National Standard of the People's Republic of China

GB 5009.4-2016

National Standards For Food Safety

Determination Of Ash In Food

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Determination of ash in food

1 Scope

The first method of this standard specifies the determination method of ash in food, the second method specifies the determination method of water-soluble ash and water-insoluble ash in food, and the third method specifies the determination method of acid insoluble ash in food.

The first method of this standard is applicable to the determination of ash in food (the method for starch based ash is applicable to starch and modified starch with ash content not exceeding 2% by mass), the second method is applicable to the determination of water-soluble ash and water insoluble ash in food, and the third method is applicable to the determination of acid insoluble ash in food.

The First Method: Determination Of Total Ash In Food

2 Principles

The residual inorganic substances after food is burned are called ash. The ash content value is calculated by burning and weighing.

3 Reagents and Materials

Unless otherwise specified, all reagents used in this method are analytical pure, and the water is Grade III water as specified in GB/T6682

3.1 Reagents

3.1.1 Magnesium acetate [(CH3COO) 2Mg · 4H2O].

3.1.2 Concentrated hydrochloric acid (HCl).

3.2 Reagent preparation

3.2.1 Magnesium acetate solution (80g/L): Weigh 8.0g of magnesium acetate and dissolve it in water to a constant volume of 100mL, and mix well.

3.2.2 Magnesium acetate solution (240g/L): Weigh 24.0g of magnesium acetate and dissolve it in water to a constant volume of 100mL, and mix well.

3.2.3 10% hydrochloric acid solution: Measure 24mL of analytical pure concentrated hydrochloric acid and dilute to 100mL with distilled water.

4 Instruments and equipment

4.1 High temperature furnace: The maximum operating temperature is ≥ 950 ℃.

4.2 Analytical balance: The sensitivity values are 0.1mg, 1mg, and 0.1g, respectively.

4.3 Quartz or porcelain crucibles.

4.4 Drier (with desiccant inside).

4.5 Electric heating plate.

4.6 Constant temperature water bath: temperature control accuracy ± 2 ℃.

5 Analysis steps

5.1 Crucible pretreatment

5.1.1 Foods with high phosphorus content and other foods

Take a quartz or porcelain crucible of appropriate size and place it in a high-temperature furnace. Burn it at 550 ℃± 25 ℃ for 30 minutes, cool it to about 200 ℃, take it out, cool it in a dryer for 30 minutes, and accurately weigh it. Repeated burning until the difference between the two weights does not exceed 0.5mg is considered constant weight.

5.1.2 Starch based foods

Wash with boiling dilute hydrochloric acid first, then rinse with a large amount of tap water, and finally rinse with distilled water. Place the cleaned crucible in a high-temperature furnace, burn it at 900 ℃± 25 ℃ for 30 minutes, and cool it to room temperature in a dryer,accurate to 0.0001g.

5.2 Sample weighing

Foods and other foods with high phosphorus content: Weigh 2g to 3g (accurate to

0.0001g) of samples with ash content greater than or equal to 10g/100g;Weigh 3g~10g of samples with an ash content less than or equal to 10g/100g (accurate to 0.0001g, for samples with lower ash content, the sample weight can be appropriately increased).

Starch food: Quickly weigh 2g to 10g of the sample (at least 5g of potato starch, wheat starch, and rice starch, and 10g of corn starch and cassava starch) accurate to 0.0001g. Distribute the sample evenly in the crucible without compressing it tightly.

5.3 Determination

5.3.1 Beans and their products with high phosphorus content, meat and poultry and their products, eggs and their products, aquatic products and their products, milk and dairy products.

5.3.1.1 After weighing the sample, add 1.00mL magnesium acetate solution (240g/L) or 3.00mL magnesium acetate solution (80g/L) to completely wet the sample.After placing for 10 minutes, evaporate the water in a water bath and heat the sample on an electric heating plate with low heat to fully carbonize it until smokeless. Then, place it in a high-temperature furnace and burn it at 550 ℃± 25 ℃ for 4 hours.Cool to around 200 ℃, take it out, and cool it in a dryer for 30 minutes. If carbon particles are found in the burning residue before weighing, a small amount of water should be dropped into the sample to moisten it, so that the clumps are loose. Evaporate the dry water and burn it again until there are no carbon particles,only after complete ashing can weighing be carried out.Repeated burning until the difference between the two weights before and after weighing does not exceed 0.5mg is considered constant weight.

5.3.1.2 Take three magnesium acetate solutions of the same concentration and volume as 5.3.1.1 and conduct three reagent blank tests. When the standard deviation of the three test results is less than 0.003g, take the arithmetic mean as the blank value. If the standard deviation is greater than or equal to 0.003g, the blank value test should be repeated.

5.3.2 Starch based foods

Place the crucible on a high-temperature furnace or electric heating plate, with a half lid on the crucible cover. Carefully heat the crucible to completely carbonize the sample to smokeless under ventilated conditions. Immediately place the crucible in a high-temperature furnace, raise the temperature to 900 ℃± 25 ℃, and maintain this temperature until all remaining carbon disappears. Generally, ashing can be completed within 1 hour.Cool to around 200 ℃, take it out, and cool it in a dryer for 30 minutes. If carbon particles are found in the burning residue before weighing, a small amount of water should be dropped into the sample to moisten it and loosen the clumps. After evaporating the dry water, burn it again until there are no carbon particles, indicating complete ashing can be weighed.

Repeated burning until the difference between the two weights before and after weighing does not exceed 0.5mg is considered constant weight.

5.3.3 Other foods

Liquid and semi solid samples should be evaporated dry on a boiling water bath first. The solid or evaporated sample is first heated on an electric heating plate with low heat to fully carbonize until smokeless, and then placed in a high-temperature furnace and burned at 550 ℃± 25 ℃ for 4 hours.Cool to around 200 ℃, take it out, and cool it in a dryer for 30 minutes. If carbon particles are found in the burning residue before weighing, a small amount of water should be dropped into the sample to moisten it and loosen the clumps. After evaporating the dry water, burn it again until there are no carbon particles, indicating complete ashing before weighing. Repeated burning until the difference between the two weights does not exceed 0.5mg is considered constant weight.

6 Expression of analysis results

6.1 Based on the mass of the sample

6.1.1 The ash content in the sample, with the addition of magnesium acetate solution, is calculated according to formula (1)

$$X_1 = \frac{m_1 - m_2 - m_0}{m_3 - m_2} \times 100 \qquad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (1)$$

In the formula:

X_1- The ash content in the sample with magnesium acetate solution added, in grams per hundred grams (g/100g);

m_1- Mass of crucible and ash, in grams (g);

m_2- Mass of the crucible, in grams (g);

m_0- mass of magnesium oxide (product of magnesium acetate calcination), in grams (g);

m_3- Mass of crucible and sample, in grams (g);

100- Unit conversion coefficient

6.1.2 The ash content in the sample, calculated according to formula (2) for the sample without adding magnesium acetate solution.

$$X_2 = \frac{m_1 - m_2}{m_3 - m_2} \times 100 \qquad \cdots\cdots (2)$$

In the formula:

X_2- The ash content in the sample without adding magnesium acetate solution, in grams per hundred grams (g/100g);

m_1- Mass of crucible and ash, in grams (g);

m_2- Mass of the crucible, in grams (g);

m_3- Mass of crucible and sample, in grams (g);

100-Unit conversion coefficient

6.2 Calculated by dry matter

6.2.1 Calculate the ash content in the sample containing magnesium acetate solution according to formula (3):

$$X_1 = \frac{m_1 - m_2 - m_0}{(m_3 - m_2) \times \omega} \times 100 \qquad \cdots\cdots (3)$$

In the formula:

X_1- The ash content in the sample with magnesium acetate solution added, in grams per hundred grams (g/100g);

m_1- Mass of crucible and ash, in grams (g);

m_2- Mass of the crucible, in grams (g);

m_0- mass of magnesium oxide (product of magnesium acetate calcination), in grams (g);

m_3- Mass of crucible and sample, in grams (g);

ω - Sample dry matter content (mass fraction),%;

100-Unit conversion coefficient.

6.2.2 Calculate the ash content in the sample without adding magnesium acetate solution according to formula (4):

$$X_2 = \frac{m_1 - m_2}{(m_3 - m_2) \times \omega} \times 100 \qquad \cdots\cdots(4)$$

In the formula:

X_2- The ash content in the sample without adding magnesium acetate solution, in grams per hundred grams (g/100g);

m_1- Mass of crucible and ash, in grams (g);

m_2- Mass of the crucible, in grams (g);

m_3- Mass of crucible and sample, in grams (g);

ω - Sample dry matter content (mass fraction),%;

100-Unit conversion coefficient.

When the ash content in the sample is ≥ 10g/100g, retain three significant digits; When the ash content in the sample is less than 10g/100g, retain two significant digits.

7 Precision

The absolute difference between two independent measurement results obtained under repeatability conditions shall not exceed 5% of the arithmetic mean.

The Second Method: Determination Of Water-Soluble Ash And Water Insoluble Ash In Food

8 Principles

Extract the total ash content with hot water, filter it with ashless filter paper, burn it, and weigh the residue. Measure the water insoluble ash content, and calculate the water soluble ash content based on the difference in mass between the total ash content and the water insoluble ash content.

9 Reagents and Materials

Unless otherwise specified, the water used in this method is Grade III water according to GB/T6682.

10 Instruments and Equipment

10.1 High temperature furnace: The maximum temperature is ≥ 950 ℃.

10.2 Analytical balance: The sensitivity values are 0.1mg, 1mg, and 0.1g, respectively.

10.3 Quartz or porcelain crucibles.

10.4 Desiccator (with desiccant inside).

10.5 Ash free filter paper.

10.6 Funnel.

10.7 Surface vessel: diameter 6cm.

10.8 Beaker (high type): Capacity 100mL.

10.9 Constant temperature water bath pot: temperature control accuracy ± 2 ℃.

11 Analysis steps

11.1 Crucible pretreatment

The method can be found in "5.1 Crucible pretreatment".

11.2 Sample weighing

The method can be found in "5.2 Sample weighing".

11.3 Preparation of Total Ash

The method can be found in "5.3 Determination".

11.4 Determination

Wash the total ash content from the crucible into a 100mL beaker with about 25mL of

hot distilled water, cover it with a surface vessel, and heat it with low heat until slightly boiling to prevent the solution from splashing out.Filter with ashless filter paper while it is hot, and wash the residue in the cup with hot distilled water until the filtrate and washing volume reach about 150mL. Transfer the filter paper and residue into the original crucible, place it on a boiling water bath, carefully evaporate the water, and then dry the crucible and move it into a high-temperature furnace. Burn at 550 ℃± 25 ℃ until there are no carbon particles (usually 1 hour).When the furnace temperature drops to 200 ℃, place it in a dryer, cool to room temperature, and weigh it(accurate to 0.0001g).Then place it in a high-temperature furnace and burn it at 550 ℃± 25 ℃ for 30 minutes. Cool and weigh it as before. Repeat the operation until the difference between two consecutive weighings does not exceed 0.5mg, and record the lowest mass.

12 Expression of analysis results

12.1 Calculated by Sample Mass

12.1.1 The content of water insoluble ash is calculated according to formula (5):

$$X_1 = \frac{m_1 - m_2}{m_3 - m_2} \times 100 \qquad \cdots\cdots(5)$$

In the formula:

X_1- Content of water insoluble ash, in grams per hundred grams (g/100g);

m_1- Mass of crucible and water insoluble ash, in grams (g);

m_2- Mass of the crucible, in grams (g);

m_3- Mass of crucible and sample, in grams (g);

101-Unit conversion coefficient

12.1.2 The content of water-soluble ash, calculated according to formula(6):

$$X_2 = \frac{m_4 - m_5}{m_0} \times 100 \qquad \cdots\cdots(6)$$

In the formula:

X_2- Mass of water-soluble ash, in grams (g/100g);

m_0- Mass of the sample, in grams (g);

m_4- Mass of total ash, in grams (g);

m_5- Mass of water insoluble ash, in grams (g);

100- Unit conversion coefficient.

12.2 Calculated by dry matter

12.2.1 The content of water insoluble ash is calculated according to formula (7):

$$X_1 = \frac{m_1 - m_2}{(m_3 - m_2) \times \omega} \times 100 \qquad \cdots\cdots(7)$$

In the formula:

X_1- Content of water insoluble ash, in grams per hundred grams (g/100g);

m_1- Mass of crucible and water insoluble ash, in grams (g);

m_2- Mass of the crucible, in grams (g);

m_3- Mass of crucible and sample, in grams (g);

ω ——— Sample dry matter content (mass fraction),%;

100- Unit conversion coefficient.

12.2.2 The content of water-soluble ash is calculated according to formula(8):

$$X_2 = \frac{m_4 - m_5}{m_0 \times \omega} \times 100 \qquad \cdots\cdots(8)$$

In the formula:

X_2- Mass of water-soluble ash, in grams (g/100g);

m_0- Mass of the sample, in grams (g);

m_4- Mass of total ash, in grams (g);

m_5- Mass of water insoluble ash, in grams (g);

ω - Sample dry matter content (mass fraction),%;

100- Unit conversion coefficient

When the ash content in the sample is ≥ 10g/100g, retain three significant digits; When the ash content in the sample is less than 10g/100g, retain two significant digits.

13 Precision

The absolute difference between two independent measurement results obtained under repeatability conditions shall not exceed 5% of the arithmetic mean.

The Third Method:Determination Of Acid Insoluble Ash In Food

14 Principles

Treat the total ash with hydrochloric acid solution, filter, burn, and weigh the residue.

15 Reagents and Materials

Unless otherwise specified, all reagents used in this method are analytical pure, and the water is Grade III water as specified in GB/T6682.

15.1 Reagents

Concentrated hydrochloric acid (HCl).

15.2 Reagent Preparation

Dilute 24 mL of analytical pure concentrated hydrochloric acid in 10% hydrochloric acid solution with distilled water to 100 mL.

16 Instruments and equipment

16.1 High temperature furnace: maximum temperature ≥ 950 ℃.

16.2 Analytical balance: The sensitivity values are 0.1mg, 1mg, and 0.1g, respectively.

16.3 Quartz or porcelain crucibles.

16.4 Desiccator (with desiccant inside).

16.5 Ash free filter paper.

16.6 Funnel.

16.7 Surface vessel: diameter 6cm.

16.8 Beaker (high type): Capacity 100mL.

16.9 Constant temperature water bath pot: temperature control accuracy ± 2 ℃.

17 Analysis steps

17.1 Crucible pretreatment

The method can be found in "5.1 Crucible pretreatment".

17.2 Sample weighing

The method can be found in "5.2 Sample weighing".

17.3 Preparation of total ash

The method can be found in "5.3 Determination".

17.4 Determination

Wash the total ash into a 100mL beaker in batches with a 25mL 10% hydrochloric acid solution, cover with a watch glass, and carefully heat on a boiling water bath until the solution changes from turbid to transparent. Continue heating for 5 minutes, filter with ashless filter paper while hot, and repeatedly wash the residue on the beaker and filter paper with a small amount of boiling distilled water until neutral (about 150mL).Transfer the filter paper and residue into the original crucible, carefully evaporate the water on a boiling water bath, transfer to a high-temperature furnace, and burn at 550 ℃± 25 ℃ until there are no carbon particles (usually 1 hour).When the furnace temperature drops to 200 ℃, take out the crucible, place it in a dryer, cool it to room temperature, and weigh it(accurate to 0.0001g).Then place it in a high-temperature furnace and burn it at 550 ℃ ± 25 ℃ for 30 minutes. Cool and weigh it as before. Repeat the operation until the difference between two consecutive weighings does not exceed 0.5mg, and record the lowest mass.

18 Expression of analysis results

18.1 Calculate the content of acid insoluble ash based on the mass of the sample using formula (9):

$$X_1 = \frac{m_1 - m_2}{m_3 - m_2} \times 100 \qquad \cdots\cdots (9)$$

In the formula:

X_1- Content of acid insoluble ash, in grams per hundred grams (g/100g);

m_1- Mass of crucible and acid insoluble ash, in grams (g);

m_2- Mass of the crucible, in grams (g);

m_3- Mass of crucible and sample, in grams (g);

101- Unit conversion coefficient.

18.2 Calculate the content of acid insoluble ash based on dry matter using formula (10):

$$X_1 = \frac{m_1 - m_2}{(m_3 - m_2) \times \omega} \times 100 \qquad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (10)$$

In the equation:

X_1- Content of acid insoluble ash, in grams per hundred grams (g/100g);

m_1- Mass of crucible and acid insoluble ash, in grams (g);

m_2- Mass of the crucible, in grams (g);

m_3- Mass of crucible and sample, in grams (g);

ω -Sample dry matter content (mass fraction),%;

100- Unit conversion coefficient.

When the ash content in the sample is ≥ 10g/100g, retain three significant digits; When the ash content in the sample is less than 10g/100g, retain two significant digits.

19 Precision

The absolute difference between the measurement results obtained from the same sample under repeatability conditions shall not exceed 5% of the arithmetic mean.