



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 5009.4-2016

**National food safety standard - Determination of Ash in Foods
(English Translation)**

Issued on: August 31, 2016

Implemented on: March 1, 2017

**Issued by: National Health and Family Planning Commission of the
People 's Republic of China**

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1 Scope

Method One of this Standard specifies the determination method of ash in foods. Method Two specifies the determination method of water-soluble ash and water-insoluble ash in foods. Method Three specifies the determination method of acid insoluble ash in foods.

Method One of this Standard is applicable to the determination of ash in foods (starch ash method is applicable to starch and modified starch of which the mass fraction is not greater than 2%). Method Two is applicable to the determinations of water-soluble ash and water-insoluble ash in foods. Method

Three is applicable to the determination of acid insoluble ash in foods.

Method One Determination of total ash in foods

2 Principle

The inorganic matter left after the burning of the food is called ash. Ash value is calculated by burning and weighing.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are of analytical grade and water is Grade 3 water specified in GB/T 6682.

3.2.1 Magnesium acetate solution (80 g/L). weigh 8.0 g of magnesium

acetate, add water to dissolve and set volume to 100 mL; well mix.

3.2.3 10% hydrochloric acid solution. weigh 24 mL of concentrated hydrochloric acid of analytical grade; use distilled water to dilute it to 100 mL.

4.1 High temperature furnace. the maximum temperature $\geq 950^{\circ}\text{C}$

4.2 Analysis of balance. resolutions are 0.1 mg, 1 mg, 0.1 g

5.1.1 Foods of high phosphorus content and other foods

Take the appropriate size of the quartz crucible or porcelain crucible. Place it in the high temperature furnace. Burn it at $550^{\circ}\text{C} \pm 25^{\circ}\text{C}$ for 30 min. Cool it to around 200°C . Take it out. Put it into the dryer to cool for 30 min.

5.1.2 Starchy food

Wash with boiling dilute hydrochloric acid at first, then with a lot of tap water, and with distilled water finally. Place the clean crucible in the high temperature furnace. Burn it at $900^{\circ}\text{C} \pm 25^{\circ}\text{C}$ for 30 min.

5.2 Sample weighing

Foods of high phosphorus content and other foods. weigh 2 g ~ 3 g (nearest to 0.0001 g) of sample of which the ash is greater than or equal to 10 g/100 g;

weigh 3 g ~ 10 g (nearest to 0.0001 g; for the sample of less ash content, it can increase in the amount of sample appropriately) of sample of which the ash is less than or equal to 10 g/100 g.

5.3.1 Beans and their products, meat and poultry and their products, eggs

and their products, aquatic products and their products, milk and dairy products of high phosphorus content

5.3.1.1 After weighing sample, add 1.00 mL of magnesium acetate solution

(240 g/L) or 3.00 mL of magnesium acetate solution (80 g/L) to make the sample completely wet. After 10 min of placement, evaporate the water to dryness on the water bath. Heat the sample on a hot plate with a small fire to fully carbonize the sample to smoke-free. Then place it in the high temperature furnace. Burn it at $550^{\circ}\text{C} \pm 25^{\circ}\text{C}$ for 4 h. Cool it to around 200°C .

Take it out.

5.3.1.2 Pipet 3 magnesium acetate solutions of same concentration and

volume with 5.3.1.1. Carry out 3 reagent blank tests. When the standard deviation of 3 test results is less than 0.003 g, take the arithmetic mean as the blank value.

5.3.2 Starchy food

Place the crucible on a high temperature oven or a hot plate. Half-lid the crucible cover. Carefully heat the sample to completely carbonize to smoke-free in a ventilated condition. Immediately place the crucible in a high temperature oven. Raise the temperature to $900^{\circ}\text{C} \pm 25^{\circ}\text{C}$. Keep this temperature until the remaining carbon disappears. Generally, it takes 1 h to complete the ashing. Cool it to around 200°C . Take it out. Put it into the dryer to cool for 30 min. Before weighing, if the burning residues are found to have carbon particles, it shall drip a little water into the sample to moist, loosening the agglomeration. Evaporate the water. Burn it again till there are no carbon particles, which means the ashing is completed. Then it can be weighed.

5.3.3 Other foods

Liquid and semi-solid samples should be evaporated on a boiling water bath.

Heat the solid or evaporated sample in the hot plate with a small fire. Fully carbonize to smoke-free. Then place it in the high temperature furnace. Burn for 4 h at $550^{\circ}\text{C} \pm 25^{\circ}\text{C}$. Cool it around 200°C .

6.1.1 The ash content in sample, the sample added with magnesium

acetate solution, shall be calculated according to equation (1).

6.1.2 The ash content in sample, the sample added without magnesium

acetate solution, shall be calculated according to equation (2).

6.2.1 The ash content in sample added with magnesium acetate solution

shall be calculated according to equation (3).

6.2.2 The ash content in sample added without magnesium acetate solution

shall be calculated according to equation (4).

7 Precision

The absolute difference between the two independent determinations obtained under reproducible conditions shall not exceed 5% of the arithmetic mean.

8 Principle

Extract the total ash with hot water. Filter by ashless filter paper. Burn.

Determine the water-insoluble ash.

9 Reagents and materials

Unless otherwise stated, the water used in this method is Grade 3 water specified in GB/T 6682.

10.1 High temperature furnace. maximum temperature $\geq 950^{\circ}\text{C}$

10.2 Analysis balance. resolutions are 0.1 mg, 1 mg, 0.1 g

10.6 Funnel

10.7 Surface dish. diameter is 6 cm

10.8 Beaker (high). volume is 100 mL

11.1 Crucible pretreatment

See "5.1 Crucible pretreatment".

11.2 Sample weighing

See "5.2 Sample weighing".

11.3 Total ash preparation

See "5.3 Determination"