AUTOMATED FLOW SYSTEM FOR CADMIUM EXTRACTION IN TOBACCO SAMPLES WITH ICP-MS DETECTION

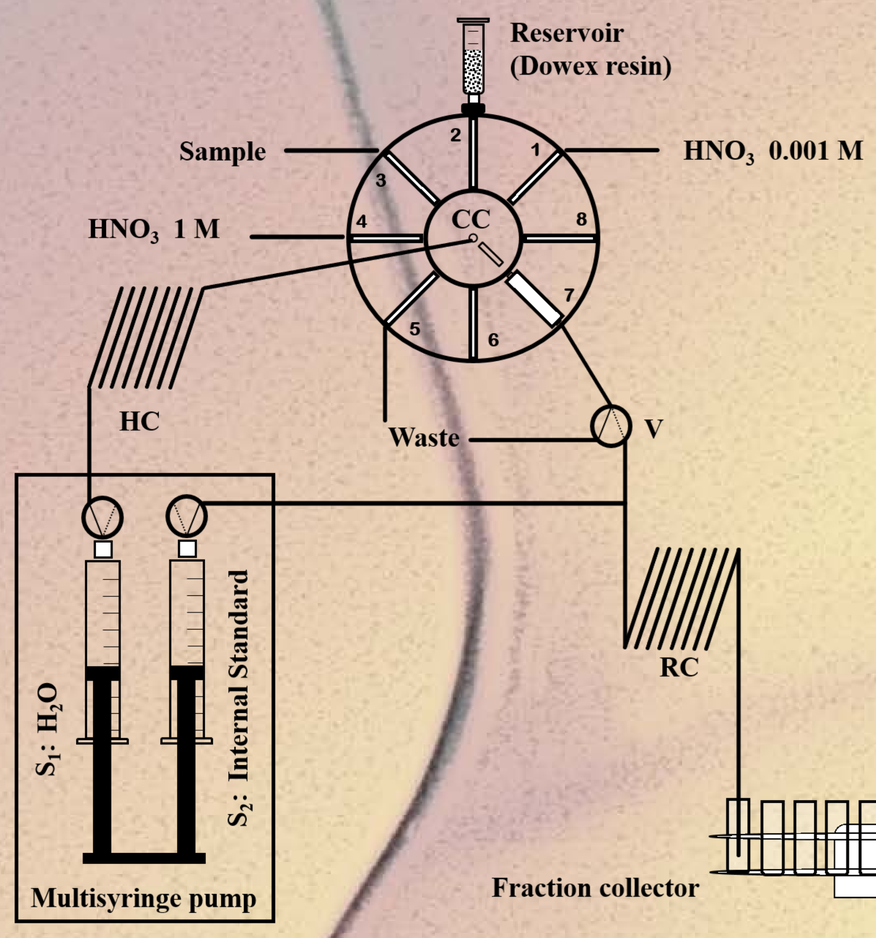
Rogelio Rodríguez-Maese [a,b], Luz O. Leal [a], Laura Ferrer [b], and Víctor Cerdà [b]

[a] Environment and Energy Department, Advanced Materials Research Center (CIMAV) S.C., Chihuahua, 31136, Mexico

[b] Department of Chemistry, University of the Balearic Islands, Palma E-07122, Spain

A miniaturized Lab-on-Valve-Multisyringe Flow Injection Analysis (LOV-MSFIA) system for cadmium determination by ICP-MS was developed. The presented method allows the automated extraction and preconcentration of cadmium by means of a Dowex 50W-X8 resin in several types of tobacco samples matrices. The on-line extraction and preconcentration system retains the analyte on the resin, followed of elution with 1 mol L^{-1} HNO_3. The proposed system permits the complete automation of solid phase renewal, which allows a durability of 130 injections for a single resin load. The best operating conditions were studied using multivariate optimization tools. The detection limit was 0.05 ng of cadmium, while the mass linear working range was 0.2–100 ng. The precision of the proposed methodology was 2.4% (RSD, n = 10) with a sample frequency of 9 injections h^{-1}. The accuracy of the method was established by means of an organic matrix certified reference material (DORM-2), and the method has been successfully applied to different samples of tobacco (commercial cigarettes, pipe tobacco and tobacco leaves), obtaining recoveries between 90 and 105%. Main advantages of the proposed methodology are the reduction of reagents volume, the minimization of sample handling, the improved reproducibility, high stability of the column and the versatility achieved by a variety of managed sample volumes, attaining a significant decrease of both time and cost per analysis.

ANALYTICAL PROCEDURE AND FEATURES



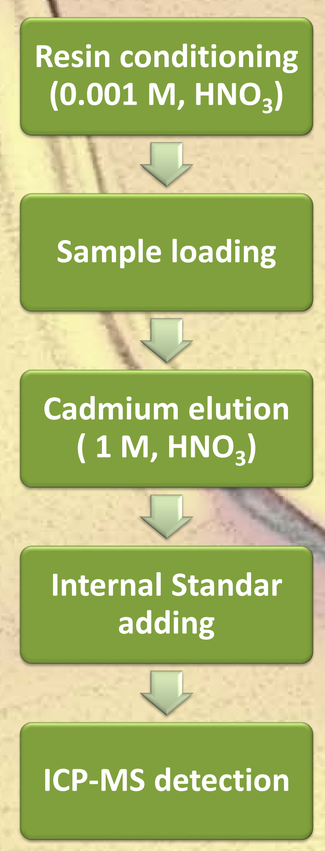


Figure 1. LOV-MSFIA system for cadmium extraction and preconcentration. Central conduit (CC), external solenoid valve (V), holding Coil (HC), Reaction Coil (RC), syringes (S1, S2).

Analytical features

Detection limit (ng)	0.05
Quantification limit (ng)	0.2
Intra-day precision (%) (n=10)	2.4
Inter-day precision (%) (n=5)	3.2
Sample volume (mL)	1-50
Resin durability (Injections)	130
Preconcentration volume (mL)	50
Preconcentration Factor	8.3
Sensitivity (^{114}Cd, CPS ng^{-1})	256.1
Linear working range (ng)	0.2-100
Coefficient of determination (R^2)	0.9993
Extraction throughput a (h^{-1})	1.6-9 h^{-1}

[a] Depending on the sample volume to preconcentrate

Sample Analysis

Sample*	Added Cd (ng)	Found (ng)	Recovery (%)
Pueblo	0	31.2±0.4	
	26	58.0±1.0	103
Camel	0	37±0.1	
	21	56±0.8	90.5
Philip Morris	0	31.2±2.7	
	25	54.0±1.2	91.2
Gold Copot	0	27.4±0.9	
	25	51.0±0.4	94.4
Marlboro	0	29.4±0.8	
	25	52.9±0.1	94
Tobacco Plant	0	8.70±0.2	
	25	35.5±0.3	107

Sample*	Certified value (mg kg^{-1})	Found (mg kg^{-1})**
CRM (DORM-2, NRC)	0.043 ± 0.008 mg kg^{-1}	0.0480 ± 0.0005 mg kg^{-1}

*Results are expressed as average ± standard deviation (*n*=3).
**The t-test of comparison of means revealed no significant differences at the 95% confidence level.

CONCLUSIONS

A fully automated extraction and preconcentration LOV-MSFIA method has been developed for cadmium determination using ICP-MS as detection technique. The present analytical methodology was successfully applied to the determination of trace levels of cadmium in different commercial tobacco samples.

Main features of the developed method are the fully automation of the isolation/preconcentration procedure, including the automatic on-line replacement of the microcolumn; a low detection limit at ultra-trace levels and high precision in handling volumes of sample and reagents used. In addition, with the proposed automated system ICP-MS limitations to analyze cadmium are overcome, enlarging the good performance of the ICP-MS and improving its selectivity and sensitivity.

ICFIA 2017