Simultaneous determination of 13 elements in pet food by microwave digestion -ICP-MS

Microwave drying machinery is a relatively new industry in China, with the rapid development of China's economic level, and pet food microwave drying equipment has also been rapid development in recent years, regardless of the size of the industry, or the quality of products, have a rapid improvement. However, compared with European and American countries, China's pet food standards are still very deficient, especially in nutritional standards (AAFCO, 1993), basically blank. ICP-MS is a fast, sensitive and anti-interference method for simultaneous determination of multi-elements in foods (Zmozinski et al., 2015; Herwig et al., 2011). Especially, the introduction of collision/reaction cell technology has greatly reduced the interference of polyatomic ions on the detection results. Compared with ICP-AES, ICP-MS has the advantages of less reagent consumption, lower blank value and higher sensitivity. However, there is still a gap in the application of ICP-MS in the determination of multi-elements in pet food in China, and there are few related reports.

1. Major Instruments and Reagents Inductively Coupled Plasma Mass Spectrometer (USA), Agilent 7500 CX; Microwave Digestor (USA), CEM Corporation, MARS 5; Ultra Pure Water System (USA), Millipore Corporation, Milli-Q A10; Electronic Balance (Germany), Metler Corporation, accuracy 1/10,000. Standard reserve solutions of K, Na, Ca, Mg, Fe, Mn, Cu, Zn, Hg, As, Pb, Cd, Cr: GBW produced by the General Institute of Steel Research, National Iron and Steel Material Testing Center, with a concentration of 1000 mg/L.

2. Instrument working conditions: microwave digestion instrument working procedure microwave digestion process. Inductively Coupled Plasma Mass Spectrometer Compensation Gas Flow 0.2 L/min, Carrier Gas Flow 1.05 L/min, Atomization Chamber Temperature: 2.0 C, Peristaltic Pump Acquisition Turn: 0.1 r/s, Radio Frequency Power 1500W; Sampling Depth: 8.0 mm, Repetition Number: 3; Eight Level Pole Voltage: - 18V, Four Level Pole Voltage: - 16V, Focusing Voltage 10V, Collision Collision pool mode.

3. A microwave digestion-inductively coupled plasma mass spectrometry (ICP-MS) method was developed for the determination of some minerals and toxic heavy metals in pet food. The results show that the method is simple, reliable, accurate and precise for the determination of mineral elements in pet food. The method was applied to the quantitative analysis of 13 elements in dog food and dog food chewables. It was found that there were significant differences in the contents of essential mineral elements between the two kinds of dog food, while the contents of toxic heavy metal elements were not significantly different. [Keywords] ICP-MS; pet food; mineral elements; heavy metals.

4. Microwave Digestion Procedure Initial Temperature/ Temperature Rising Time/ Min Endpoint Temperature/ Temperature Holding Time/ Min Normal Temperature 6 1202 120 5 1605 4 185 20 30 2016 Phase 21 China Feed Collision Gas (Helium) Flow: 0.4 L/min, the Number of Elements to be Measured and the Corresponding Internal Standard Elements: 39 K, 23 Na, 43 Ca, 24 Mg, 57 F E, 55 Mn, 65 Cu, 68 Zn (45Sc), 75As (72Ge), 208 Pb, 202 Hg (209Bi), 111 Cd (115 In), 53 Cr (45 Sc), integral time 75 As, 202 Hg 0.3 s, 111 Cd 0.5 s, the rest are 0.1 s.

Sample pretreatment method accurately weighed 0.5 g pet food sample, accurate to 0.0001 g, placed in a microwave digestion tube, microwave digestion tube to add 6 mL nitric acid, placed at room temperature for 10 minutes, sealed microwave digestion tube and placed in microwave digestion device, according to the set digestion procedures for digestion. After digestion, the digestion tube is lowered to normal temperature, and then the digestion solution is transferred to 50 mL plastic volumetric bottle. The digestion solution is rinsed with ultra-pure water for 2 to 3 times. The cleaning solution is also transferred to volumetric bottle. Then the volume is fixed with ultra-pure water to the scale, and the test solution is shaken evenly to make reagent blank.

5. RESULTS AND DISCUSSION: Choice of digestive acid and its dosage According to daily detection experience, pet food is easy to digest. Satisfactory results can be obtained by using only 6 mL nitric acid monoacid digestion system. In the process of inductively coupled plasma mass spectrometry (ICP-MS) with on-line addition of internal standard mixed solution, the analysis signal will drift with time, and the excessive salt content in the matrix will also have the effect of enhancing or inhibiting the measured elements. According to the selection principle of internal standard elements, this method chooses appropriate internal standard elements and adds a certain concentration of internal standard solution on-line to eliminate the above two effects. Collision/reaction cell technology can eliminate the interference of polyatomic ions. Helium is used as collision gas in the experiment. Inductively coupled plasma mass spectrometry (ICP-MS) with working curve, linear range and detection limit has a wide linear range for the determination of various elements. In the experiment, the concentration of elements in working solution is taken as abscissa (X), and the counting ratio of elements to internal standard elements is taken as ordinate (Y). After drawing the standard curve, the reagent blank solution was continuously determined 11 times, the standard deviation was calculated, and the detection limit was obtained. It can be seen that the 13 elements in the determination process showed good linear relationship, the correlation coefficient is greater than 0.9990, meet the detection requirements. Accuracy and precision accurately weigh 0.5 g dog food (accurately to 0.0001 g). According to the above pretreatment method, the recovery rate of the sample was tested. Each target element was added with high concentration and high precision.