graphite furnace atomic absorption spectrometry (GFAAS)

2. principle

After digestion, the sample was atomized in a graphite furnace and its absorbance was measured at 283.3nm. The absorbance of lead in a certain concentration range is proportional to the lead content and is quantitative compared with the standard series.

3 .Reagents and materials

Unless otherwise stated, the reagents used in this method are superior grade pure and the water is secondary water specified in GB/T6682.

3.1 reagent

3.1.1 HNO_3

3.1.2 $HClO_4$

3.1.3 $NH_4H_2PO_4$

3.1.4 $Pd(NO_3)_2$

3.2 Preparation of reagents

3.2.1 Nitric acid solution (5+95): Take 50mL nitric acid, slowly add it to 950mL water, and mix well.

3.2.2 Nitric acid solution (1+9): Take 50mL nitric acid, slowly add it to 450mL water, and mix well.

3.2.3 Ammonium dihydrogen phosphate - palladium nitrate solution: Take 0.02g palladium nitrate, add a small amount of nitric acid solution (1+9) to dissolve the solution, then add 2g ammonium dihydrogen phosphate, dissolve the solution with nitric acid solution (5+95) to 100mL, and mix well.

3.3 standard

Lead nitrate [$Pb(NO_3)_2$,CAS No. :10099-74-8]: purity >99.99%. Or a standard solution of a certain concentration of lead certified by the state and certified as a standard substance.

3.4 Preparation of standard solution

3.4.1 Lead standard reserve solution (1000mg/L): accurately weigh 1.5985g(accurate to 0.0001g) lead nitrate, dissolve it with a small amount of nitric acid solution (1+9), move it to 1000mL volumetric flask, add water to scale, and mix well.

3.4.2 Lead standard intermediate (1.00mg/L): accurately absorb 1.00ml of lead standard reserve solution (1000mg/L) into 1000mL volumetric flask, add nitric acid solution (5+95) to scale, and mix well.

3.4.3 Standard lead solution series: Absorb the standard lead intermediates (1.00mg/L)0mL, 0.500mL, 1.00mL, 2.00mL, 3.00mL and 4.00mL into a 100mL volumetric flask, add nitric acid solution (5+95) to the scale, and mix well. The mass concentrations of the standard lead series solutions are 0 μg/L, 5.00 μg/L, 10.0 μ g/L, 20.0 μg/L, 30.0 μg/L and 40.0 μg/L.

Note: The mass concentration of lead in standard solutions can be determined according to the sensitivity of the instrument and the actual amount of lead in the sample

Note: All glassware and teflon digestion tanks shall be soaked in nitric acid solution (1+5) overnight, rinsed repeatedly with tap water, and finally rinsed clean with water.

4.1 Atomic absorption spectrometer: equipped with graphite furnace atomizer and lead hollow cathode lamp.

4.2 analytical balance: sensitivity of 0.1mg and 1mg.

4.3 Adjustable electric heating furnace.

4.4 Adjustable electric heating plate.

4.5 Microwave digestion system: equipped with ptFE digestion tank.

4.6 Constant temperature drying oven.

4.7 Pressure digestion tank: equipped with teflon digestion tank.

5 Analysis Steps

5.1 Sample Preparation Note: Sample contamination should be avoided during sampling and sample preparation.

5.1.1 Grain and bean samples shall be crushed and stored in plastic bottles after the sundries are removed.

5.1.2 Wash vegetables, fruits, fish, meat and other samples with water, dry them, take edible parts, make homogenate, and store them in plastic bottles.

5.1.3 Shake well the samples of beverage, wine, vinegar, soy sauce, edible vegetable oil, liquid milk and other liquid samples.

5.2 Sample pretreatment

5.2.1 The solid sample was digested by wet digestion, 0.2g~3g(accurate to 0.001g) was weighed, or the liquid sample was transferred into the digestive tube with scale, 10mL nitric acid and 0.5mL perchloric acid were added, and then digested in an adjustable electric heating furnace (reference condition :120 ℃/0.5h~1h; Rise to 180 ℃/2h~4h, 200 ℃~220 ℃). If the digestive juice is brown, add a small amount of nitric acid to dissolve it until it is white smoke, and the digestive juice is colorless, transparent or slightly yellow. Take out the digestive tube, cool it down with water for a constant volume of 10mL, and mix it well for later use. At the same time do reagent blank test. A conical flask on an adjustable electric heating plate can also be used for wet digestion according to the above operation method.

5.2.2 Microwave digestion Solid sample 0.2g~0.8g(accurate to 0.001g) or liquid sample 0.500ml ~ 3.00ml was accurately transferred into the microwave digestion tank, and 5mL nitric acid was added. The sample was then dissolved according to the operation steps of microwave digestion.

Refer to Appendix A for the digestion conditions. After cooling, take out the digestion tank and pour acid on the electric heating plate at 140 ℃~160℃ to about 1mL. After the digestion tank is cooled, the digestion solution is transferred to a 10mL volumetric bottle, and a small amount of water is used to wash the digestion tank for 2 ~3 times. The washing solution is combined in the volumetric bottle, and the washing solution is adjusted to the scale with water, and then mixed for later use. At the same time do reagent blank test.

5.2.3 The solid sample was digested by pressure tank, 0.2g~1g(accurate to 0.001g) was weighed, or the liquid sample was removed accurately, 0.500ml ~ 5.00ml, into the digestion tank, and 5mL nitric acid was added. Cover the inner cover, tighten the stainless steel coat, put it into a constant temperature drying oven, keep it at 140℃~160℃ for 4h~5h. After cooling, loosen the outer tank slowly, take out the digestion inner tank, and put it on an adjustable electric heating plate to drive the acid to about 1mL at 140 ℃~160 ℃. After cooling, transfer the digestion solution to a 10mL volumetric bottle, wash the inner tank and inner cover with a small amount of water for 2 ~3 times, and combine the washing solution in the volumetric bottle with water to the scale, and mix it for later use. At the same time do reagent blank test.

5.3 the determination of

5.3.1 Instrument reference conditions Shall be adjusted to the optimal state according to the performance of each instrument. See Appendix B for reference terms.

5.3.2 standard curve produced by the mass concentration of 10 μL, respectively, from low to high order will L lead standard series of solution and ammonium dihydrogen phosphate - 5 μL palladium nitrate solution (can be according to the use of the instrument to determine the best sampling amount) into the graphite furnace at the same time, measuring the absorbance values after atomization, mass concentration as the abscissa, absorbance value as the ordinate, making standard curve.

5.3.3 Determination of Sample Solution Under the same experimental conditions as the standard solution, 10 μL blank solution or sample solution and 5 μL ammonium dihydrogen phosphate - palladium nitrate solution (the optimal amount of sample injection can be determined according to the instrument used) were injected into the graphite furnace at the same time. After atomization, the absorbance was measured to compare quantitative with the standard series.

6 .The content of lead in the sample is calculated according to Formula (1) : X

$$X = (p - p_0) \times V / (m \times 1000)$$

X: -- The amount of lead in the sample in milligrams per kilogram or milligram per liter (mg/kg or mg/L);

p-- the mass concentration of lead in a sample solution in micrograms per liter (g/L);

p_0 -- the mass concentration of lead in blank solution in micrograms per liter (g/L);

V -- constant volume of the sample digestion solution, in mL;

M -- The sample weight or transfer volume, in grams or milliliters (g or mL);

1000 -- conversion factor.

When the lead content is ≥ 1.00mg /kg(or mg/L), three significant digits are retained in the calculation results; When the lead content is less than 1.00 mg/kg(or mg/L), the results retain two significant digits.

7 precision

The absolute difference between the two independent measurements obtained under repeatability conditions must not exceed 20% of the arithmetic mean.

8 other

When the sample weight was 0.5g(or 0.5mL) and the volume was 10mL, the detection limit of the method was 0.02mg/kg(or 0.02mg/L) and the determination limit was 0.04mg/kg(or 0.04mg/L).