

Application News

No. A487

Spectrophotometric Analysis

Direct Determination of Pb in Edible Oils by GF-AAS

■ Introduction

Edible oils, probably the most widely used cooking ingredient in the world, are produced from a wide variety of basic ingredients, including fruits (olives, coconuts, palm, etc.), nuts (walnuts, macadamia nuts, almonds, etc.), seeds (sesame, sunflower, etc.), and a variety of plants (soy, canola, capsicum (peppers)). These oils are not only used for frying and baking, but are also consumed as food, serving as dressings and toppings, etc. Thus, with this wide consumption of edible oils, it is important to not only ensure their safety and non-toxicity, but that they satisfy quality specifications.

Here, using the Shimadzu AA-7000 atomic absorption spectrophotometer and the GFA-7000 graphite furnace atomizer, we introduce an example of direct quantitation of lead in edible cooking oil in accordance with AOAC Official Method 994.02, Lead in Edible Oils and Fats – Direct Graphite Furnace Atomic Absorption Spectrophotometric Method.

■ Sample

Sesame oil (one type)

■ Sample Preparation

First, a matrix modifier solution was prepared by dissolving 2 g of lecithin in cyclohexane modifier (2 % w/v solution of lecithin). The measurement sample consisted of a mixture of 5 g of sesame oil and 5 g of 2 % w/v lecithin solution. For measurement validation, a spike and recovery test sample was prepared by adding 50 ppb of lead to the equivalent of a sample solution.

■ Instrument and Analytical Conditions

For the measurement, the Shimadzu AA-7000 atomic absorption spectrophotometer and GFA-7000 graphite furnace atomizer were used together with the ASC-7000 autosampler. The optics parameters that were used are shown in Table 1, and the temperature program, in Table 2.

When analyzing samples containing large amounts of organic solvents and organic compounds, accurate control of temperature and gas flowrate from drying to atomization is important to ensure the vaporization, decomposition and elimination of solvents and organic compounds.

The GFA-7000 graphite furnace atomizer supports heating at temperatures from ambient to 3000 °C, and the high-sensitivity optical sensor and digital temperature control system provide accurate temperature control over the entire temperature range from drying to atomization. In addition, the dual electronic flowrate control system permits independent setting of the argon gas flowrate to 0.01 L/min, thus providing high-accuracy measurement with high sensitivity.

Table 1 Optics Parameters

Lamp current (mA)	10
Measurement wavelength (nm)	283.3
Slit width (nm)	0.7
BGC mode	BGC-D2

Table 2 Temperature Program

	Temperature (°C)	Time (Sec)	Heating Mode	Sensitivity	Gas Type	Gas Flowrate
1	120	10	RAMP	<input type="checkbox"/>	#1	0.30
2	120	20	STEP	<input type="checkbox"/>	#1	0.30
3	800	60	RAMP	<input type="checkbox"/>	#1	0.30
4	800	20	STEP	<input type="checkbox"/>	#1	0.30
5	800	3	STEP	<input type="checkbox"/>	#1	0.00
6	1800	5	STEP	<input type="checkbox"/>	#1	0.00
7	2500	1	RAMP	<input type="checkbox"/>	#1	0.05
8	2500	3	STEP	<input type="checkbox"/>	#1	0.05

Atomization Stage No. 6

■ Analysis

Lead standard solutions of 20, 50, and 100 ppb were prepared by appropriately diluting a commercially available oil mixture standard solution (CONOSTAN®S-21) with blank oil. Preparation was conducted on a weight basis (wt%) using commercially available salad oil as blank oil for dilution.

Mixtures consisting of 5 g of each of the standard solutions and 5 g of 2 % w/v of lecithin solution were used as standard solutions. Using a platform graphite tube for measurement, the sample injection volume was 10 µL.

Analytical Results

Fig. 1 shows the calibration curve obtained using this method, and Fig. 2 shows the peak profiles for the respective standard solutions. Table 3 shows the measurement results and spike and recovery results for sesame oil. Excellent spike and recovery results were obtained with nearly 100 % recovery. Fig. 3 shows the peak profile obtained in the measurement of sesame oil.

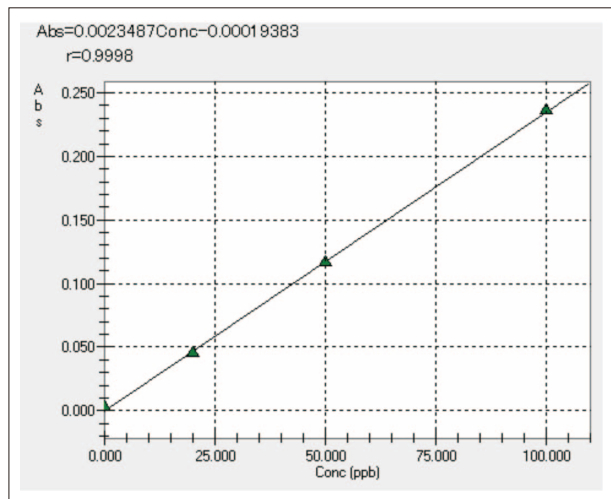


Fig. 1 Calibration Curve for Pb in Edible Oil

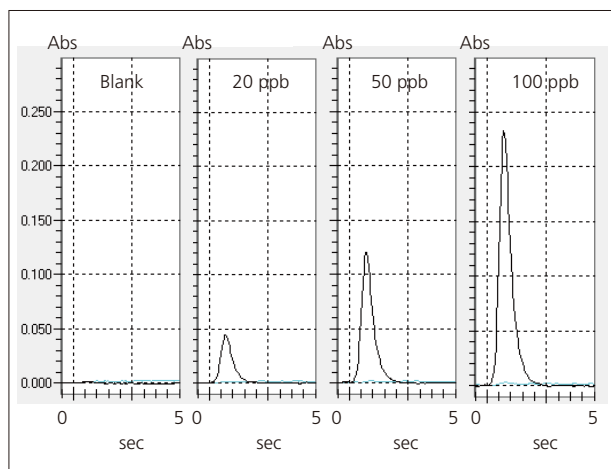


Fig. 2 Peak Profile of Pb in Standard Solution

Table 3 Measurement Results for Pb in Sesame Oil

Element	Sesame Oil	50 ppb Spike and Recovery Test Solution	Recovery (%)
Pb	< 4 ppb*	50 ppb	100 %

* Absorbance 0.01 or less

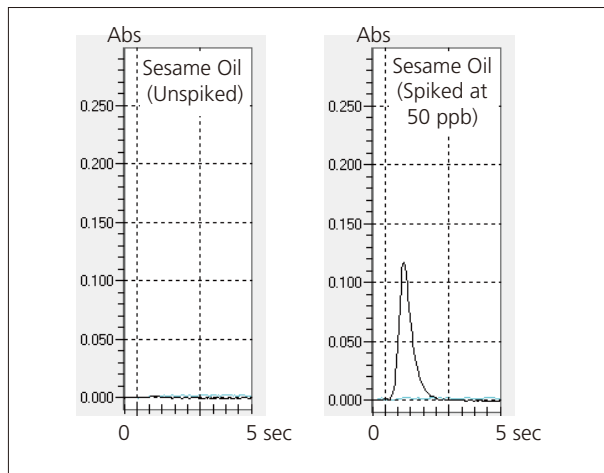


Fig. 3 Peak Profile of Pb in Sesame Oil

Summary

In this Application News, we conducted direct analysis of lead in edible oils using the AA-7000 and ASC-7000 autosampler together with the GFA-7000 graphite furnace atomizer in accordance with AOAC Official Method 994.02. Excellent sensitivity and recovery were obtained over the specified concentration range (18 µg/kg (ppb) or greater).

References

- 1) AOAC Official Method 994.02, Lead in Edible Oils and Fats, Direct Graphite Furnace Atomic Absorption Spectrophotometric Method
- 2) Application News No. AA-003 "AOAC 994.02: Determination of Pb in Edible Oils and Fats by GF-AAS" SHIMADZU SCIENTIFIC INSTRUMENTS (SSI)