Determination of sulfur in fresh fruits by microwave digestion -ICP-AES

Abstract: The fresh-keeping fruit samples were digested by microwave sterilization equipment with nitric acid-hydrogen peroxide (4+2) mixed solution as digestive agent. The content of sulfur in the sample solution was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES). 182.0 nm was selected as the analytical line for the determination of sulfur. The detection limit of sulfur was 3 Sd. The relative standard deviation (n=8) was 1.90%. Two national standard substances, tea (GBW 10016) and apple (GBW 10019), were Fruit sterilization equipment determined by this method. Compared with the barium sulfate gravimetric method, the results of this method are in good agreement, and the recovery of samples (n=8) is between 100% and 102%.

At present, litchi, longan and other fruits are mostly preserved by sulfur treatment technology, usually burning sulfur dioxide fumigated fruits or using dilute sodium sulfite solution impregnated fruits, play a role in inhibiting microbial activities, can effectively prevent or reduce pests during storage and acid deterioration, thus prolonging the storage period. This method has the advantages of low cost, good preservation effect and beautiful appearance of fruit. Although it is controversial, it is still widely used in the world. Generally speaking, the sulfur content (dry base) of fruits such as litchi and longan which have not been preserved is mostly below 600 mg 6550 Can cause harm.

At present, HNO3 + HClO4 digestion, BaSO4 turbidimetric method, acid or alkali fusion, BaSO4 gravimetric method (Eichka method), high temperature combustion neutralization and high temperature combustion iodometry are commonly used for the determination of sulfur. The turbidimetric method has low accuracy, low sensitivity and low results; the gravimetric analysis process is relatively lengthy; high temperature combustion neutralization and high temperature combustion iodometry procedures are cumbersome, poor repeatability, the results are often low. Fresh fruits have high sugar content, acid digestion time is longer than ordinary plant samples, operation is careless and easy to explode, easy to bring pollution. ICP-AES has been reported to determine sulfur content in soil, plants, cement, waste battery lead sulfate and other samples, but the determination of sulfur content in fresh-keeping fruits by ICP-AES has not been reported. The sample was digested by microwave with nitric acid-hydrogen peroxide system, and the content of sulfur in preserved fruits was determined by IC P-AES. The pretreatment time was short and the method was simple and feasible. The results of the experiment are in good agreement with each other. It is a sensitive and accurate method for rapid determination of sulfur content in preserved fruits.

All the glassware used were soaked overnight with nitric acid (65%) - water (1 + 2) solution, rinsed with water, and washed with ultra-pure water. The preparation of dry samples and the preparation of fresh samples are carried out according to the conventional food preparation method. Medium-step grating, two-dimensional cross-dispersion system; CID38 charge injection solid detector; spectral wavelength range 165-1000 nm; high frequency generator frequency 27.12 MHz; glass concentric atomizer, high swirl atomization chamber; peristaltic pump injection. Radio frequency power 1150W, cooling gas flow: 14L/min; auxiliary gas flow:

0.5L/min; carrier gas (atomized gas) pressure: 172kPa; observation height: induction coil above 14mm, solution lift 1.8mL/min, integration time 15s, sulfur analysis spectrum 180.7, 182.0nm. In view of the fact that the wavelength of sulfur element is in the vacuum ultraviolet region, a stable signal intensity can be obtained from the start of the heat engine to the determination of the sample after 2 hours of argon blowing. The SO 2-4 standard reserve solution was diluted step by step to prepare a series of SO 2-4 standard working solution with concentration of 0,50,100,200 ug/mL. The medium was 5% HNO 3. The linear regression equation of standard calibration curve was obtained by plotting the concentration C with spectral line strength I. The linear regression equation of standard calibration curve was I = 0.8565C + 0.2219 (180.7nm); I = 0.7669C-0.0521 (18.0521). 2. 0nm), in the range of 0 - 200 g /mL, the correlation coefficients of two wavelengths are all r= 0.9999.

Weigh the sample 1g (dry sample, accurate to 0.0001g) or 3G (fresh sample, accurate to 0.0001g) in the microwave digestion tank, a small amount of water wetting, adding 4mL nitric acid (65%) and 2mL hydrogen peroxide (30%), set the appropriate microwave digestion conditions (using the maximum microwave power 1500W, set the pressure 5516kPa, other digestion, digestion after the end of digestion. A small amount of water is washed into the 25mL colorimetric tube for a small amount of time. If placed for a long time, pour into polyethylene plastic bottles for preservation. At the same time, blank experiment was done. Temperature rise time (min) to temperature (?) Holding time (min) Power (W) 110 120 10 1200 2 150 15 1500 35 180 10 15003

RESULTS AND DISCUSSION: The pretreatment method of ICP-AES for the determination of sulfur content in high-sugar fruits, such as plant samples, usually uses nitric acid-perchloric acid (10+1) wet digestion and the organic matter digestion is thorough. Acid (10 + 1) system, a mixed acid, organic digestion is often not thorough, need to add 2-3 times mixed acid, digestion time is long, blank value is high, operation is not easy to explode; microwave digestion alone using nitric acid for high sugar content of fresh fruit digestion is not good, need to add oxidants to improve digestion effect, use strong oxidant perchloric acid efficiency The results are the best, but perchloric acid can not be used in microwave digestion because it is dangerous to be used in closed containers. The oxidant hydrogen peroxide can accelerate the destruction of organic matter, and the blank value is low. In this work, nitric acid - hydrogen peroxide (4 + 2) system was used to digest fresh fruit samples by microwave digestion, and the effect was very good.

After digestion, the main matrix elements in the solution are potassium (0.9% - 1.7%), phosphorus (0.2% - 0.4%), magnesium (0.06% - 0.1%), calcium (0.03% - 0.08%) and sodium (0.001% - 0.003%) in order to investigate the interference of matrix elements in the determination of sulfur in the solution. The dissolution of 10 mg L 1 SO 2 4 standard solution was determined by the method of experiment, and 10 mg L 1 SO 2 4 was determined at the wavelength of 180.7 and 182.0 nm. The matrix elements have little interference on the analytical line with the wavelength of 182.0 nm, but the matrix element calcium has a positive interference on the analytical line with the wavelength of 180.7 nm. With the increase of calcium content, the influence of the matrix elements on the determination of sulfur content is greater, while the other matrix elements have little influence on the determination of sulfur content.

In this paper, the full-spectrum direct-reading inductively coupled plasma-atomic emission spectrometer is used to determine the wavelength of two analytical lines with strong sulfur content, 180.7 nm and 182.0 nm, respectively. The concentration of calcium in the solution of fresh-keeping fruits digested by the experimental method is generally 10-30mg The detection limit of 10 blank solutions was 0.065 mg L - 1 with 3 times standard deviation.

According to the experimental method, the tea components and national standard substances such as apples were digested and determined, and the results of three parallel samples were determined. It can be seen from the results that the difference of sulfur content between the two standard samples is more than 5 times, and the results of three parallel samples are in good agreement with the standard values. Determination of Sulfur (S) in Standard Material GBW 10016 (Tea) and GBW 10019 (Apple) Results (n=3) Standard Value of Sulfur Content of Standard Material w(%) Determination Value w(%) Tea GBW 1001600.30.03 0.303 Apple GBW 1001900.063.004 0.067 etc. Microwave Digestion-ICP-AES Determination of Sulfur Content in Fresh Fruit

In order to investigate whether the digestion process caused the loss of sulfur, this paper selected dried Guiyuan, dried litchi two samples for recovery experiments, it can be seen that the digestion did not cause the loss of sulfur, recovery results are satisfactory. In order to investigate the accuracy of this method, six parallel samples of local cinnamon were determined by using anhydrous sodium carbonate melting, boiling water extraction and barium sulfate gravimetric method. The results of visible determination were in good agreement with those of this method. Comparing with the results of barium sulfate gravimetric method, the precision of the RSD (% determination times of mean value (g/g) of ICP-AES 0.111 2.28 barium sulfate gravimetric method 0.103 8.16 3.7 sample is weighed as 8 local cinnamon samples. The relative standard deviation of sulfur element (n=8) is calculated after the experimental method is processed and determined. It is 1.90%.

Samples of 16 kinds of fruits and fresh fruits were collected and processed according to the experimental method. Sample test results (n = 2) Sample name sulfur content (mg kg - 1) Sample name sulfur content (mg kg - 1), without preservation of litchi, longan and other fruits, the sulfur content is far lower than that of the same fruits after sulfur preservation.

Conclusion ICP-AES is a sensitive and accurate method for the determination of sulfur in preserved fruits by microwave digestion with nitric acid-hydrogen peroxide system.