



National Standard of the People's Republic of China

GB 5009.3-2016

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

食品安全国家标准

食品中水分的测定

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Preface

This standard replaces GB 5009.3-2010 “National food safety standard Determination of moisture in foods”, GB/T 12087-2008 “Starch-Determination of moisture content-Oven-drying method”, GB/T 18798.3-2008 “Instant tea in solid form-Part 3: Determination of moisture content”, GB/T 21305-2007 “Cereals and cereal products-Determination of moisture content-Routine reference method”, GB/T 5497-1985 “Inspection of grain and oilseeds Methods for determination of moisture content”, GB/T 8304-2013 “Tea-Determination of moisture content”, GB/T 12729.6-2008 “Spices and condiments-Determination of moisture content (entrainment method)”, GB/T 9695.15-2008 “Meat and meat products-Determination of moisture content”, GB/T 8858-1988 “Method for determination of dry matter and moisture content in fruit and vegetable products” and SN/T 0919-2000 “Method for the determination of moisture of tea for import and export”.

Compared with GB 5009.3-2010, the major changes of this standard are as follows:

— the application scopes of “Method I Direct Drying Method”, “Method II Vacuum Drying Method”, “Method III Distillation Method” and “Method IV Karl-Fischer Method” have been revised;

— the Reagents and Materials, Precision, Note and Analysis Procedures in Method I Direct Drying Method have been revised;

— the Analysis Procedures of Method III Distillation Method has been revised;

— the Description of Karl-Fischer Coulomb’s Law in Method IV Karl-Fischer Method has been deleted.

National Food Safety Standard Determination of Moisture in Foods

1. Scope

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

Method I (Direct Drying Method) in this standard is applicable to the determination of moisture in vegetable, cereal and its products, aquatic products, soy products, dairy products, meat products, braised vegetables, grain (with moisture content of less than 18%), oilseeds (with moisture content of less than 13%), starch and tea *etc.* under the temperature between 101 °C and 105 °C, and is not applicable to the sample with moisture content less than 0.5 g/100 g. Method II (Vacuum Drying Method) is applicable to the determination of moisture in the labile and watery samples (such as sugar and monosodium glutamate, *etc.*) and is not applicable to the determination of candies with addition of other ingredients (such as milk candy and soft candy, *etc.*); meanwhile, this method is not applicable to the sample with moisture content less than 0.5 g/100 g (excluding sugar and monosodium glutamate). Method III (Distillation Method) is applicable to the determination of moisture in the foods containing more water and volatile substances, including fruits, spices and condiments, meat and meat products, *etc.* and is not applicable to the sample with moisture content less than 1 g/100 g. Method IV (Karl-Fischer Method) is applicable to the determination of trace amount of water in foods, and is not applicable to the determination of moisture in such foods as oxidant, reducing agent, alkaline oxide, hydroxide, carbonate, boric acid, *etc.* Volumetric Karl-Fischer Method is applicable to the sample with moisture content higher than 1.0×10^{-3} g/100 g.

Method I Direct Drying Method

2. Principles

According to the physical property of the moisture in food and under the conditions of 101.3 kPa (1 atm) and 101 °C~105 °C, the weight loss on drying,

including hygroscopic moisture, part of crystal water and the substances that can volatilize under these conditions, is determined by adopting volatilization method, and the moisture content is calculated in accordance with the weighing values before and after drying.

3. Reagents and Materials

Unless otherwise stated, all the reagents used in this method are analytically pure, and the water used is Grade 3 water as specified in GB/T 6682.

3.1 Reagents

3.1.1 Sodium hydroxide (NaOH).

3.1.2 Hydrochloric acid (HCl).

3.1.3 Sea sand.

3.2 Reagent preparation

3.2.1 Hydrochloric acid solution (6 mol/L): measure 50 mL of hydrochloric acid, dilute with water to 100 mL.

3.2.2 Sodium hydroxide solution (6 mol/L): weigh 24 g of sodium hydroxide, add water to dissolve it and dilute to 100 mL.

3.2.3 Sea sand: take sea sand, river sand, quartz sand or the analogue thereof, which has been washed with water to remove the mud, boil it with hydrochloric acid (6 mol/L) for 0.5 h firstly, wash it with water until neutral, boil it with sodium hydroxide solution (6 mol/L) for 0.5 h, wash it again with water until neutral, and dry it under 105 °C for use later.

4. Apparatus and Equipment

4.1 Flat aluminum or glass weighing bottle.

4.2 Electrothermal constant-temperature drying oven.

4.3 Dryer: with efficient drying agent inside.

4.4 Balance: with sensitivity of 0.1 mg.

5. Analysis Procedures

5.1 Solid test sample: Take a clean flat aluminum or glass weighing bottle, put it into a drying oven (101 °C~105 °C), lean the cap against the edge of bottle, heat for 1.0 h, take it out and cover the cap. Place the bottle into the dryer, cool for 0.5 h and weigh it. Repeat drying until the mass difference of two consecutive weighings is not more than 2 mg, which is to achieve constant weight. Immediately grind the well-mixed test sample into particles less than 2 mm. Cut the sample that is hard to be grinded as finely as possible. Weigh 2 g~10 g of the test sample (accurate to 0.0001 g) and put it into the weighing bottle which has been pre-dried to a constant weight. The test sample layer shall not be more than 5 mm (10 mm for loose test sample) in thickness. Cover the cap, accurately weigh the bottle, put it into the drying oven (101 °C~105 °C), lean the cap against the edge of the bottle, dry for 2 h~4 h, cover the cap and take the bottle out, put it into the dryer, cool it for 0.5 h and weigh it. Put the bottle back into drying oven (101 °C~105 °C), dry for 1 h, take it out and put into the dryer, cool it for 0.5 h and weigh it again. Repeat the above operation procedure until the mass difference between the two consecutive weighings is not more than 2 mg, which is to achieve a constant weight.

Notes: The smaller weighing value of the two values of constant weight shall be taken for the final calculation.

5.2 Semi-solid or liquid test sample: Take a clean weighing bottle, add 10 g of sea sand (more sea sand can be added as required during the experiment), place a small glass rod in the bottle and put it into the drying oven (101 °C~105 °C), dry for 1.0 h, take it out, put into the dryer, cool for 0.5 h, weigh the bottle and repeat drying it to constant weight. Then weigh 5 g~10 g of the test sample (accurate to 0.0001 g), put it into an weighing bottle, stir well with the small glass rod, evaporate to dryness on a boiling water bath with stirring. Wipe off the water

droplets on the bottom of bottle, put it into the drying oven (101 °C~105 °C), dry for 4 h, cover the cap, take the bottle out, put it into the dryer, cool for 0.5 h and weigh it. Once again, put the bottle back into drying oven (101 °C~105 °C), dry for 1 h, take it out and put into the dryer, cool it for 0.5 h and weigh it again. Repeat the above operation procedure until the mass difference between the two consecutive weighings is not more than 2 mg, which is to achieve a constant weight.

6. Expression of Analysis Results

The moisture content in the test sample is calculated in accordance with Formula (1):

$$\text{—————} \quad (1)$$

Where,

X —the moisture content in the test sample, g/100 g;

m_1 —the mass of weighing bottle (including sea sand and glass rod) and test sample, g;

m_2 —the mass of weighing bottle (including sea sand and glass rod) and test sample after drying, g;

m_3 —the mass of weighing bottle (including sea sand and glass rod), g;

100—the unit conversion factor.

In case of moisture content higher than or equal to 1 g/100g, the calculated result shall keep three significant figures; in case of moisture content lower than 1 g/100g, the calculated result shall keep two significant figures.

7. Precision

The absolute difference between the two independent determination results obtained under the repeatability conditions shall not exceed 10% of their arithmetic mean value.

Method II Vacuum Drying Method

8. Principles

According to the physical property of the moisture in food, the test sample is heated to $60\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ after the pressure reaches 40 kPa~53 kPa, the moisture in the test sample is removed by adopting vacuum drying method, and then the moisture content is calculated according to weighing values before and after drying.

9. Apparatus and Equipment

9.1 Flat aluminum or glass weighing bottle.

9.2 Vacuum drying oven.

9.3 Dryer: with efficient drying agent inside.

9.4 Balance: with sensitivity of 0.1 mg.

10. Analysis Procedures

10.1 Preparation of test sample: For powder and crystal test samples, weigh it directly. For larger blocks of hard sugar, grind in mortar and mix well for use later.

10.2 Determination: Take 2 g~10 g (accurate to 0.0001 g) of the test sample into a weighing bottle pre-dried to constant weight, put it into vacuum drying oven, and 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 ($60\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$). Close the piston on the vacuum pump and stop pumping to maintain a certain temperature and pressure in the drying oven. After 4 h, open the piston, make the air slowly pass through drying device into the vacuum drying oven, and open it when the pressure returns to normal state. Take the weighing bottle out, put it into the dryer for 0.5 h and weigh it. Repeat the above operation procedure until the mass difference between the two consecutive weighings is not more than 2 mg, which is to achieve a constant weight.

11. Expression of Analysis Results

The same as specified in Section 6.

12. Precision

The absolute difference between the two independent determination results obtained under the repeatability conditions shall not exceed 10% of their arithmetic mean value.

Method III Distillation Method

13. Principles

According to the physicochemical property of the moisture in food, the moisture and toluene or xylene in the food is evaporated by adopting moisture content analyzer, and then the moisture content in the test sample is calculated according to the volume of water received. This method is applicable to foods containing more other volatile substances, such as spices, *etc.*

14. Reagents and Materials

Unless otherwise stated, all reagents used in this method are analytically pure, and the water used is Grade 3 water specified in GB/T 6682.

14.1 Reagents

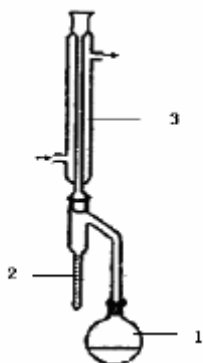
Toluene (C₇H₈) or xylene (C₈H₁₀).

14.2 Reagents preparation

Preparation of toluene or xylene: Take toluene or xylene, saturate it with water, remove the water layer, conduct distillation and collect the distillate for use later.

15. Apparatus and Equipment

15.1 Moisture content analyzer: As shown in Figure 1 (with adjustable electrothermal mantle). The capacity of moisture receiving tube is 5 mL, with a minimum scale value of 0.1 mL and a capacity error of less than 0.1 mL.



Notes:

1—250mL distillation flask;

2—Moisture receiving tube, with scale;

3—Condensation tube.

Figure 1 Moisture content analyzer

15.2 Balance: with sensitivity of 0.1 mg.

16. Analysis Procedures

Accurately weigh appropriate amount of the test sample (it shall ensure the water evaporated within the range of 2 mL~5 mL, but the maximum sampling amount shall not exceed 2/3 of the distillation flask), put it into a 250mL distillation flask, add 75 mL of freshly-distilled toluene (or xylene), connect the condensation tube and the moisture receiving tube, inject toluene from the top of condensation tube and fill up the water receiving tube. At the same time, perform the reagent blank test for toluene (or xylene).

Heat it slowly to maintain two drops of distillate per second. When most of the moisture is evaporated, accelerate distillation to achieve 4 drops per second. When the moisture is completely evaporated and volume of moisture in the receiving tube no longer increases, add toluene from the top of condensation tube to rinse the tube. If water drops adhere to the wall of condensation tube, wipe down using a copper wire with a small rubber head. Then distill for a moment until no water drops adhere to 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. Expression of Analysis Results

The moisture content in the test sample is calculated in accordance with Formula (2):

$$\text{—————} \quad (2)$$

X —the moisture content in the test sample, mL/100g (or calculate the mass according to the water density of 0.998 at 20 °C and 20 g/mL);

V —the volume of the water in the receiving tube, mL;

V_0 —the volume of the water in the receiving tube when performing the reagent blank test, mL;

m —the mass of test sample, g;

100— the unit conversion factor.

The result shall be expressed as the arithmetic mean value of two independent determination results obtained under the repeatability conditions and keep three significant figures.

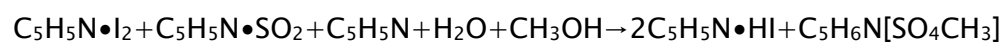
18. Precision

The absolute difference between the two independent determination results, obtained under the repeatability conditions, shall not exceed 10% of their arithmetic mean value.

Method IV Karl–Fischer Method

19. Principles

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



Karl–Fischer moisture determination method can be divided into coulometric

method and volumetric method. For the volumetric method, iodine is added as the titrant for determination, and the concentration of iodine in the titrant is known. Based on the volume of the consumed titrant, the consumed amount of iodine is calculated, thus the moisture content is calculated.

20. Reagents and Materials

20.1 Karl–Fischer reagent.

20.2 Anhydrous methanol (CH₄O): guaranteed reagent.

21. Apparatus and Equipment

21.1 Karl–Fischer moisture content analyzer.

21.2 Balance: with sensitivity of 0.1 mg.

22. Analysis Procedures

22.1 Calibration of Karl–Fischer reagent (volumetric method)

Add a certain volume of methanol into the reaction bottle (to immerse the platinum electrode), titrate to the end point with Karl–Fischer reagent while stirring. Add 10 mg of water (accurate to 0.0001 g), titrate to the end point and record the consumed amount of Karl–Fischer reagent (V). The titer of Karl–Fischer reagent is calculated in accordance with Formula (3):

$$— \qquad \qquad \qquad (X)$$

Where,

T —the titer of Karl–Fischer reagent, mg/mL;

m —the mass of moisture, mg;

V —the volume of Karl–Fischer reagent consumed by titration of moisture, mL.

22.2 Pretreatment of test sample

Crushable solid test sample shall be crushed as finely as possible to make it

uniform. The test sample difficult to be crushed can be cut up.

22.3 Determination of moisture in the test sample

Add a certain volume of methanol or solvent specified by Karl–Fischer analyzer into the reaction bottle to immerse platinum electrode, titrate to the end point with Karl–Fischer reagent while stirring. Promptly add the test sample easily soluble in the above–mentioned methanol or solvent directly into the titration cup. If the test sample is difficult to dissolve, heat the titration cup or add other solvent with determined moisture to assist dissolving, and then titrate to the end point with Karl–Fischer reagent. It is suggested that moisture content in the test sample shall be higher than 100 µg when the volumetric method is used for determination. For the test sample requiring long time to titrate and easily leading to drift, its drift amount shall be subtracted.

22.4 Determination of the drift amount

Add the solvent consistent with that used for sample determination into the titration cup, titrate to the end point, stand for at least 10 min and titrate to the end point again. The volume change per unit time between two titrations is the drift amount (*D*).

23. Expression of Analysis Results

The moisture content in the solid test sample is calculated in accordance with Formula (4), while the moisture content in the liquid test sample is calculated in accordance with Formula (5):

$$\frac{(V \quad t)}{\quad} \quad (4)$$

$$\frac{(V \quad t)}{\quad} \quad (5)$$

Where,

X—the moisture content in the test sample, g/100 g;

*V*₁—the volume of Karl–Fischer reagent consumed by titration of the sample, mL;

D—the drift amount, mL/min;

t —the time consumed for titration, min;
 T —the titer of Karl–Fischer reagent, g/mL;
 m —the mass of sample, g;
100— the unit conversion factor;
 V_2 —the volume of liquid sample, mL;
 ρ —the density of liquid sample, g/mL.

In case of the moisture content higher than or equal to 1 g/100g, the calculated result shall keep three significant figures; in case of the moisture content lower than 1 g/100g, the calculated result shall keep two significant figures.

24. Precision

The absolute difference between the two independent determination results obtained under the repeatability conditions shall not exceed 10% of their arithmetic mean value.