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New injection technique for flow injection atomic absorption spectrometry using tubing clamp valves

Received: 1 December 1995 / Revised: 27 December 1995 / Accepted: 8 January 1996

Abstract Flow injection devices for atomic absorption spectrometry usually are equipped with a sixport-valve consisting of two PTFE-cylinders. This kind of valve can be substituted by an arrangement of tubing clamp valves. The advantages and limits of this new injection technique for flow injection atomic absorption spectrometry are discussed.

Introduction

Sample injection in flow injection atomic absorption spectrometry [1] is usually carried out by a sixport-valve consisting of two PTFE-cylinders (stator and rotor). A weak point of this part of the instrument is the blockage caused by suspended matter in the liquid stream.

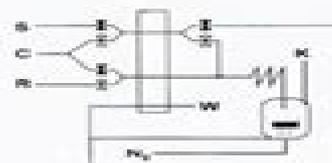
It has been reported that in non segmented continuous flow analysis such liquids difficult to handle can be analysed if the liquid stream is managed by tubing clamp valves [2]. Even in flow injection preconcentration tubing clamp valves have been used successfully [3].

In this paper, however, a new and very simple simulated sixport valve construction only consisting of tubing clamp valves is described.

Experimental

Mercury in suspended particulate matter was determined by cold vapour AAS. The samples were sieved and the fraction < 20 µm was dried and digested by various procedures [4]. Cold vapour AAS has been carried out with a "SpectroMesc" instrument (Spectro Analytical Instruments, Kleve, Germany) equipped with three low cost tubing clamp valves (Sirat, Prolabio, Italy). The manufac-

Fig. 1 Continuous flow system for mercury determination; S: sample, C: carrier, R: reagent, W: waste, K: to cuvette of CVAAS, N₂: nitrogen



turer recommend continuous flow conditions as shown in Fig. 1. As carrier (C) nitric acid solution (2 mol/L) and as reagent (R) stannous (II) chloride solution (25 g SnCl₂ dissolved in 25 ml conc. HCl filled up to 500 ml with distilled water) were used. These chemicals (analytical grade) and the mercury stock solution for preparing standards were obtained from Merck (Darmstadt, Germany).

Results and conclusions

Working with real samples in the recommended mode (and sometimes standard solutions, too) gives a poor reproducibility (e.g. r.s.d. of about 5%; [S(Hg)] = 5 µg/L), probably because the tubing clamp valves at the suction side of the pump might open with delay. The reproducibility is improved significantly (r.s.d. of about 1%; [S(Hg)] = 5 µg/L) by changing the flow path (Fig. 2) into a flow injection system. Now, in a first step the sample is filled into an injection loop with an exactly given volume

Fig. 2 Flow injection system for mercury determination; S: sample, C: carrier, R: reagent, W: waste, K: to cuvette of CVAAS, N₂: nitrogen



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296

(the three valve positions characterized by filled symbols are closed, the three others are open) and in a second step the chosen sample volume is injected into the carrier stream (the three valve positions characterized by filled symbols are open, the three others are closed). To realize this flow injection system for valve synchronization only two signals are necessary: (i) filling the loop: all valves are switched off, (ii) injection: all valves are switched on.

Twenty samples of suspended particulate matter, ten soil samples and a few other samples have been analyzed using two working ranges: 1-10 µg Hg²⁺/L with a 500 µL sample loop and 10-100 µg Hg²⁺/L with a 15 µL sample loop. This device is very robust and it shows the typical advantages of a flow injection system.

The use of tubing clamp valves offers a more flexible flow analysis, because these valves easily can be integrated into the manifold. The six port valve now can be divided into different parts, one of these can be located close to the sample, and the other part close to the instrument and so on. Another advantage: they are not impaired by particles in the flow stream. Moreover the costs of the valve construction described in this paper is several times lower than those of the classical six ports. As restriction must be mentioned that tubing clamp valves inserted at the suction side of the pump might open with delay. In addition, PTFE tubes cannot be used in clamp valves. Working with very corrosive chemicals demands a suitable material (resistant and flexible for clamping) which might be difficult to obtain.

Flow Injection Atomic Absorption Spectrometry

Wenbin Ji



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Table of Contents Flow Injection Atomic Absorption Spectrometry

1. Understanding the eBook Flow Injection Atomic Absorption Spectrometry
 - The Rise of Digital Reading Flow Injection Atomic Absorption Spectrometry
 - Advantages of eBooks Over Traditional Books
2. Identifying Flow Injection Atomic Absorption Spectrometry
 - Exploring Different Genres
 - Considering Fiction vs. Non-Fiction
 - Determining Your Reading Goals
3. Choosing the Right eBook Platform
 - Popular eBook Platforms
 - Features to Look for in an Flow Injection Atomic Absorption Spectrometry
 - User-Friendly Interface
4. Exploring eBook Recommendations from Flow Injection Atomic Absorption Spectrometry
 - Personalized Recommendations
 - Flow Injection Atomic Absorption Spectrometry User Reviews and Ratings
 - Flow Injection Atomic Absorption Spectrometry and Bestseller Lists

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 - Flow Injection Atomic Absorption Spectrometry eBook Subscription Services
 - Flow Injection Atomic Absorption Spectrometry Budget-Friendly Options
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 - Flow Injection Atomic Absorption Spectrometry Enhanced eBook Features
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 - Interactive Elements Flow Injection Atomic Absorption Spectrometry
8. Staying Engaged with Flow Injection Atomic Absorption Spectrometry
 - Joining Online Reading Communities
 - Participating in Virtual Book Clubs
 - Following Authors and Publishers Flow Injection Atomic Absorption Spectrometry
9. Balancing eBooks and Physical Books Flow Injection Atomic Absorption Spectrometry
 - Benefits of a Digital Library
 - Creating a Diverse Reading Collection Flow Injection Atomic Absorption Spectrometry
10. Overcoming Reading Challenges
 - Dealing with Digital Eye Strain
 - Minimizing Distractions
 - Managing Screen Time
11. Cultivating a Reading Routine Flow Injection Atomic Absorption Spectrometry
 - Setting Reading Goals Flow Injection Atomic Absorption Spectrometry
 - Carving Out Dedicated Reading Time
12. Sourcing Reliable Information of Flow Injection Atomic Absorption Spectrometry
 - Fact-Checking eBook Content of Flow Injection Atomic Absorption Spectrometry
 - Distinguishing Credible Sources
13. Promoting Lifelong Learning

- Utilizing eBooks for Skill Development
 - Exploring Educational eBooks
14. Embracing eBook Trends
- Integration of Multimedia Elements
 - Interactive and Gamified eBooks

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