concentric nebulizer.*

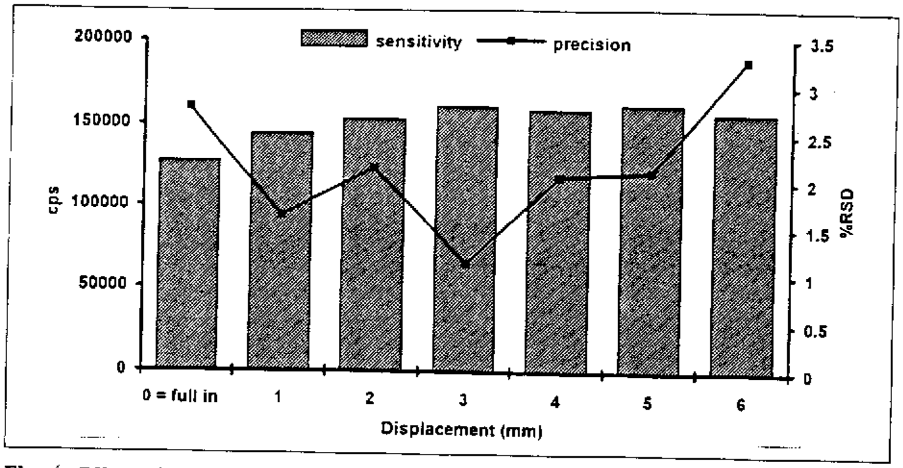

Fig. 4. *Effect of nebulizer position on sensitivity and precision.*

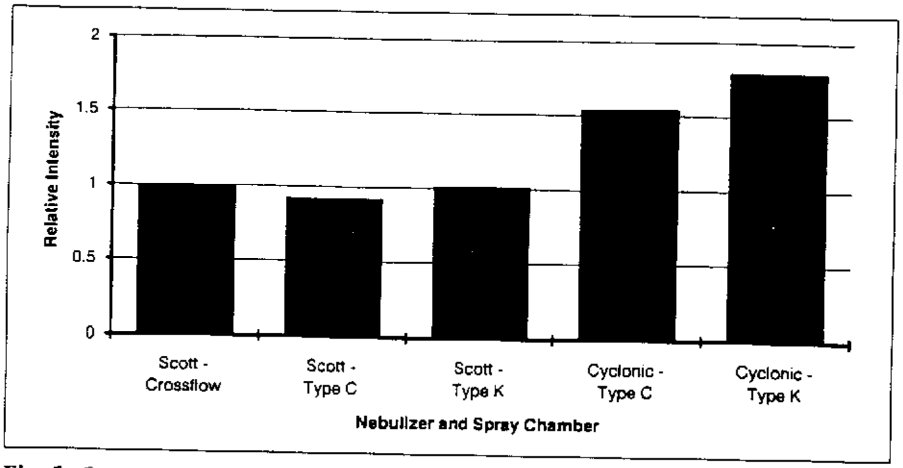

Fig. 5. *Comparison of relative sensitivity of Rb for different nebulizer and spray chamber combinations.*

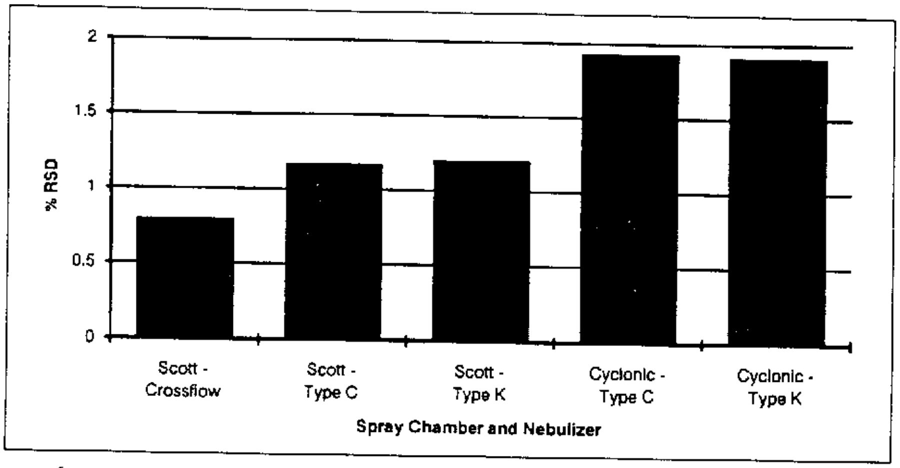

Fig. 6. *Comparison of precision for 10 μg/L Rb for different nebulizer and spray chamber combinations.*

found to be nearly the same as those obtained with the conventional spray chamber and the crossflow nebulizer. Oxide and the doubly-charged ion levels for both sample introduction systems are compared in Table II.

TABLE II
Oxide Levels and Doubly-Charged Ions for the Sampling Systems Tested

	Scott Type spray chamber/ crossflow neb. (%)	Cyclonic spray chamber/ concentric neb. (%)
BaO	0.11	0.15
Ba^{++}	1.20	1.35
CeO	1.60	2.00
Ce^{++}	0.55	0.62

Stability

Because of the increase in efficiency of aerosol generation, the long-term stability achieved using the cyclonic spray chamber was investigated. Long-term stability for an aqueous 1% nitric acid solution and a solution containing 0.1% Ca was measured over a period of over 100 minutes. The results of this evaluation are presented in Figures 7 and 8.

The stability observed using the cyclonic spray chamber was as good as that typically achieved using the standard Ryton Scott spray chamber and crossflow nebulizer. For the group of elements studied, varying widely in mass, stability (based on % RSD of all measurements) ranged from about 0.7% to 1.6% both in the nitric acid matrix as well as in the 0.1% Ca matrix. It is important to note that all of these stability results were obtained without the use of internal standardization.

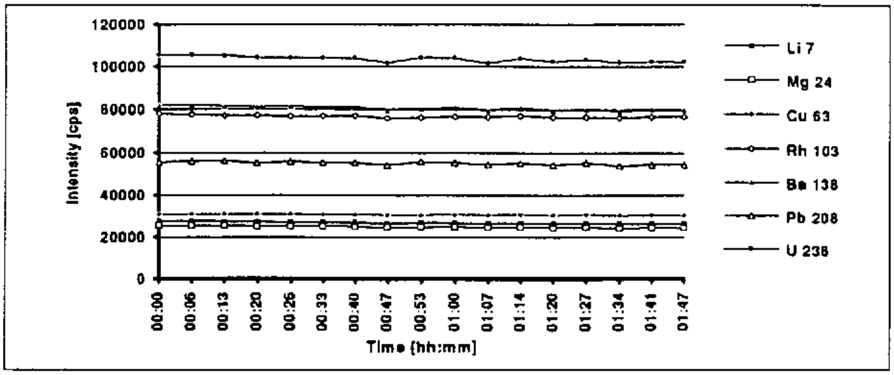

Fig. 7. *Long-term stability for cyclonic spray chamber using type-C concentric nebulizer aspirating a 1% nitric acid solution. Pure nitric acid solution spiked with analytes at 10 µg/L.*

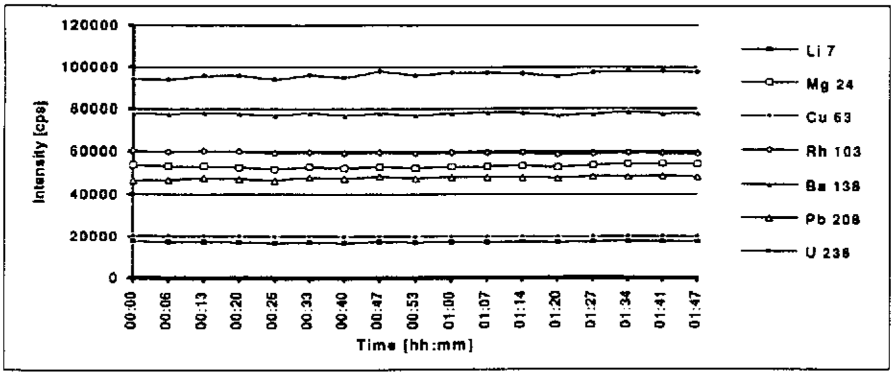

Fig. 8. *Long-term stability for cyclonic spray chamber using type-C concentric nebulizer aspirating a 0.1% Ca solution. Calcium matrix solution spiked with analytes at 10 µg/L - note that the matrix solution was contaminated with some elements.*

CONCLUSION

The cyclonic spray chamber is a simple, low-cost device and can be easily installed and optimized on the ELAN 5000. The increased sensitivity and attendant improvement in detection limits indicate that the system has a high sample transport efficiency. The fact that the level of oxides and doubly-charged species does not change, however, might suggest that aerosol vapor content and/or particle-size distribution may also be important factors contributing to the improved performance observed. Physical draining of waste solution may be a potential problem, but only in those cases where a precision of better than about 2% RSD is absolutely required. The cyclonic spray chamber has been shown to be a useful accessory for applications that do not require resistance to hydrofluoric acid.

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