LEAD LIMIT TEST（GB 5009. 12—2010）

Hydride atomic fluorescence spectrometry

After digestion and in acidic solution, lead in the samples will reacte with sodium borohydride (NaBH4) or potassium borohydride (KBH4) and will generate volatile lead hydride (PbH4).With argon as carrier, the volatile lead hydride (PbH4) will be transferred into quartz atomizer and be atomized. With exposure to the light of cathode lamp, lead atom will be activated and will emit special fluorescence. The strength of this fluorescence is proportional to the content of lead therefore could be calculated by the standard lead solution.

1 Reagent and materials

1.1 Nitric acid + high chlorine acid mixed acid (9 + 1) : take 900 mL of nitric acid and high chlorine acid 100 mL respectively,then mix them well together.

1.2 Hydrochloric acid (1 + 1 in quantity) : take 250 mL hydrochloric acid into 250 mL water, blend them well.

1.3 Oxalic acid solution (10 g/L) : take 1.0 g oxalic acid and dilute to 100 mL with water, blending them well.

1.4 Potassium ferricyanide solution [K3Fe (CN) 6] (100 g/L): take 1.0 g potassium ferricyanide and dilute to 100 mL with water, blending them well.

1.5 Sodium hydroxide solution (2 g/L) : take 2.0 g sodium hydroxide and dissolve in 1 L water, blending.

1.6 Sodium borohydride (NaBH4) solution (10 g/L) : NOTE that this solution should be parpared only before it is needed Take 5.0 g sodium borohydride and dissolve it in 500 mL sodium hydroxide solution (2 g/L), blending.

1.7 Standard Lead Solution On the day of use, dilute 10.0 mL of Lead Nitrate Stock Solution to 100.0 mL with Hydrogen Peroxide–Nitric Acid Solution, and mix. Each milliliter of standard Lead Solution contains the equivalent of 10 μg of lead (Pb) ion.

Standard Solutions Prepare a series of lead standard solutions serially diluted from the *Standard Lead Solution .* Pipet into separate 100-mL volumetric flasks 0.00 mL、0.125 mL、0.25 mL、0.50 mL、0.75 mL、1.00 mL、1.25 mL, respectively, of *Standard Lead Solution*, dissolve with a little water and then add 0.5 mL prepared hydrochloric acid and 0.5 mL prepared oxalic acid solution,mix and then add 1.0 mL Potassium ferricyanide solution. Then dilute to volume with water and mix. The *Standard Solutions* contain, respectively,0.02, 0.05, 0.1, and 0.2 _g of lead per milliliter. (For lead limits greater than 1 mg/kg, prepare a series of standard solutions in a range encompassing the expected lead concentration in the sample.)

2. Instruments and equipment

2. 1 Atomic fluorescence photometric

2. 2 Hollow cathode lamp made of lead.

2. 3 Electrothermal board for heating.

2.4 Electrothermal balance: sensitivity to 1 mg.

3. Procedure

3.1 Sample treatment

wet nitration: take 0.2 g ~ 2 g sample (accurate to 0.001 g) into a 50- mL or 100- mL digestive containers (such as tapered bottle), then add the nitric acid + high chlorine acid mixed acid 5 mL ~ 10 mL, mix them well and then place for a night. Heat them on a

electrothermal board until the remained solution turns into pale yellow or colorless (if the color during the heat is dark, cool it later and add some nitrite acid again then continue the heat).Cool the remained solution for a few minutes and then add 20ml water and continue the heat until the volume of the remained solution becomes 5ml~10ml.Cool it again then transfer it into a 25-ml volumetric flask with the help of a little water. Then add 0.5ml prepared hydrochloric acid and 0.5 ml oxalic acid solution.Mix and then add 1.0ml prepared potassium ferricyanide solution. After that accurately dilute the solution in the volumetric flask to 25ml with water, shake to make the solution mix well and then place for 30min.At the same time perform the reagent blank test.

3.2 Test

3.2.1 Adjust the instruments to the below working conditions:

Negative pressure: 323 V;

Electric current of the hollow cathode lamp : 75 mA;

furnace temperature of the atomizer:750 ℃ ~ 800 ℃, and the furnace height 8 mm;

Argon gas velocity: speed 800 mL/min;

Shielding gas: 1000 mL/min;

Time of add reductant: 7.0 s;

Reading time: 15.0 s;

Delay time: 0.0 s;

The measurement method: standard curve method;

Reading way: peak area;

Sample volume: 2.0 mL.

3.2.2 After setting the working conditions of the instrument, gradually increase the furnace temperature to 750 ℃ ~ 800 ℃ and keep the temperature steady for 10min~20min.Then continuous use standard series of zero tube samples wait until the reading is stable. Then test the standard solution to draw standard curve. Finally test the sample and the blank.

3.3 Calculation

$X=(c_1-c_0)*25*1000/(m*1000*1000)$

Reference

X: content of lead in the sample, the unit is milligrams per litre per kg or mg (mg/kg or mg/L)

c_1: concentration of the sample, the unit is the gram per milliliter (ng/mL)

c_0: concentration of the reagent blank, the unit is the gram per milliliter (ng/mL)

M:the weight or the volume of the taken sample

Repeat the whole procedure until the absolute difference of results of two tests of may not exceed 10% of their arithmetic mean. Take their arithmetic mean as the final result.