

Determination of aluminum in wheaten food by Microwave Digestion Graphite Furnace Atomic Absorption Spectrometry

Graphite furnace atomic absorption spectrometry (GFAAS) was used to analyze trace aluminum in food with matrix modifier. The method is simple, rapid, accurate, sensitive and stable.

PE800 Graphite Furnace Atomic Absorption Spectrometer, Al Hollow Cathode Lamp, Pyrolysis Coated Graphite Tube, [Microwave Drying Mechanical](#) Digester, Aluminum Standard Reserve Liquid (100ug/ml), Aluminum Standard Use Liquid diluted to 1000ug ug/L with 1.0% Nitric Acid Solution, Nitric Acid, Perchloric Acid, Magnesium Nitrate Solution, Deionized Water.

The wavelength is 257.4nm, the lamp current is 8mA, and the slit is 0.5nm. Measurement mode: concentration, injection volume 10ml. Graphite furnace procedures: drying 50 ~80 ~120 ~40 s, slope temperature and keep 40 s; ashing 1 200 ~8 s, slope temperature and keep 8 s; atomization 2 600 ~3 s; purification 2 500 ~2.5 s.

The crushed sample was dried in an oven at 85 C for 4 hours and 0.5 ml concentrated sulfuric acid was added to the drip. The reaction was mild and then transferred to a microwave digestion apparatus for digestion. The digestion process was the first step of 3 kg/cm², 60 s, the second step of 8 kg/cm², 120 s, the third step of 15 kg/cm², 120 s. After digestion, it is cooled to about 50, the digestion tank is opened, the digestion solution is poured into a 100 ml beaker, the digestion tank is washed with 20 ml deionized water, and then heated to about 10 ml of liquid left in the electric furnace, and the cooling volume is set to 25 ml. At the same time, the reagent is blank.

Aluminum standard solution was diluted with 10% nitric acid solution to prepare 0, 10.0, 20.0, 40.0, 60.0 ug/L aluminum standard series. According to the working conditions of the above instruments, the absorbance is measured and the standard curve is drawn. After digestion, the sample and blank solution were injected into the graphite furnace in turn to determine the absorbance, and the sample results deducted the reagent blank.

Three kinds of aluminium standard solutions with different concentrations were added into the sample to carry out the recovery test and calculate the recovery rate. Relative standard deviation (RSD) of 1.15.21 20 33.63 92.1 Sample 230.72 30 58.86 93.8 Sample 338.54 50 89.74 102.42.2 was obtained from the precision test results of two samples repeated for six times. 6% and 3.6%, the sample pretreatment plays a key role in the accurate determination of aluminum content, in the work found that wet digestion acid consumption is large, digestion time is long, and the digestion effect of oil-rich samples is not good, seriously affecting the determination results; microwave digestion method is simple, time-saving, labor-saving, digestion effect OK.

Through repeated experiments, it is found that the precision of peak shape and result is better at wavelength 257.4nm. The practice of selecting graphite furnace shows that the pyrolytic

coated graphite tube has higher sensitivity and better reproducibility than ordinary stone, and the ordinary graphite tube is easy to produce memory effect, which leads to poor linearity of standard curve. Therefore, the pyrolytic coated graphite tube is adopted in this experiment.

In this paper, a three-step continuous ramp heating method of 50, 80, 120, with sufficient heating time, is adopted to ensure thorough drying and no sudden boiling. The result is accurate and reproducible.

The selection of ashing temperature and the increase of ashing temperature can drive most of the interfering materials before atomization. When ashing temperature is greater than 1400 C, the aluminum begins to lose and the signal decreases. In this paper, the ashing temperature of 1200 C is selected, which has the advantages of small interference, good linearity of standard curve and high recovery. It was found that the pyrolysis coated graphite tube was atomized at 2700 C. After repeated use, the graphite particles would fly out with argon and interfere with the determination. It is found that the peak shape and reproducibility are good when the atomization temperature is between 2400-2700 C.

In this paper, magnesium nitrate is added as matrix modifier. By comparing with no matrix modifier, it is found that adding matrix modifier can stabilize the experimental results and eliminate the interference of many ions. To sum up, this paper uses [Noodle drying equipment](#) for sample processing, the method is convenient, simple and fast, good digestion effect, while reducing the intensity of work. Magnesium nitrate can be used as matrix modifier to eliminate the interference of most ions. Pyrolytic coated graphite tube is selected to further improve the sensitivity and reproducibility of the signal. Satisfactory results are obtained through recovery and precision tests.