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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 5009.94-2012

Replacing GB/T 5009.94-2003; GB/T 7630-1987; GB/T 22290-2008; GB/T 23199-2008

Determination of rare earth elements in plant food

植物性食品中稀土元素

(English Translation)

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Foreword

This standard replaces GB/T 5009.94-2003 "Determination of rare earths in vegetable foods" and at the same time instead of GB/T 22290-2008 "tea Leaves of rare earth elements by inductively coupled plasma mass spectrometry ", GB/T 23199-2008" tea rare earth elements by inductively coupled Combined plasma atomic emission spectrometry and inductively coupled plasma mass spectrometry, "rare earth oxide wheat GB/T 7630-1987 1 Scope this standard specifies the method of rare earth elements in plant foods measured by inductively coupled plasma mass spectrometry.

This standard applies to grain cereals, legumes, vegetables, fruits, tea and other plant foods scandium (Sc), yttrium (Y), lanthanum (La), Cerium (Ce), praseodymium (Pr), neodymium (Nd), samarium (Sm), europium (Eu), gadolinium (Gd), terbium (Tb), dysprosium (Dy), holmium (Ho), Erbium (Er), thulium (Tm), ytterbium (Yb), lutetium (Lu) measurement.

Principle 2

The sample was treated as sample digestion solution, the sample solution is atomized by a carrier gas is fed into the plasma torch or ICP, after evaporation, Dissociation and ionization of atoms and other processes, converted to positively charged ions, the ion-collection system into the mass spectrometer, the mass spectrometer according to their mass charge

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Separation ratio. For the mass-to-charge ratio, the signal intensity is proportional to the mass number of the ions into the mass spectrometer, i.e., the concentration of the sample mass and the letter Number proportional to the intensity. Element concentration was determined by measuring the mass of the sample solution in the signal strength.

3.1 Reagents

Note: Unless otherwise indicated, the reagents used in this method are excellent pure water as a water GB/T 6682 regulations.

3.1.1

nitric acid (HNO3).

3.1.2

argon (Ar). purity argon gas (> 99.999%) or liquid argon.

3.2 Reagent preparation

Nitric acid solution (5 + 95). 50 mL of nitric acid, dilute with water to 1000 mL.

3.3 Standard

3.3.1

REE stock solution (10 µg/mL) (Sc, Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu).

3.3.2

the internal standard stock solution (10 µg/mL) (Rh, In, Re).

3.3.3

instrument tuning stock solution (10 ng/mL) (Li, Co, Ba, Tl).

3.4 Standard Solution

3.4.1

The use of rare earth elements mixed standard solution (100 ng/mL). Take the right amount of Sc, Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, the elements Ho, Er, Tm, Yb, Lu single standard or standard stock solution element mixed standard stock solution with a nitric acid solution by Grade diluted to a concentration of 100.0 µg/L using the element mixed standard solution.

3.4.2

standard curve working solution. Apply the solution using standard elements mixed with nitric acid solution formulated at a concentration of 0 µg/L, 0.0500 µg/L, 0.100 µg/L, 0.500

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μg/L, 1.00 μg/L, 2.00 μg/L standard series or concentration of 0 μg/L, 1.00 μg/L, 2.00 μg/L, 5.00 μg/L, 10.0 μg/L, 20.0 μg/L standard series, also based on the concentration of rare earth elements in the sample solution appropriate standards regulating the concentration series Range.

3.4.3
an internal standard solution (1 μg/mL). proper amount of internal standard stock solution (10 μg/mL), diluted 10-fold with a solution of nitric acid (595), the concentration of of 1 μg/mL.

3.4.4
instrument tuning use solution (1 ng/mL). instrument tuning take appropriate stock solution was diluted with a solution of nitric acid (595) 10-fold concentration of 1 ng/mL.

4 Instruments and equipment

4.1
Inductively coupled plasma mass spectrometry (ICP-MS).

4.2
Balance. a sense of the amount of 0.1 mg and 1 mg.

4.3
high-pressure sealed microwave digestion system with high pressure teflon digestion tank.

4.4
closed high pressure digestion device with digestion inner tank.

4.5
thermostatic oven (oven).

4.6 50 °C ~ 200 °C temperature heating plate.

5.1.1 Sample pretreatment

5.1.1.1
dry sample. grain cereals, beans and other edible parts taken by high speed grinding mill, mix and set aside.

5.1.1.2
wet sample. vegetables, fruits and other edible parts taken, washed clean, dry gauze or wipe dry, homogenized by homogenizer, spare.

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5.2 Sample Digestion

5.2.1

Microwave Digestion. Weigh 0.2 g ~ 0.5 g (accurate to 0.001 g) at high pressure digestion tank, added 5 mL HNO3, tighten the lid, Place 1 h, according to the microwave digestion instrument's standard operating procedures digestion (digestion conditions see Table A.1). Removed after cooling, slowly open the Lid exhaust, high pressure digestion tank placed on a hot plate temperature, rush acid at 140 °C. Remove and let cool digestion tank, digestive juices will be transferred to 10 mL ~ 25 mL flask with a small amount of water 3 washes tank washings were combined in the flask and dilute to volume, mix standby; while for trial Agent blank.

5.2.2

closed high pressure tank Solution. Weigh sample 0.5 g ~ 1.g (accurate to 0.001 g) in digestion in the tank, added 5 mL of nitric acid immersion overnight. Inside cover cap, tighten stainless steel jacket, put in oven temperature, 140 °C ~ 160 °C holding 4 h ~ 6 h, the natural cooling box to room temperature, slowly unscrew the stainless steel jacket, inside the digestion tank removed and placed on a hot plate temperature, rush acid at 140 °C. Digestion inner tank discharge After cooling, the digest was transferred to a 10 mL ~ 25 mL volumetric flask, with a small amount of water 3 washes tank washings were combined in the flask and is scheduled to volume, mix standby; at the same time as a reagent blank.

5.3 Instrument Reference conditions

5.3.1

The device according to standard operating procedures for commissioning the instrument start, quality calibration, argon gas flow rate and the like. Suitable conditions for selection, including Nebulizer flow rate detector and the ion lens voltage, RF incident power, so that the formation of oxides CeO/Ce 6 expression analysis I-th sample REE contents in accordance with the formula (1).

Where.

- I-th sample rare earth element content in milligrams per kilogram (mg/kg);

- I-th sample solution measured values of rare earth elements, in micrograms per liter (μg/L);

- Sample blank solution in the i-th measured value of rare earth elements, in micrograms per liter (μg/L);

V-- the sample digest volume to volume, in milliliters (mL of);

m-- sample weight in grams (g);

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