

The determination of lead in spinach by AAS coupled microwave digestion system as sample preparation method

1. Introduction

Lead is a toxic heavy metal which would cause extreme harm to human health. When lead and its compounds enter human body, it will cause multiple harm to body system as nerves, hematopoiesis, digestion, kidneys, and cardiovascular etc. The accumulation of lead inside the human body would cause poison and harm to human health. The source of the lead inside vegetable comes from the accumulation or migration from the environment as soil, dust, printing package etc.. So that the determination of lead content inside the food is of great significance to human health. Here we present microwave digestion as simple, fast sample, low risk of contamination for sample preparation. M3 microwave digestion vessel couple with HP 10 rotor ensures an accurate analysis of lead in spinach powder.

2. Instrument and reagent

The digestions were carried out with M3 microwave digestion system and HP 10 high pressure digestion vessels. The determination of the trace element was conducted by AAS.



M3 microwave digestion system



HP10 rotor



G-160 hot block

Reagent: HNO_3 (GR)

Sample: spinach powder quality control sample

3. Method

1. Weigh 0.3 g spinach powder quality control samples into sample cup.
2. Add HNO_3 into the sample cup swirl the cup to mix the sample and acid thoroughly.
3. Add the same amount of HNO_3 into the sample cup as sample blank, then seal the vessel.
4. Set the microwave digestion program as shown in following table:

Table1: Microwave digestion program

Step	Setting temperature(°C)	Ramp time (min)	Temperature holding (min)
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1	140	8	5
2	190	8	20

- Take the vessels out of the cavity when the temperature falls under 60 °C.
- Open the vessels and place them on the hot block to evaporated acid. Dilute the sample to 50 mL with deionized water when the temperature of the vessels cools to room temperature.
- The final solutions were tested by AAS according to the national food standard GB5009.12-2017.

4. Result

Table 2: Founded Pb concentration in spinach certified sample

Sample name	Found value (mg/kg)	Average (mg/kg)	Certified value (mg/kg)	RSD%	Recovery (%)
Spinach powder	10.25	10.3	10.1±0.9	2.18	102
	10.15				
	10.58				

The result shows a good correlation of lead level between found value and certified concentration, which is a proof that the microwave digestion method can provide accurate and reproducible sample preparation process for AAS test.

5. Conclusion

Preekem's M3 microwave digestion system coupled with HP10 rotor can digestion the food as vegetable thoroughly to a clear solution. Thanks to the advanced full vessel real-time temperature monitor and pressure control technique, the digestion unit not only ensures the safe and precise sample digestion but also improves the accuracy and stability during the experiment. The microwave digestion method can also be applied in the determination of other metals inside various vegetable.