

National Food Safety Standard of P. R. Of China

GB 5009.3—2010

**National Food Safety Standard
Determination of Moisture in Foods**

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Preface

This standard replaces GB/T 5009.3-2003 *Determination of Moisture in Foods* and GB/T 14769-1993 *Method for Determination of Moisture in Foods*.

The major changes in this standard compared with GB/T 5009.3-2003 are as following:

- Add Karl-Fischer Method as " Method IV";
- Revise the temperature range of direct drying method;
- Clarify the units used for Method I and Method II;
- Revise the application scope of hypobaric drying method.

The previous edition replaced by this standard:

- GB/T 5009.3-1985、GB/T 5009.3-2003;
- GB/T 14769-1993.

National Food Safety Standard

Determination of Moisture in Foods

1 Scope

This standard specifies the method for determination of moisture in foods.

Direct drying method in this standard is applicable to determine the moisture at 101 - 105 degree for products which do not contain volatile or slightly volatile substances such as grain and its products, aquatic products, soy products, dairy products, meat, stewed meat etc . It is not applicable to the sample with moisture content of less than 0.5g/100g.

Hypobaric drying method is applicable to determine the moisture for labile products such as sugar and monosodium glutamate. It is not applicable for candies consisting of other ingredients, such as milk candy and softgum candy. Meanwhile, this method is not applicable to the sample with moisture content of less than 0.5g/100g.

Distillation method is applicable to determine the moisture for products with volatile substances, such as fat and spices. It is not applicable to the sample with moisture content of less than 1g/100g.

Karl-Fischer Method is applicable to determine the moisture of various foods. Karl-Fischer volumetric method is applicable to the sample with moisture content higher than 1.0×10^{-3} g/100g and Karl Fischer coulometry Method is applicable to the sample with moisture content of higher than 1.0×10^{-5} g/100g.

Method I Direct Drying Method

2 Principle

Utilize the physical properties of the moisture in food to adopt volatilization method to determine weight loss of the sample drying under 101.3kPa (1 atm) at 101 °C ~ 105 °C, including hydration moisture, part of crystallization moisture and substances that can volatilize under these conditions. Calculate the moisture content using weighted values before and after drying.

3 Reagents and Materials

Unless otherwise noted, all the reagents used in this method are analytically purity

3.1 HCl (HCl): top-grade purity

3.2 NaOH (NaOH): top-grade purity.

3.3 HCl solution (6mol/L): Weigh 50mL HCl, dilute with water to 100mL.

3.4 NaOH solution (6mol/L): Weigh 24g NaOH, dissolve in water and dilute to 100mL.

3.5 Sea sand: Wash sea/river sand with water to remove the mud. Firstly boil the sand with HCl (as prepared 3.3) for 0.5h and then neutralize with water. Then boil the sand with

NaOH (as prepared 3.4) for 0.5h, neutralize with water and dry under 105°C for use.

4 Equipment and apparatus

- 4.1 Flat aluminum or glass weighing bottle.
 4.2 Electrothermal constant-temperature drying oven.
 4.3 Desiccator: charged with efficient desiccant.
 4.4 Analytical balance: precision 0.1mg.

5 Analysis Procedure

5.1 Solid sample: Take flat aluminum or glass weighing bottle, put it into a drying oven (101°C~105°C), diagonally support the lid on the edge of bottle, heat for 1.0h, take it out and cover the lid. Cool the bottle in the desiccator for 0.5h before weighing. Repeat drying until the mass difference of two consecutive weighing is less than 2mg, which is constant weight. Immediately grind the uniformly mixed sample into particles less than 2mm. Cut the sample that is hard to grind as fine as possible. Weigh 2g~10g sample (precision to 0.0001g) and put it into the weighing bottle. Sample layer should not be more than 5 mm in height. When it is loose sample, the layer should not exceed 10 mm. Cover the lid, accurately weigh the bottle, put it into the drying oven (101°C~105°C), lean the lid against bottle, dry for 2h~4h, cover the lid and take the bottle out, put it into the desiccator, cool it for 0.5h and weigh. Put the bottle back into drying oven (101°C~105°C), dry for 1h, take it out, put into the desiccator, cool it for 0.5h and weigh. If the mass difference between the two measurements does not exceed 2mg, it can be recorded as constant weight.

Note: Use the last measured value from the two values of constant weight in the final calculation.

5.2 Semi-solid or liquid sample: Take a clean weighing bottle (contain 10g sea sand and a small glass rod), put it into drying oven (101°C~105°C), dry for 0.5 h~1.0 h, take it out, put into the desiccator, cool for 0.5h, weigh the bottle and repeatedly dry it to constant weight. Then weigh 5g~10g sample (precision to 0.0001g), put it into the evaporating dish, stir well with small glass rod, boil and dry in boiling water bath with agitation. Wipe off the water droplets on the bottom of evaporating dish, put it into drying oven (101°C~105°C), dry for 4h, cover the lid, take the bottle out, put it into the desiccator, cool for 0.5h and weigh. Follow the instruction as that in 5.1 starting "Put the bottle back into drying oven (101°C~105°C), dry for 1h..."

6 Calculation and Expression of the Results

Moisture content in the sample is calculated in accordance with formula (1).

$$X = \frac{m_1 - m_2}{m_1 - m_3} \times 100 \quad \dots\dots\dots (1)$$

WHERE:

X —THE MOISTURE CONTENT IN THE SAMPLE, THE UNIT IS G/100G;

m_1 —The mass of weighing bottle (include sea sand and glass rod) and sample, the unit is g;

m_2 ——The mass of weighing bottle (include sea sand and glass rod) and dried sample, the unit is g;

m_3 ——The mass of weighing bottle (include sea sand and glass rod), the unit is g.

When moisture content $\geq 1\text{g}/100\text{g}$, keep 3 significant digits for the calculation result;
When moisture content $< 1\text{g}/100\text{g}$, keep 2 significant digits for the calculation result.

7 Precision

The difference between the absolute values of the two independent determination results obtained under the repeatability condition must not exceed 5% of the average arithmetic value.

Method II Hypobaric Drying Method

8 Principle

Utilize physical property of the moisture in food, heat to $60^\circ\text{C} \pm 5^\circ\text{C}$ after pressure has reached to 40kPa ~ 53kPa, remove moisture in the sample with vacuum drying method. Then calculate the moisture content according to weighed values before and after drying.

9 Equipment and apparatus

- 9.1 vacuum drying oven.
- 9.2 Flat aluminum or glass weighing bottle.
- 9.3 Desiccator (charged with efficient desiccant).
- 9.4 Analytical balance (Precision 0.1mg).

10 Analysis Procedure

10.1 sample preparation: For powder and crystal samples, weigh directly. For larger blocks of hard sugar, grind in mortar and blend for use.

10.2 Determination: Take 2g~10 g (precision to 0.0001g) sample into a weighing bottle with constant weight, put it into vacuum drying oven, connect the drying oven with vacuum pump to pump the air out of drying oven until reaching the desired pressure (typically 40 kPa ~ 53 kPa). Meanwhile, heat to the required temperature ($60^\circ\text{C} \pm 5^\circ\text{C}$), close the piston on the vacuum pump and stop pumping to maintain certain temperature and pressure in the drying oven. After 4h, open the piston to make the air slowly pass through drying device into the drying oven, open it when the pressure returns to normal state. Take the weighing bottle out, put it into the desiccator for 0.5h and weigh. Repeat above operation until the difference between two masses is within 2mg, which is the constant weight.

11 Calculation and Expression of the Results: The same as 6.

12 Precision

The difference between the absolute values of the two independent determination results obtained under the repeatability condition must not exceed 10% of the average arithmetic value.

Method III Distillation Method

13 Principle

Utilize the physical and chemical properties of moisture in food, vaporize the moisture and toluene or xylene by water determination apparatus. Then calculate the moisture content in the sample according to the volume of received water. This method is applicable to foods containing more volatile substances, such as fats and spices.

14 Reagent and Material

toluene or xylene: (chemical purity). Take toluene or xylene, saturate it with water. Then remove the water layer, conduct distillation and collect distillate for use.

15 Equipment and apparatus

Water determination apparatus: As shown in Figure 1 (with adjustable electrothermal case). Volume of water receiving tube is 5 mL. Its minimum scale value is 0.1 mL and volume error is less than 0.1 mL.

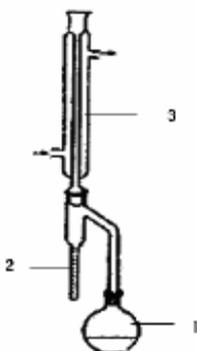


Figure 1 Water Determination Apparatus

1. 250 mL distillation bottle;
2. Water receiving tube with scale;
3. Condensation tube.

16 Analysis Procedure

Accurately weigh proper quantity of sample (the volume of vaporized water should reach 2~ 5 mL but the maximum sampling volume should not exceed 2/3 of the distillation bottle). Put the sample into a 250 mL conical flask, add 75 mL newly-distilled toluene (or xylene), connect the flask with condensation tube and water receiving tube, inject toluene from the top of condensation tube and fill the water receiving tube.

Heat slowly to obtain two drops of distillate per second. When most of the moisture has been vaporized, accelerate distillation to 4 drops per second. When the moisture is completely vaporized and volume of moisture in the receiving tube doesn't increase, inject toluene from the top of condensation tube for washing. If water drops adhered on the wall of condensation tube, they can be wiped by using copper wire with small rubber head. Then distill for a moment until no water drop adhered on the upper part of the receiving tube as well as the wall of condensation tube. If the water level of the receiving tube keeps constant for 10 min, then it is taken as the distillation end point. Read the volume of water layer in the receiving tube.

17 Calculation and Expression of the Results

moisture content in the sample is calculated in accordance with formula (2).

$$X = \frac{V}{m} \times 100 \dots\dots\dots (2)$$

Where:

X—The moisture content in the sample. The unit is mL/100 g (Or mass should be calculated according to the water density of 0.998 at 20 °C; 20 g/mL).

V—The water volume in the receiving tube. The unit is mL;

m—Sample mass. The unit is g. 3 significant digits should be kept for the calculation result.

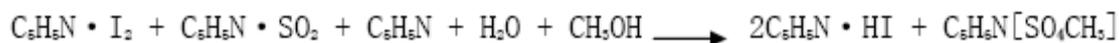
18 Precision

The difference between the absolute values of the two independent determination results obtained under the repeatability condition must not exceed 10% of the average arithmetic value.

Method IV Karl Fischer Method

19 Principle

According to chemical reaction of iodine with sulfur dioxide and water, 1mol iodine only reacts with 1mol water with the co-existence of pyridine and methanol. The reaction formula is as following:



Karl Fischer moisture measurement method can be divided into the coulometric method and the volumetric method. According to the coulometric method, iodine for determination is generated through chemical reaction and can react with water (1: 1) in accordance with reaction formula as long as there is water in the electrolyte. When the water is completely involved in chemical reaction, excessive iodine forms on the anode area of the electrode and reaction is finished. According to the volumetric method, iodine for determination is added as the titrant. The concentration of iodine in the titrant is known. Based on the volume of the consumed titrant and the consumption of iodine, the moisture is calculated.

20 Reagent and Material

20.1 Karl Fischer Reagent.

20.2 Anhydrous methanol (CH₄O): Top-grade pure.

21 Equipment and apparatus

21.1 Karl Fischer moisture determinator

21.2 Balance (precision to 0.1 mg)

22 Analysis procedure

22.1 Calibration for Karl Fischer Reagent (volumetric method)

Add a certain volume of methanol in the reaction bottle (immerse platinum electrode),

titrate to the endpoint with Karl Fischer Reagent by stirring, add 10mg water (precision to 0.0001g), titrate to the endpoint and record the consumption of Karl Fischer Reagent (V). Titer of Karl Fischer Reagent is calculated in accordance with formula (3):

$$T = \frac{M}{V} \dots\dots\dots (3)$$

Where:

T—The titer of Karl Fischer Reagent. The unit is mg/ mL;

M—Water mass. The unit is mg;

V—The volume of Karl Fischer Reagent consumed to titrate the water. The unit is mL.

22.2 Pre-treatment for sample.

Crushable solid sample should be crushed as fine as possible to make it uniform. For the sample difficult to crush, it can be cut.

22.3 Determination of moisture in the sample

add a certain volume of methanol or solvent, specified by Karl Fischer determinator, in the reaction bottle to immerse platinum electrode, titrate to the endpoint with Karl Fischer Reagent by stirring and promptly add sample, which is easily soluble in above solvent, directly into the titration cup. If the sample is difficult to solve, heat the titration cup or add other solvent with determined moisture for assisting solution, and then titrate to the endpoint with Karl Fischer Reagent. It is suggested that water content in the sample should be higher than 10 μ g when coulometric method is used for determination, while water content should be higher than 100 μ g for volumetric method. Meanwhile, its drift amount should be deducted.

22.4 Determination of the drift amount

Add solvent, which is consistent with sample for determination, into the titration cup, titrate to the endpoint, settle for at least 10min and titrate to the endpoint again. volume change per unit time between two titrations is the drift amount (D).

23 Calculation and Expression of the Results

The moisture content in the solid sample is calculated in accordance with formula (4), while moisture content in the liquid sample is calculated in accordance with formula (5).

$$X = \frac{(V_1 - D \times t) \times T}{M} \times 100 \dots\dots\dots (4)$$

$$X = \frac{(V_1 - D \times t) \times T}{V_2 \rho} \times 100 \dots\dots\dots (5)$$

Where:

X— The moisture content in the sample. The unit is g /100g;

V₁—The volume of Karl Fischer Reagent to titrate the sample. The unit is mL.;

T— The accurate value of titer of Karl Fischer Reagent. The unit is g/ mL;

M ——The value of sample mass. The unit is g;

V_2 ——The value of liquid sample volume. The unit is mL.;

D ——Drift amount. The unit is mL/min;

T ——Titration time. The unit is min;

ρ ——The density of liquid sample. The unit is g/ mL.

When moisture content $\geq 1\text{g}/100\text{g}$, 3 significant digits should be reserved for the calculation result; When moisture content $< 1\text{g}/100\text{g}$, 2 significant digits should be reserved for the calculation result.

24 Precision

The difference between the absolute values of the two independent determination results obtained under the repeatability condition must not exceed 10% of the average arithmetic value.
