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### **Investigation of Transport Efficiency of Pneumatic Nebulization for Dissolved Solids in Flame and Furnace Atomic Absorption Spectrometry**

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INVESTIGATION OF TRANSPORT EFFICIENCY OF PNEUMATIC  
NEBULIZATION FOR DISSOLVED SOLIDS IN FLAME AND  
FURNACE ATOMIC ABSORPTION SPECTROMETRY

Keywords: Sample Introduction, Dissolved Solids,  
Pneumatic Nebulization, Impaction Bead, Flame, Furnace,  
Atomic Absorption Spectrometry.

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ABSTRACT

The use of a pneumatic nebulization with impaction bead system for sample introduction of dissolved solids in flame and furnace atomic absorption spectrometry is investigated. The distance between the aerosol emerging from the capillary tip and the impaction bead is critical for dissolved solids in terms of nebulization efficiency, imprecision, absorbance signal, and interferences. The experimental results are considered to be dependent on the particle size generated by the pneumatic nebulization with impaction bead system.

## 1. INTRODUCTION

Sample introduction techniques for atomic spectrometry have recently been reviewed by Browner and Boorn<sup>1,2</sup>. They noted that pneumatic nebulization for sample introduction is the most widely used, readily available, and accepted approach in the majority of situations and is unlikely to change in the near future despite some reservations over poor transport efficiency. When the aerosol emerges from the nebulizer capillary tip, several aerosol modifying devices are used, such as spray chambers, impaction beads, paddles, etc., which are placed in the aerosol path and produce secondary aerosols which are then carried to the atomizer. In this paper, we report the results on the use of a pneumatic nebulization with an impaction bead system for sample introduction to a flame and furnace with particular interest on the effect of increased concentration of dissolved solids. Cresser and co-workers<sup>3,4</sup> have investigated the effect of impactors on aerosol size distributions produced by pneumatic nebulizers.

## 2. EXPERIMENTAL

A schematic diagram of the pneumatic nebulization system for a premixed burner is shown in FIG. 1. The sample uptake is through the capillary tube and mixes with the oxidant as it emerges from the capillary. A pressure drop exists at this point and coupled with the high velocity produces primary droplets (venturi effect). These primary droplets collide with an impaction bead to produce secondary and finer droplets which pass

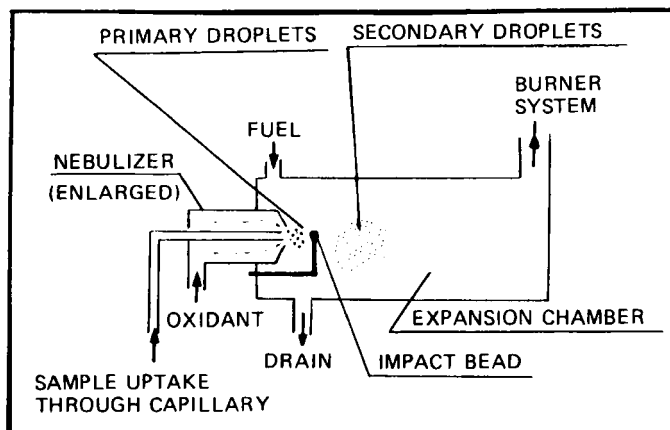


FIG. 1. Schematic diagram of pneumatic nebulization system for a premixed burner.

through an expansion chamber to the burner system. The expansion chamber allows thorough mixing of fuel, oxidant, and aerosol. An Allied Instrumentation Laboratory Fastac II pneumatic nebulization aerosol deposition system is used for sample introduction to the furnace. This has been described previously<sup>5</sup> and is similar to that shown in FIG. 1, except that the secondary droplets are pulled by vacuum through a jet which is directed at the entrance port of the graphite furnace. For the furnace work, an Allied Instrumentation Laboratory 655 Temperature Controlled Furnace is used as described previously<sup>6</sup>. An Allied Instrumentation Laboratory 457 spectrometer and air-acetylene flame is used. All experimental and instrumentation parameters are optimized as recommended by the manufacturer. Reagents were of the highest

purity available (J. T. Baker Chem. Co., Phillipsburg, NJ), typically  $1000 \mu\text{g ml}^{-1}$  for copper and 25% for sodium chloride concentration, and diluted with high purity deionized water as required. For the flame work,  $3.0 \mu\text{g ml}^{-1}$  copper and for the furnace work  $10 \mu\text{g l}^{-1}$  copper is used. The distance from the internal nebulization capillary tip and impaction bead surface is measured using cardboard of a precise thickness. To ensure reproducible distances, the cardboard fits tightly into the distance and was previously measured using digi-matic electronic calipers (Rutland Tool & Supply Co., City of Industry, CA).

### 3. RESULTS AND DISCUSSION

#### 3.1 Effect of Increasing Dissolved Solids on Nebulization

##### Efficiency

The nebulization or transport efficiency can be defined as

$$\text{Nebulization} = \frac{\text{Uptake Rate} - \text{Drainage}}{\text{Uptake Rate}} \times 100 - (1)$$

Efficiency

Uptake Rate

and is an often quoted figure for evaluating the performance of nebulizers and spray chambers, although concern in using this parameter for comparing different systems has been expressed<sup>1</sup>. In this work, nebulization efficiency is used as the effect of dissolved solids on a particular pneumatic nebulization system is being evaluated. The effect of uptake rate on absorbance signal from a  $3.0 \mu\text{g ml}^{-1}$  copper solution in an air-acetylene flame is shown in FIG. 2 for a fixed distance of 1.09 mm between the emerging aerosol and the impaction bead. The nebulizer capillary

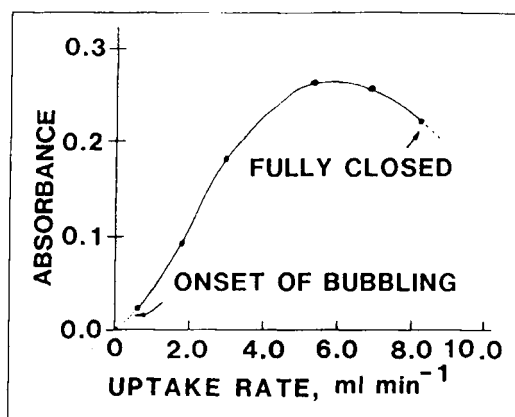


FIG. 2. The effect of uptake rate on absorbance signal from a 3.0  $\mu\text{g ml}^{-1}$  solution of copper.

position is adjusted using the screw thread from just after the onset of bubbling to fully closed. A maximum absorbance signal for copper is obtained at an uptake rate of 5.3  $\text{ml min}^{-1}$  and this rate is used throughout this work. The effect of increased dissolved solids on the nebulization efficiency at various distances from the emerging aerosol and impaction bead is shown in FIG. 3. The greater the distance between the emerging aerosol and impaction bead, the greater the nebulization efficiency. The smaller the distance between the emerging aerosol and impaction bead, the more linear the relationship between dissolved solids and nebulization efficiency. O'Grady *et al*<sup>3</sup> found that a more linear relationship was obtained for a magnesium chloride calibration curve at a distance of 1.8 mm than 8.0 mm.

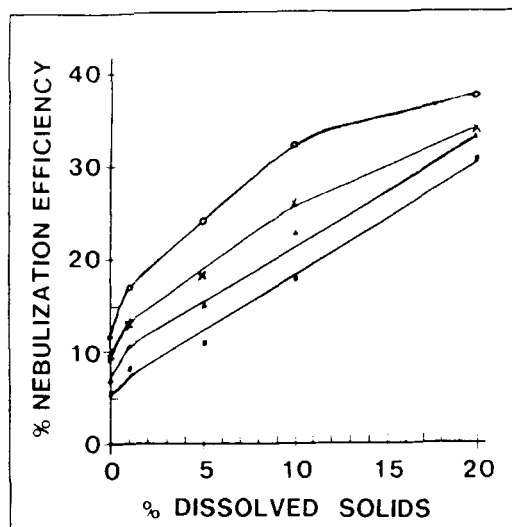


FIG. 3. The effect of increasing concentration of dissolved solids on nebulization efficiency at various distances of the emerging aerosol and impaction bead: o - no bead, x - 4.06 mm, ▲ - 2.93 mm, and ■ - 1.09 mm.

### 3.2 Effect of Increasing Dissolved Solids on Imprecision and Absorbance

As shown previously in FIG. 3, the nebulization efficiency is increased with increasing concentration of dissolved solids and the larger the distance between the emerging aerosol and impaction bead. However, increasing dissolved solids and the distance from emerging aerosol to impaction bead will increase the imprecision as shown in FIG. 4 for a 1.09 mm distance and with no impaction bead. The imprecision of aqueous solutions is typically less than

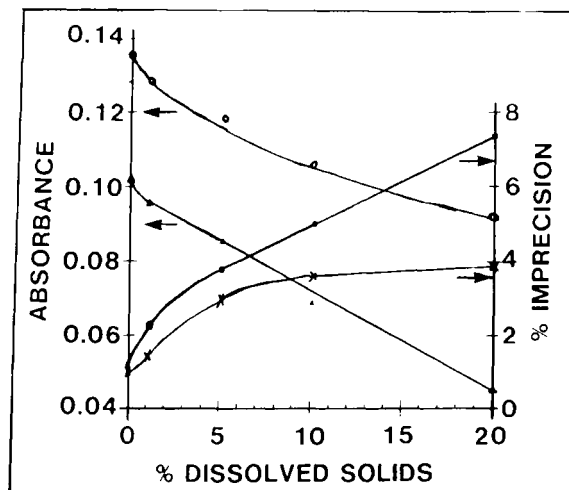


FIG. 4. The effect of increasing concentration of dissolved solids on imprecision: ■ - no bead and x - 1.09 mm and absorbance signal: o - no bead and ▲ - 1.09 mm.

1% and increases to approximately 4% with 20% dissolved solids at a 1.09 mm distance. When the system contains no impaction bead the imprecision increases to over 7% at 20% dissolved solids. Therefore, high nebulization efficiency leads to an increase in imprecision and has been attributed to allowing large droplets to reach the atomizer<sup>2,3</sup>. The greater the distance between the emerging aerosol and impaction bead, the larger the droplets reaching the atomizer and subsequently the higher the imprecision. Increasing dissolved solids will decrease the signal and is shown in FIG. 4 for a 1.09 mm distance and no impaction bead. The absorbance is approximately 25% higher with no impaction bead



compared to an impaction bead distance of 1.09 mm for aqueous solutions. Increasing the concentration of dissolved solids will decrease the absorbance signal, and at 20% dissolved solids, there is a 31% decrease in signal with no impaction bead and a 56% decrease in signal with a 1.09 mm distance. The magnitude of the depression is dependent on the distance of the emerging aerosol and impaction bead, with the closer the distance of the bead the more severe the depression. The critical positioning of the impaction bead may lead to differences in magnitude of interferences and may account for the lack of transferability of interferences between different laboratories using identical instrumentation.

### 3.3 Effect of Distance Between Emerging Aerosol and Impaction Bead for Aerosol Deposition in Furnace Atomic Absorption Spectrometry

Previous results from this laboratory<sup>5</sup> show the effect of increasing magnesium chloride concentration in determining manganese by furnace atomic absorption spectrometry. Modest amounts (<0.5%) of magnesium chloride introduced by a pneumatic nebulization aerosol deposition technique to the furnace do not interfere, but greater than 0.5% cause severe depression. Alkali and alkaline earth chlorides are well known interferences in furnace AAS and have been attributed to the formation of molecular chlorides on the atomizer surface and in the vapor phase<sup>7,8</sup>. The results of 10  $\mu$ l manual injections and of various distances

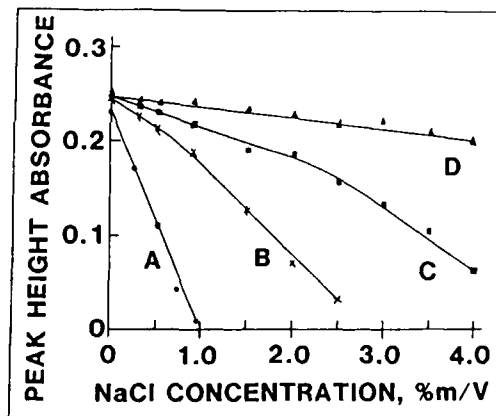


FIG. 5. Effect of increasing sodium chloride concentration on absorbance signal: A - 10 µl aliquot injections, B-aerosol deposition for 10s at no impact bead, C-aerosol deposition for 10s at 4.06 mm distance, and D-aerosol deposition for 10s at 1.09 mm distance.

between the emerging aerosol and impaction bead for  $10 \mu\text{g l}^{-1}$  of copper with increasing sodium chloride concentration is shown in FIG. 5. For manual injection (A) a severe depression is obtained with only a small increase in NaCl concentration. When no impaction bead is present, a depression occurs (B) and when the distance is 4.06 mm the depression is less severe (C). At a distance of 1.09 mm (D), a depression occurs, but at 4% NaCl it is only 16% compared to almost complete depression of the signal at previous distances. An examination of the graphite tube by electron microscopy shows much smaller particles at the 1.09 mm

distance compared to the other distances. It is probable that the particle size generated by the pneumatic nebulizer plays an important part in reducing interferences. The aerosol deposition technique when combined with a platform and matrix modification is shown to reduce interferences associated with  $\text{NaCl}^9$ .

#### CONCLUSION

The distance between the emerging aerosol and impaction bead for the pneumatic nebulization of increasing concentration of dissolved solids in a flame and furnace is shown to be critical for nebulization efficiency, imprecision, absorbance signals, and interferences. The particle size generated is considered to be the major reason for these changes although no experimental data is provided to support this hypothesis. Studies in correlating particles size with experimental data is in progress.

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#### FOOTNOTE

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