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Research Article

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Determination of nickel and lead in hogwash oil by graphite furnace atomic absorption spectrometry with graphite digestion

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ABSTRACT

This article studies the method for determination of Nickel and Lead in hogwash oil by graphite furnace atomic absorption spectrometry with graphite digestion. The digest reagent, matrix modifier, ashing temperature, and atomization temperature are being optimized. Results showed that the detection limit of Nickel and Lead are 0.03 and 0.10 mg/kg, respectively. The results of recovery and RSD are satisfactory, indicating that this method is feasible for determination of Nickel and Lead in hogwash oil.

Key words: graphite digestion; graphite furnace atomic absorption spectrometry; hogwash oil

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INTRODUCTION

Hogwash oil generally refers to the poor quality domestic oil including recycled cooking oil and repeated fried oil. Consumption of hogwash oil could bring a high level healthy risk such as indigestion, abdominal pain and diarrhea, and the long term consumer may have a high potential risk to get gastrointestinal cancer. In recent years, the accident of hogwash oil abused happens frequently which becomes a social problem and cause wide public concerns. Then in Dec 2011, the Ministry of Health openly collected the method for detecting hogwash oil. Until May 2012 there are 4 instrument methods and 3 rapid detection tests identified and to be used on-site, which represents hogwash oil detection technology made some progress [1]. However most methods are focus on PAHs detection, conductivity detection, specific genes detection and cholesterol detection, and there is a limitation for the heavy metal detection which is just focus on sodium ion and aluminum ion, such as Deng Hua [2] applied flame AAS determination to identify the concentration of sodium ion absorption in the oil. As we know, nickel and lead are toxic metal element which could cause serious health risks. Intake large amount of nickel would lead retrogression of internal organs and increase the risk of cancers. And the lead also could poison human and relate to disease occur, such as dizziness, muscle and joint pain, insomnia, anemia, abdominal pain, irregular and menstruation. In this condition, National Food Safety Standard GB2762-2012[3] regulated the limitation of these two elements. And in this article, the method for determination of Nickel and Lead in hogwash oil by graphite furnace atomic absorption spectrometry with graphitedigestion could achieve the standards.

EXPERIMENTAL SECTION

2.1 Experimental Instruments and Related Parameters

Graphite furnace atomic absorption spectrometry (USA, PerkinElmer, Inc.), nickel hollow cathode lamp, lead hollow cathode lamp, ST40 type graphite digestion instrument(Beijing Puli Tyco Instrument Co. Ltd.) and1/10000 electronic analytical balance (Sartorius Scientific Instrument Co. Ltd.).
The operating parameters of graphite furnace are shown in Table 1.

Table1 The operating parameters of graphite furnace

element	Analyzing Wavelength(nm)	Lamp Current(mA)	matrix modifier	Sample amount(μl)	Background correction
Ni	232.0	4	$NH_4H_2PO_4$	20	Zeeman
Pb	283.3	4	$NH_4H_2PO_4$	20	Zeeman

2.2 Main reagent and Material

500mg/L of nickel standard solution (National Standard Material Center), 500mg/L of lead standard solution (National Standard Material Center).HNO_3 (GR), hydrogen peroxide (analytical pure), perchloric acid (analytical pure), ammonium dihydrogen phosphate (analytical pure).Experimental water has resistivity of 18.2M Ω * cm deionized water which is produced by ultra-pure water manufacturing system (Chengdu ultra-pure Technology Co. Ltd.).

Hogwash oil sample 1: waste oil from hotel kitchen.

2.3 Experimental Methods

Take about 0.5g (accurate to 0.1mg) hogwash oil sample into PTFE digestion tank, then put digestion tank into a graphite digestion device. Set the digestion program for digestion, waiting until the sample cools to room temperature, then add nitrate acid 1:1 (volume ratio 1mL) to dissolve residue and transfer to a 50mL volumetric flask immediately. To reduce the error, take a small amount of deionized water to wash digestion tank and transfer to the volumetric flask. Under operation parameters in table 1, by using graphite furnace atomic absorption spectrometry, the concentration of the digested samples could be determined.

Electro thermal graphite digestion procedure is shown below in table 2.

Table2 The digestion procedure of hogwash oil

Procedure	Operation
1	Putting nitric acid 10mL and perchloric acid 2ml
2	Heating to 100℃,keeping 120 minutes
3	Cooling 10 minutes
4	Putting nitric acid 10mL
5	Heating to 120℃,keeping 120 minutes
6	Cooling 10 minutes
7	Heating to140℃,keeping 120 minutes
8	Cooling ,ending digestion

RESULTS AND DISCUSSION

3.1 Pretreatment related matters

Pretreatment would affect the final results directly. In order to get digestion process success and without the loss of lead/nickel, three digestion systems are applied in this test which areHNO_3, HNO_3 -H_2O_2 and HNO_3 -$HClO_4$. Then select the best digestion systems by comparison of digestion results.

Digestion results are shown in figure 1.

by Nitric acid by nitric acid -hydrogen peroxide by nitric acid - perchloric acid

Fig.1 Comparison of effect with different digestion reagent

By the comparison of three digestion system, both nitric acid and nitric acid-hydrogen peroxide systems could not achieve digestion completely. Only nitric acid - perchloric acid system could get clear yellow liquid after digestion which means the reaction of digestion is complete.

During the digestion process, perchloric acid must be added at room temperature, because the hogwash oil contains large amount of organics which is easy to be exploded at high temperature. Also digestion process should start with low temperature, to avoid carbonization of sample.

3.2 Function of Modifier

As cooked oil and hogwash oil contain salt, it contributes large amount of chloride ions which could react with lead and nickel then form volatile salt. And it would disturb the reactions in the graphite furnace atomic absorption spectrometry [4]. To remove this disturbance the modifier is needed, it contains ammonium salt which is much easier to react with chloride ion and form ammonium chloride, and this gas is easy to be evaporated. In this condition, the chloride ions are removed by the modifier, and all the lead/nickel could maintain. In this test, 2% (mass volume ratio)di-ammonium hydrogen phosphate is chosen, because only 5μ L ammonium dihydrogen phosphate could remove the disturbance.

3.3 Optimization of ashing temperature and atomization temperature

Generally ashing temperature is the high temperature to keep evaporating of modifier and remove the interference without any loss of lead and nickel. Oppositely atomization temperature is a low temperature to reduce the damage of graphite pipe with clearance of the interference [5]. In this test, 50μg/L nickel standard solution and 100μg/L lead standard solution is applied to optimize the result.

The curves of ashing temperature are shown in Fig 2 and atomization temperature are shown in Fig3.

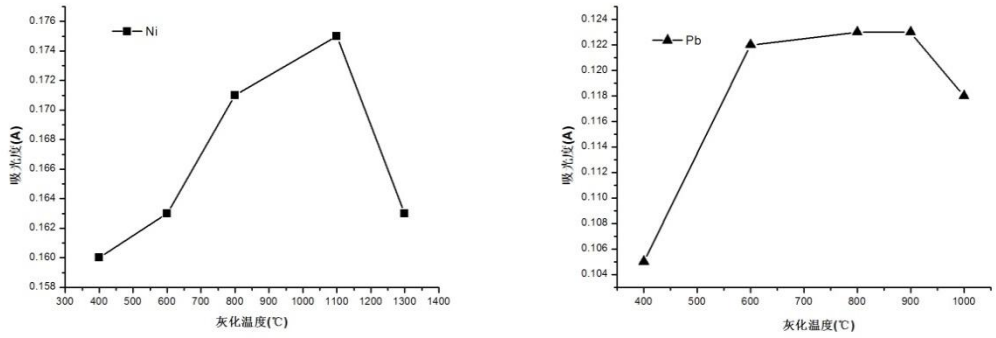

Fig.2 The curves of ashing temperature

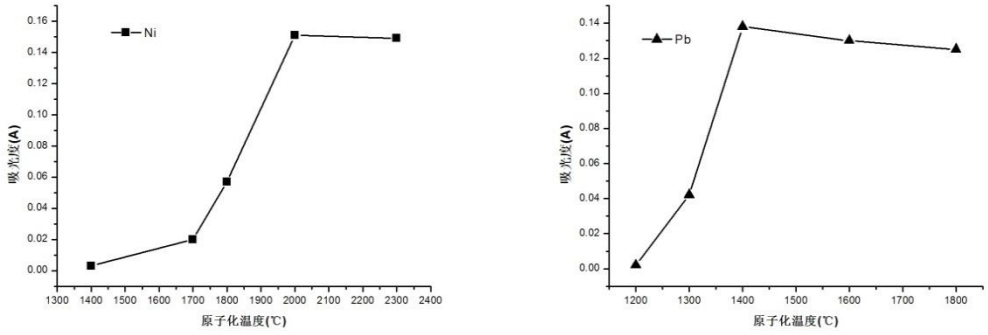

Fig.3 The curves of atomization temperature

Fig 2 and Fig 3 show that the sensitivity and absorbance are at the highest level when Nickel ashing temperature is 1100℃ and atomization temperature is 2000℃. The sensitivity and absorbance are at the highest level when Lead ashing temperature is 900℃and atomization temperature is 1400℃. During the experiment, peak tailing phenomenon has gradually improved and peak is gradually sharp as ashing temperature increases.

3.4 Calibration Curve

Prepare 500mg/L nickel solution and lead solution then dilute to 50μ g/L and 100μ g/L. By using graphite oven to get 0,10,20,30,40,50μ g/L nickel solution and 0,20,40,60,80,100μ g/L lead solution automatically. Under the settled condition of 2.3, a calibration curve could be calculated which is y=0.00353x+0.002, r=0.9994 for nickel and y=0.00141x+0.002, r=0.9992 for lead.

3.5 Precision and Recovery

Take drainage oil sample 1 into HNO3-bu HClO4 digestion system, the results of 6 times digestion with graphite furnace atomic absorption spectrometric method show in table 3. In table 3, RSD value (The relative standard deviation of the determination results) of nickel and lead element are small, it shows there is no sample contamination. The concentration of lead in drainage oil samples is 1.21μg/g, which is far more than the 0.1 μg/g lead limit in the national food safety standardsGB2762-2012 of fat class food provisions. Concentration of nickel is 0.86 μg/g, which is lower than 1 μg/g Ni limit in GB2762-2012 of fat class food stipulated.

Table 3 Precision test of the method (n=6)

m sample/g	w/(μg·g-1)	
	Ni	Pb
0.5020	0.82	1.25
0.5102	0.86	1.16
0.5074	0.89	1.22
0.5063	0.84	1.22
0.5027	0.87	1.21
0.5121	0.85	1.20
average	0.86	1.21
RSD(%)	2.8	2.5

The 500mg/L nickel and lead standard solution was diluted into 10mg/L. Weighing hogwash oil sample 1#0.5000g,recovery tests were made through the 10mg/L nickel and lead standard solution added each 50μ L. The Table 4 shows the result as below. It shows that, Nickel recovery is 94.0%, Lead recovery is 95.0%, which satisfy the analysis requirements.

Table4 Recovery test of the method

element	mB(μg)		ρB/(μg)		Recovery
	Sample 1# addition	Standard solution addition	Theoretical content determination	Actual content determination	R(%)
Ni	0.43	0.50	0.93	0.90	94.0
Pb	0.605	0.50	1.105	1.08	95.0

3.6Detection Limit

The blank solution is continue-determined 20times separately under the condition of graphite furnace, taking the sample of 0.50g, the concentration of 3 times of standard deviation of the average value is limit of each element. The detection limit of each element is: Pb0.10mg/kg,Ni0.03mg/kg.

CONCLUSION

Using mixed acid system 20mLHNO_3-2mL$HClO_4$ as digestion solution, through gradient heating with graphite digestion, the operation is fast and convenient and reduces the reagent dosage and physical labor of operator by the method for determination of Nickel and Lead in hogwash oil by graphite furnace atomic absorption spectrometry. Less reagent dosage is used and samples are completely digested by the method which is efficient to determine heavy metal elements in hogwash oil by graphite furnace atomic absorption spectrometry.

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