Journal of Food and Drug Analysis, Vol. 11, No. 3, 2003, Pages 214-219 藥物食品分析 第十一卷 第三期

Determination of Arsenic in Edible Oils by Direct Graphite Furnace Atomic Absorption Spectrometry

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(Received: August 28, 2001; Accepted: January 27, 2003)

ABSTRACT

A direct analytical method of arsenic content in edible oil with graphite furnace atomic absorption spectrophotometer was studied. Edible oil was diluted with n-heptane followed by the determination of arsenic with Zeeman graphite furnace atomic absorption spectrophotometer and transverse heated graphite atomizer (THGA). The arsenic organic AAS standard solution was used as calibration standard, and bis (benzonitrile) dichloro palladium solution was used as a matrix modifier. The results showed that the optimal ashing and atomizing temperatures were 1200˚C and 2300˚C, respectively. The detection limit was estimated to be 0.010 ppm, and the percentages of recovery for the spike of 0.05, 0.10 and 0.20 ppm arsenic to salad oil, fish oil, palm oil, and lard were 91.2~94.3%, 94.7~95.6%, 94.2~96.7% and 93.3~94.1%, respectively. With the use of this method, thirty edible oils were analyzed and the arsenic contents were found to be below the detection limits.

Key words: graphite furnace atomic absorption spectrophotometer (GFAAS), arsenic, edible oils

INTRODUCTION

Arsenic is a hazardous heavy metal. The malignant symptoms may appear even when trace arsenic is ingested. The toxicity mechanism of arsenic has been shown that it binds to enzymes, which are inhibited for functioning[1]. The toxicity of arsenide depends on the forms of chemical compound. The trivalent arsenic shows higher toxicity. It can be relatively retained in tissue but slows excretion. The organic arsenic complexes, which are commonly found in food, are readily absorbed in the body and excreted through urination. Arsenic in body shows high affinity to cuticle. Therefore, arsenic can be more readily accumulated into hair and nail than other tissues. In Taiwan, the tolerance level of arsenic in edible oil is set to be 0.1 ppm.[2].

Arsenic can be determined by using the following methods: Colorimetry[3, 4], Atomic Absorption Spectrometry[4, 5], hydride generation system combined with Atomic Absorption Spectrmetry[4, 6], and Atomic Fluorescence Spectrometry[7]. Using above methods, digestion is required prior to instrumental analysis. Edible oils are low in arsenic concentration and are difficult to be digested. The literatures on the analysis of arsenic in edible oils are also limited.

Graphite furnace atomic absorption spectrophotometer (GFAAS) is the analytical instrument used for trace element analysis. It has been widely applied to the determination of lead in food[8-17], biological samples[18], and environmental contaminates[19-24]. Trace amount (20 mL) of samples is dried and ashed by a way of electrothermal heating to eliminate sample matrix, followed by a process of atomization for analyte detection. This method enables test samples to be directly introduced into graphite tube without digestion. The advantages of using this method are requiring only little amount of samples, allowing direct introduction of samples, high sensitivity, and rapid in terms of analysis. In this study, the application of this method on arsenic analysis in edible oils was performed. The optimal ashing and atomizing conditions, limit of detection, and recoveries from various edible oils were studied in order to develop a rapid and precise method for arsenic analysis.

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MATERIALS AND METHODS

I. *Source of Test Samples*

In total, 30 test samples were purchased from supermarkets and distributors in Taipei during the time period between August and December 2000. These included sunflower oils, vegetable oils, sesame seed oils, salad oils, olive oils, grape seed oils, canola oils, corn embryo oils, peanut oils, lards (refined and fractionated), and palm oils (refined and fractionated).

II. *Reagents*

Arsenic solution (in 0.5 mole nitric acid), nickel nitrate solution, cyclohexane, n-heptane, and n-hexane were purchased from E. Merck (Darmstadt, Germany). Pd &

$Mg(NO_3)_2$ matrix modifier was obtained from High-Purity Standards (USA). Arsenic organic AAS standard solution (in xylene) was purchased from Alfa (USA). Bis(benzonitrile) dichloro palladium was obtained from Aldrich (USA). Soybean lecithin was the product of Nacalai Tesque (Japan). Pure water was prepared in our laboratory by Milli-Q SP (Millipore, USA.). To make a standard stock solution, arsenic standard (1 mL) was quantitatively diluted to 100 mL with n-heptane. Standard working solutions were freshly prepared by diluting the stock solution with n-heptane to an appropriate concentration before analysis. Bis(benzonitrile) dichloro palladium (0.3604 g) was accurately weighed into a 100 mL volumetric flask and dissolved with 5 mL of acetone. Alcohol was then added to the volume to make a concentration of 1000 mg/mL Pd solution.

III. *Equipment*

Zeeman Graphite Furnace Atomic Absorption Spectrophotometer PE 4110ZL, transverse heated graphite atomizer (THGA) B050-4033 and arsenic electroless discharge lamp (EDL) were made by Perkin-Elmer (Germany). Thermostated oven was the product of Memmert (Germany). Volumetric flasks and all glassware were Pyrex bland. Before use, they were washed with detergent and water, soaked with nitric acid solution (HNO_3: H_2O = 1 : 1, v/v) overnight, rinsed with water , and dried.

IV. *Methods*

In order to obtain the optimum operation conditions, 20 μL of oil solution containing 50 ng/mL arsenic was injected into GFAAS and the temperatures were programmed according to operation manual. The optimal ashing temperature was tested under fixed drying and atomizing temperatures. The atomizing temperature was optimized at a set ashing temperature. The instrumental parameters for GFAAS are listed in Table 1.

While analyzing the arsenic in oil samples, the drying temperature was increased from 80˚C to 110˚C in 20 sec for removing n-heptane (bp = 98˚C) from oil. The temperature was then raised up to 130˚C in 20 sec and kept at this temperature for another 20 sec to prevent the oil from splattering and leading to an analytical error. Soot may appear when the temperature of oil samples reach 350~450˚C. The ashing temperature was therefore held at 450˚C for 20 sec before programmed to the optimum (Table 2), allowing soot to be completely evacuated, as well as keeping oil from spattering.

An inorganic arsenic standard solution was diluted to 50 ng/mL with water, while an organic arsenic standard solution was diluted to the same concentration with n-heptane. Studies on the effect of different standard solutions on absorbance were conducted by introducing the above solutions to GFAAS.

The different dilution solvents were compared for the effect of absorbance. A series of concentrations (10, 20, 30, 40, and 50 ng/mL) of arsenic were prepared using the following dilution solvents: n-hexane, n-heptane, cyclohexane, and 2% lecithin in cyclohexane followed by detection of GFAAS.

The modifier effect on absorbance was studied by introducing different concentrations (10, 20, 30, 40, and 50 ng/mL) of arsenic solution with bis(benzonitrile)dichloro palladium, Pd & $Mg(NO_3)_2$, or nickel nitrate as a modifier to GFAAS.

The Characteristic mass (m_0) was determined by injecting 20 μL of arsenic standard solution (50 μg/L) to GFAAS under the optimal temperature programming and calculated according to the equation as follows:

$$m_0 = C \times V \times 0.0044 / A$$

Where C is concentration of analyte (mg/L), V is sample injection volume (mL), and A is absorbance.

Standard deviation (SD) was calculated after making 10 injections of blank reagent and the instrumental detection limit (IDL) was determined using the following equation: $IDL = 3 \times SD \times m_0 / 0.0044$.

The detection limit of method was also determined. Arsenic standard solution with concentration of 5 times of IDL was spiked into salad oil (n = 7) and analyzed using the developed method. The concentrations of each sample were calculated. The detection limit was set to be 3 times of standard deviation.

The effect of sample matrix on absorbance was conducted as follows: Standard arsenic solution was spiked into different oil samples: salad oil, palm oil, fish oil, and

Table 1. Instrument parameters for the determination of arsenic in edible oils using graphite furnace atomic absorption spectrophotometer

Parameter	Setting
Wavelength (nm)	193.7
Slit width (nm)	0.7
Signal measurement	Peak area
Lamp	Electrodeless discharge
Lamp current (mA)	380
Purge gas	Argon
Sample vol.	20 μL
Modifier vol.	2 μL

Table 2. Temperature program for the determination of arsenic content in edible oils by GFAAS

Temperature (˚C)	Ramp Time (sec)	Hold Time (sec)	Internal Flow (mL/min)	Gas Type	Read
110	10	20	250	Argon	—
130	20	20	250	Argon	—
350	60	60	250	Argon	—
450	20	20	250	Argon	—
1200	30	30	250	Argon	—
2300	0	5	0		+
2500	1	5	250	Argon	—

lard. Heptane was then added to the volume of 10 mL. A series of concentrations (10, 20, 30, 40, and 50 ng/mL) of spiked samples were thus prepared. Solid fat was heated and melted at 80˚C oven for 15 min prior to application. The spiked samples were then analyzed using GFAAS.

Recovery test was performed using the following procedure. Five mL of each oil sample, including salad oil, palm oil, fish oil, and lard, was spiked with arsenic standard solution and quantitatively added with n-heptane to 10 mL. The finial spiked arsenic concentrations 0.05, 0.10, and 0.20 ppm in oil were thus prepared. The spiked samples were analyzed, and the recoveries of arsenic from different oil samples were calculated by detection of the arsenic concentrations in blank samples from those of spiked samples.

V. *Analysis of Arsenic in Commercial Oils/Fats*

The commercial available oils or fats (3g) were accurately weighed. The fats in solid form were heated and melted in 80˚C oven for 15 min before weighing. The above samples were then quantitatively standardized to 10 mL with n-heptane and analyzed using GFAAS. Bis (benzonitrile) dichloro as a matrix modifier.

RESULTS AND DISCUSSION

I. *Optimum Conditions for Analysis*

A concentration of 50 ng/mL arsenic oil solution was prepared by spiking arsenic standard to salad oil followed by diluting with n-heptane. The optimal ashing temperature for detecting arsenic in oil was studied with the atomizing temperature set at 2300˚C. Results showed that peak absorbance was found at 1200˚C when the temperature was programmed between 1100˚C and 1300˚C. The optimal atomizing temperature was also studied by programming the temperature between 2200˚C and 2400˚C at a fixed ashing temperature of 1200˚C. Results showed that the peak absorbance was reached at 2300˚C (Figure 1).

The above optimal ashing and atomizing temperatures were set to be the temperatures for the detection of two arsenic standard solutions in this study. The standard curves plotted with peak areas versus concentrations were obtained by a linear regression calculation. The linear equation for the inorganic arsenic standard solution was calculated to be Y = 0.00231X + 0.0007 with relative coefficient 0.9977. As for the organic arsenic, standard solution was Y = 0.00164X + 0.0014 with relative coefficient 0.9966 (Figure 2). The slope of the standard curve of inorganic arsenic solution is significantly higher than the curve of organic arsenic solution, indicating the instrumental response to inorganic arsenic is more sensitive than that to the organic arsenic solution. However, the organic arsenic standard solution was chosen in this study, because test samples were dissolved in organic solvent, which is incompatible with water soluble standard solution in conducting recovery test.

II. *Effect of Diluting Solvents*

Organic arsenic standard was diluted with the

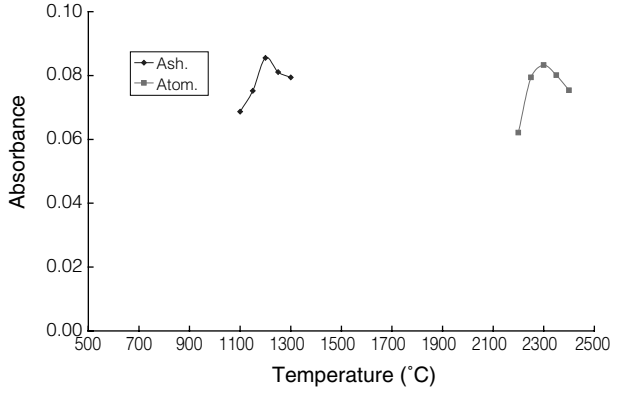

Figure 1. Optimal ashing and atomizing temperature to measure arsenic in edible oils. (Optimize ashing temperature with the atomizing temperature at 2300˚C, and atomizing temperature with the ashing temperature at 1200˚C.)

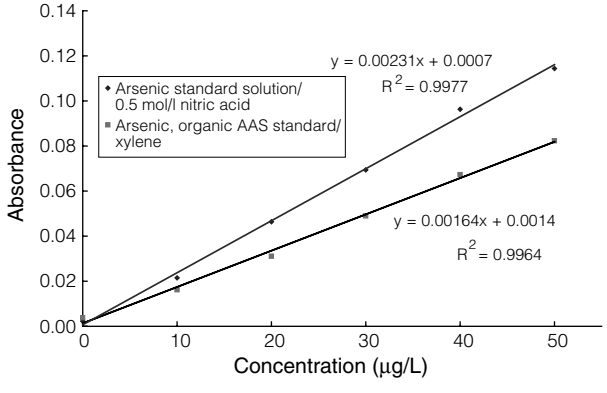

Figure 2. Ratios of linear calibration curves obtained from various type of arsenic standard solution.

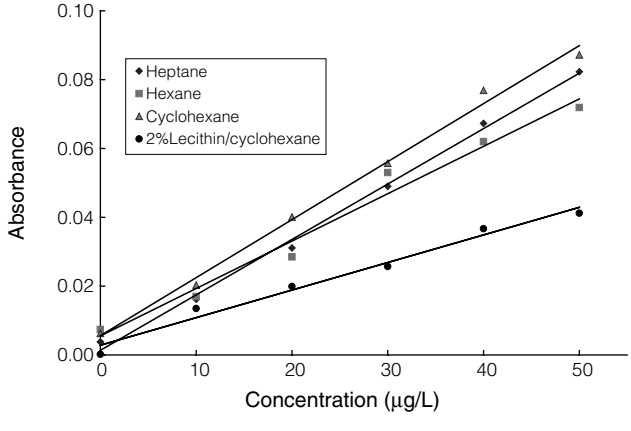

Figure 3. Effect of solvent on absorbance of arsenic standard solution in edible oils.

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following solvents: n-heptane, n-hexane, cyclohexane, or 2% lecithin/cyclohexane to concentrations of 10, 20, 30, 40, and 50 ng/mL. Above solutions were then analyzed with GFAAS to study the effect on absorbance. The standard curves are shown in Figure 3. The slopes of the standard curve using solvent cyclohexane, n-heptane, n-hexane, and 2% lecithin/cyclohexane are 0.00172, 0.00164, 0.00143, and 0.00082, respectively. The results revealed that lecithin could interfere the detection of arsenic resulting in the lower absorbance. Among those diluting solvents, cyclohexane could lead to the highest absorbance in arsenic detection. However, it was not selected due to its high toxicity. Instead, n-hexane was used as the diluting solvent in this study in order to reduce the hazard to the analysts.

III. *Effect of Modifiers*

The inorganic and organic modifiers were compared on the effect of absorbance. Arsenic standard solutions with concentrations, 10, 20, 30, 40, and 50 ng/mL were separately spiked with adequate amount of matrix modifier, nickel nitrate solution, Pd & $Mg(NO_3)_2$, or bis (benzonitrile) dichloro palladium, followed by analysis with GFAAS under optimum ashing and atomizing temperatures. Above arsenic detection corresponding to the three modifiers generated three calibrators with slopes 0.00095, 0.00103, and 0.00164, respectively (Figure 4). The inorganic matrix modifiers, nickel nitrate solution and Pd & $Mg(NO_3)_2$, which are water soluble chemicals, are difficult to be well mixed with sample matrix, resulting in inaccuracy in GFAAS detection. Therefore, the slope is lower. On the contrary, the organic modifier, bis(benzonitrile) dichloro palladium, is capable of being thoroughly mixed with sample solution, allowing palladium to be well reacted with arsenic in sample as temperature of GFAAS is raised. The purpose of using modifying matrix was thus achieved. Therefore, bis (benzonitrile) dichloro palladium was used as the matrix modifier in this study.

IV. *Limit of Detection*

The absorbance on an optimal temperature program was obtained by injecting 20 μL of arsenic standard solution (50 ng/mL) to GFAAS. The characteristic mass (m_0) was thus calculated to be 54 pg. The detection limit of instrument was determined to be 2.0 μg/L after detection of blank reagent. An adequate amount of standard solution was spiked to salad oil, which was then analyzed (n = 7). The detection limit of method was determined to be 0.010 ppm, based on 3 times of standard deviation from 7 analyses.

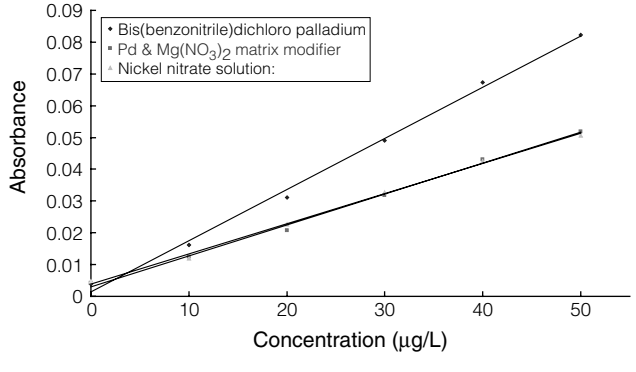

Figure 4. Effect of matrix modifier on absorbance of arsenic standard solution in edible oils.

V. *Comparison of the Slopes of Standard Curves Generated from Different oil Samples*

Slope ratio of the standard curves between spiked sample and standard solution can be used as a reference for evaluation of sample matrix effect on analysis system. A slope ratio close to 1 indicates the standard curve generated from spiked sample is similar to that generated from standard solution. It also illustrates that the analysis is less affected by sample matrix. A slope ratio less than 1 indicates the analysis is interfered by sample matrix; while a slope greater than 1 represents a positive effect occurred from the matrix. When examining the slope ratio generated from different oils, the standard curves generated from the following oils showed the slope ratio to be greater than 1: grape seed oil, olive oil, canola oil, corn embryo oil, fish oil, lard (refined and fractionated), and peanut oil. The slopes were 1.13, 1.13, 1.07, 1.06, 1.11, 1.04, and 1.12, respectively. On the other hand, standard curves generated from the following oil showed the slope less than 1: sunflower oil, vegetable oil, sesame seed oil, salad oil, and palm oil (refined and fractionated). The slopes were 0.91, 0.85, 0.34, 0.88, and 0.99, respectively. All oils tested in this study could produce the standard curves with slope ratio in the range of 0.85~1.15, with the exception of

Table 3. Ratios of slope of linear calibration curves obtained from various type of standard solution and edible oils

Standard solution and edible oils	Slope	Ratio*
Standard solution	0.00169	1
Grape seed oil	0.00192	1.13
Olive oil	0.00192	1.13
Canola oil	0.00182	1.07
Corn embryo oil	0.00180	1.06
Fish oil	0.00188	1.11
Lard (refined and fractionated)	0.00175	1.04
Peanut oil	0.00190	1.12
Sunflower oil	0.00153	0.91
Vegetable oil	0.00144	0.85
Sesame seed oil	0.00058	0.34
Salad oil	0.00150	0.88
Palm oil (refined and fractionated)	0.00168	0.99

*: Slope of curve from standard arsenic added in edible oil and fat was divided by slope of curve from arsenic organic AAS standard solution (in xylene).

sesame oil, which had low slope ratio (Table 3). Therefore, it is suggested that calibration curve method can be used for analysis of arsenic in most oil samples. Although analysis of oil sample such as sesame oil, which could generate much matrix effect, using standard addition method instead of linear calibration method is recommended. Standard addition method is used for calibration of matrix effect. It has been used for analysis of copper content in infant formula[13], heavy metal in fish oil[24], and lead and copper contents in edible oils[10, 11].

VI. *Recovery Test*

Recovery test was performed by spiking several concentrations of arsenic standard to salad oil, palm oil, fish oil, and lard, which were then analyzed using the established method. Recoveries from salad oil at 0.05, 0.10, and 0.20 ppm were found to be 91.2 ± 4.9%, 92.5 ± 4.2%, and 94.3 ± 4.0%, respectively; from fish oil were 94.7 ± 5.2%, 95.3 ± 4.5%, and 95.6 ± 4.3%, respectively; from palm oil were 94.2 ± 5.1%, 95.3 ± 4.7%, and 96.7 ± 4.5%, respectively; and from lard were 93.3 ± 4.8%, 93.7 ± 4.7%, and 94.1 ± 4.6%, respectively (Table 4). All recoveries were greater than 90% with coefficient of variation less than 5%. Above results indicate the established method is capable of yielding a satisfactory recovery.

VII. *Analysis of Arsenic Content in Commercialized Edible Oils*

Several commercial available oils including sunflower oil, vegetable oil, sesame oil, salad oil, olive oil, grape seed oil, canola oil, corn embryo oil, peanut oil, lard (refined and fractionated), and palm oil (refined and fractionated) were tested using the method developed in this study. Results showed that the arsenic contents in all tested oils containing arsenic were below the detection limit of 0.015 ppm. A similar result was observed by Chen *et al.*[7]. They analyzed numbers of edible oil using a microwave digestion method followed by atomic fluorescence spectrometer detection. Except one peanut oil and one palm oil samples, which contained 0.027 and 0.025 ppm arsenic, respectively, others were detected to be less than 0.015 ppm. These included peanut oil (0.006 ppm), sesame oil (0.006 and 0.011 ppm), olive oil (0.006 and 0.012 ppm), sunflower oil (0.005 and 0.006 ppm), salad oil (0.006, 0.007, and 0.012 ppm), tea seed oil (0.006 ppm), butter (0.009 ppm), lard (0.007 ppm), corn oil (0.011 ppm), refined and fractionated lard (0.011 ppm), canola oil (0.013 ppm), and vegetable oil (0.013 ppm).

Table 4. Arsenic recoveries for palm oil, salad oil, lard and fish oil

Spiked (ppm)	Recovery (%)			
	Salad oil	Fish oil	Palm oil*	Lard*
0.05	91.2** ± 4.9***	94.7 ± 5.2	94.2 ± 5.1	93.3 ± 4.8
0.10	92.5 ± 4.2	95.3 ± 4.5	95.3 ± 4.7	93.7 ± 4.7
0.20	94.3 ± 4.0	95.6 ± 4.3	96.7 ± 4.5	94.1 ± 4.6

*: refined and fractionated

**: Average of three determinations.

***: Relative standard deviation.

CONCLUSION

The method developed in this study allowed oil sample to be simply diluted with n-heptane, added with bis (benzonitrile) dichloro palladium as a matrix modifier, and directly introduced to transverse heated graphite tube and GFAAS for arsenic analysis. The standard addition method was used to replace linear calibration method to analyze the oil samples such as sesame oil, which could generate significant matrix effect on analysis. This developed method eliminates the complicated and dangerous oil digestion procedures. It not only ensures accuracy, but efficiency in analysis.

ACKNOWLEDGEMENTS

We thank Dr. C. W. Chen for his translation.

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