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Determination of lead and cadmium in tea by graphite furnace atomic absorption

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Abstract

Lead and cadmium were determined in the presence of matrix modifiers. Under the optimal measure conditions, lead and cadmium in tea showed excellent linearities with correlation coefficients of 0.9996 and 0.9993 after digestion of tea with HNO_3/HCl (3:1, v/v). The recoveries were 94–102% and 91–103% and RSD values were 8.86% and 13.1%, respectively.

Keywords: cadmium; graphite furnace atomic absorption spectrometry; lead; matrix modifiers; tea.

Introduction

Tea, as a natural health beverage, has a long history (Todorovska et al. 2002). Tea contains various substances beneficial to human health, such as tea tannins, aromatic oil, vitamins and alkaloids, and is considered a health drink. The quality of tea is of great concern with the application of various pesticides in tea planting (Fang et al. 1991). Much attention has been focused on the pesticide residues (Zhu et al. 2006). Heavy metals such as lead and cadmium have serious effects on organisms and are strictly monitored (GB 9679-1988). These metals can be effectively detected using ICP-AES, ICP-MS and GFAAS, etc. (Bing 2004, Husáková et al. 2007). Despite the abundant detection approaches, the methods for rapid detection of heavy metals in tea are still challenging. Herein we wish to establish a fast and accurate approach for lead and cadmium detection based on the application of graphite furnace atomic absorption.

Materials and methods

Instrument and reagents

All measurements were carried out on a graphic furnace atomic absorption spectrometer (Analytik Jena, Jena, Germany) with a hollow cathode lamp at 283.3 and 228.8 nm for lead and cadmium, respectively. Standard solution was prepared according to the standard procedure. Pure water was redistilled from a Human UP-900 Ultra-pure water system (conductivity of 18.3 MΩ·cm). Tempering electrothermal board type. V (HNO_3)+V (HCl)=3+1.

Methods

Instrumentation and operating conditions (Table 1)

Graphite furnace temperature selection (Table 2)

Sample preparation 0.2–0.25 g of tea leaves was washed, dried and crushed and treated with 15 ml of mixed acid in a beaker (100 ml) covered with watch glass. The mixture was slowly heated on a heat electrothermal board until no yellow gas (NO_2) liberates. The solution was then evaporated to nearly dryness. Upon cooling, the mixture was transferred to a volumetric flask and diluted with water to a volume of 50 ml. The blank solution was prepared according to the procedure except that no tea was used.

Results and discussion

Sample pretreatment process

The accuracy of the measurement mainly depends on the sample treatment and conditions for the determination. In this study, the effect of various concentrations of acid on the determination was investigated. The results indicate that a mixture of HNO_3/HCl (3:1) was found to be optimal in light of the absorption.

Effects of ashing and atomization temperature

Ashing temperature and atomization temperature of lead The relationship between atomization, ashing temperature and absorbency when the standard solution of lead (10 ppb) was used is shown in Figure 1.

Ashing temperature and atomization temperature of cadmium The relationship between atomization, ashing temperature and absorbency when the standard solution of cadmium (2 ppb) was used is shown in Figure 2.

Effects of matrix modifiers

Matrix modifiers can reduce the decomposition temperature of the matrix and improve the thermal stability of the analyte, thereby eliminating or reducing the matrix interference on

Table 1 Instrument parameters.

Element	Wavelength	Current (mA)	Voltage (V)	Band pass (nm)	Measure methods	Playback mode
Pb	283.3	3	308	0.8	Standard curve	Peak height
Cd	228.8	3	250	0.8	Standard curve	Peak height

Pb, lead; Cd, cadmium.

Table 2 Program for GFAAS.

Entry	Type	Temperature (°C)	Rates (°C/s)	Hold (s)	Time (s)
1	Drying	75 (75)	3 (3)	20 (20)	37 (41.3)
2	Drying	90 (90)	1 (1)	20 (20)	35 (35)
3	Drying	110 (110)	2 (2)	10 (10)	20 (20)
4	Pyrolysis	800 (400)	250 (250)	25 (25)	27.4 (26.4)
5	Autozero	800 (400)	0 (0)	4 (4)	4 (4)
6	Atomization	1600 (1500)	1500 (1500)	3 (3)	3.6 (3.7)
7	Cleanout	2300 (2300)	500 (500)	4 (4)	5.4 (5.6)

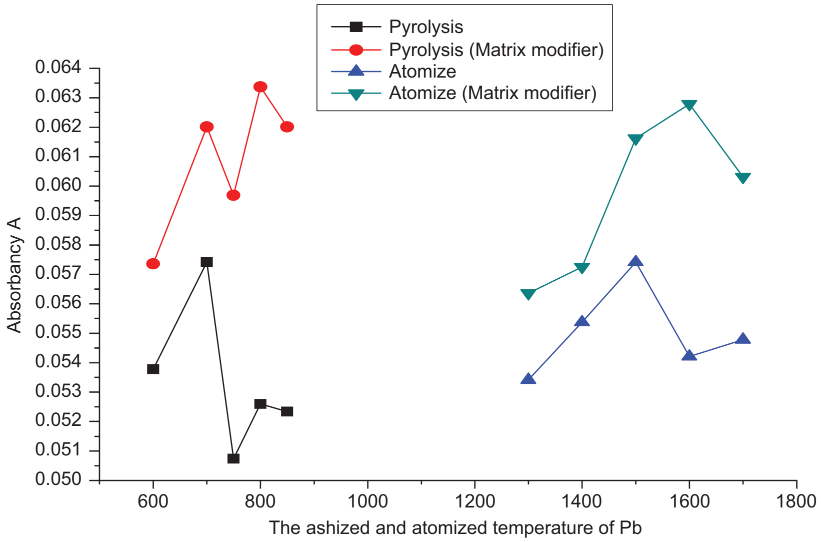

Figure 1 Ashing temperature and atomization temperature of lead.

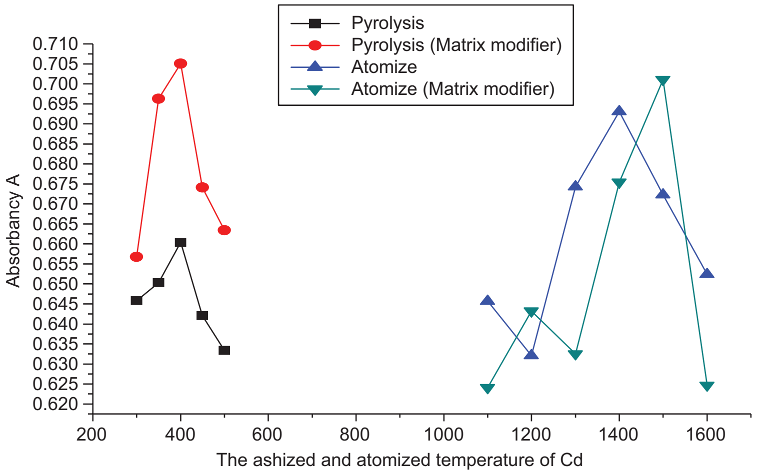

Figure 2 Ashing temperature and atomization temperature of cadmium.

Table 3 Determination results for tea (mg/kg).

Sample	Element	1	2	3	4	5	6	Average
Green tea	Pb	0.8852	0.8450	0.8809	0.8627	0.8175	0.8520	0.8568
	Cd	0.0346	0.0336	0.0340	0.0365	0.0335	0.0342	0.0344
Black tea	Pb	0.6693	0.7218	0.6789	0.7284	0.7266	0.7380	0.7105
	Cd	0.0433	0.0447	0.0444	0.0402	0.0425	0.0441	0.0432
Scented tea	Pb	0.9286	0.8966	0.9395	0.9061	0.9214	0.8855	0.9130
	Cd	0.0557	0.0533	0.0554	0.0503	0.0539	0.0545	0.0538

Pb, lead; Cd, cadmium.

Table 4 Precision determination results (ng/ml).

Element	1	2	3	4	5	6	7	RSD%
Pb	1.6332	1.5615	1.7688	1.6748	1.7906	1.7488	1.7904	8.86
Cd	1.1547	0.9523	1.2654	1.0593	1.0579	1.0565	0.8602	13.1

Pb, lead; Cd, cadmium.

Table 5 Recovery results[a].

Element	Background value (ng/ml)	Sample+standard solution+ultrapure water (ml)	Results of determination (ng/ml)	Recovery (%)
Pb	1.7097	5+0+15	0.4034	94.4
		5+5+10	0.9157	98.7
		5+10+5	1.4485	101.5
		5+15+0	1.8997	98.6
Cd	1.0580	5+0+15	0.2434	94.4
		5+5+10	0.7332	95.9
		5+10+5	1.2945	102.4
		5+15+0	1.7543	99.4

Pb, lead; Cd, cadmium.

[a]The experimental results show that the recovery rate of the standard addition method for lead and cadmium were 94–102% and 91–103%, respectively.

the measurement results. $NH_4H_2PO_4$ (4.0 g/l) and $Mg(NO_3)_2$ (0.1%) were used in this experiment, respectively. It can be seen in Figures 1 and 2 that the atomization temperature of lead increased to 100°C while that of cadmium remained unchanged. In the case of 0.1% $Mg(NO_3)_2$, the ashing temperature of both lead and cadmium increased to 100°C.

Determination of sample

Under the optimal conditions, lead and cadmium were determined and the results are summarized in Table 3.

Precision experiment

Under the identical conditions, seven parallel samples were analyzed and the results and RSD values of repeated measurements were excellent, as summarized in Table 4.

Determination of recovery

In the tea samples tested, the mean value of determination (n=7) was used as the background value. The experimental results show that the recovery rate of the standard addition method for lead and cadmium was 94–102% and 91–103%, respectively (Table 5).

Conclusions

In conclusion, an accurate and rapid approach for the determination of lead and cadmium in tea are disclosed. The present measurement shows excellent linearities with correlation coefficients of 0.9996 and 0.9993 after digestion of tea with HNO_3/HCl (3:1 v/v). The recoveries were 94–102% and 91–103%, and RSD values were 8.86% and 13.1%, respectively.

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