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GB

NATIONAL STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 5009.7-2008

Replacing GB/T 5009.7-2003

Determination of reducing sugar in foods

食品中还原糖的测定

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Standardization Administration of the People's Republic of China

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Foreword

This Standard replaces GB/T 5009.7-2003 of *Determination of reducing sugar in foods*.

Compared with GB/T 5009.7-2003, main changes of this Standard are as follow:

- The detection limits are included;
- The categories of food samples are redefined;
- The anti-titration formula of the first method "Direct Titration" is included;
- The calculated significant digits are defined.

The Standard was proposed by and shall be administered by Ministry of Health of the People's Republic of China.

Drafting organizations of this Standard: National Institute for Nutrition and Food Safety of Chinese Center for Disease Control and Prevention, and Beijing Center for Diseases Prevention and Control.

The main drafters of this Standard are: Yang Dajin, Chang Di, Zhao Xin, Wu Guohua, and Xue Ying.

The previous versions of the standard replaced by this standard are as follows:

- GB/T 5009.7-1985 and GB/T 5009.7-2003.

Determination of reducing sugar in foods

1 Scope

This Standard specifies the determination method of reducing sugar content in food.

This standard applies to the determination of reducing sugar content in food.

When 5.0g of sample is taken, the detection limit of direct titration is 0.25g/100g, and the detection limit of permanganate titration is 0.5g/100g.

The First method - Direct titration

2 Principle

After the sample's protein is removed, under the condition of heating, TAKE the methylene blue as the indicator. TITRATE the marked alkaline copper tartrate solution (marked by reducing sugar standard solution). Calculate the reducing sugar content according to the consumed volume of the sample solution.

3 Reagents

Unless otherwise specified, reagents applied in this method are all analytical reagents.

3.1 Hydrochloric acid (HCl).

3.2 Copper sulfate ($CuSO_4 \cdot 5H_2O$).

3.3 Methylene blue ($C_{16}H_{18}C1N_3S \cdot 3H_2O$): Indicator.

3.4 Sodium potassium tartrate [$C_4H_4O_5KNa \cdot 4H_2O$].

3.5 Sodium hydroxide (N_aOH).

3.6 Zinc acetate [$Zn(CH_3COO)_2 \cdot 2H_2O$].

3.7 Glacial acetic acid ($C_2H_4O_2$).

3.8 Potassium ferrocyanide [$K_4Fe(CN)_6 \cdot 3H_2O$].

3.9 Dextrose ($C_6H_{12}O_6$).

3.10 Fructose ($C_6H_{12}O_6$).

water to dissolve it. Put it into a triangular flask with plug. Then add 5mL of hydrochloric acid (1+1). Heat it for 15min in water at 68°C-70°C. And then place it till the temperature turning to the indoor temperature. After that, transfer the solution into a 1000mL volumetric flask and dilute to 1000mL. Per milliliter of this standard solution equal to 1.0mg of inverted sugar.

4 Instruments

4.1 Acid burette: 25mL;

4.2 Adjustable electric furnace: equipped with asbestos board.

5 Analytical procedure

5.1 Sample processing

5.1.1 General food: Take about 2.5g-5g of smashed solid sample or 5g-25g blended fluid sample, with the measurement accurate to 0.001g. Put it into a 250mL volumetric flask, add 50mL of water. Slowly inject 5mL of zinc acetate solution and 5mL of Potassium ferrocyanide solution. Then raise the solution level with water to the scale. Blend the solution. Place it for 30min. Filter it with dry filter paper. Dispose the primary filtrate. Take out the subsequent filtrate for standby.

5.1.2 Stimulant: Take about 100g of blended sample, accurate to 0.01g. Put it into an evaporating dish. Neutralize it with sodium hydroxide (40g/L) solution to neutral state. Evaporate it to 1/4 of its initial volume by bathing it in the water. Transfer it to a 250mL volumetric flask. Manipulate the following procedure according to the regulations of 5.1.1 from "slowly inject 5mL of zinc acetate solution".

5.1.3 Foods containing large quantity of starch: Take about 10g-20g of smashed or blended sample, with the measurement accurate to 0.001g. Put it into a 250mL volumetric flask. Add 200mL of water. Heat it for 1h in a 45°C water bath, shaking it frequently. After cooled, add water to the scale. Blend it. Place it and precipitate it. Absorb 200mL of supernate and put it into another 250mL volumetric flask. Manipulate the following

too high, it shall dilute appropriately before starting the formal determination. Ensure that the volume of the consumed sample solution each time is similar to that of the consumed reducing sugar standard solution used for the determination of the alkaline copper tartrate solution, which is about 10mL. The result is calculated according to formula (1). When the concentration is too low, add 10mL of sample solution rather than 10mL of water. Add reducing sugar standard solution to the end. Record the difference between the consumed volume AND the volume of the reducing sugar standard solution consumed for determination, with such difference is equivalent to the reducing sugar content in 10mL sample solution. Calculate the result according to formula (2).

5.4 Determination of the sample solution

Absorb 5.0mL of alkaline copper tartrate A solution and 5.0mL of alkaline copper tartrate B solution. Put them into one 150mL conical flask. Add 10mL of water and two glass balls. Then use burette to drop sample solution which is 1mL less than the forecasted volume to the conical flask. Ensure it get boiling within 2min. Keep the boiling and drop solution at a speed of 1 drop/2s to the moment of the disappearance of the blue color. Record the total consumed volume. Operate 3 such solutions simultaneously and in parallel. Obtain the average consumed volume.

6 Result calculation

The reducing sugar content in the sample (calculating a certain kind of reducing sugar) shall be determined according to formula (1):

$$X = \frac{m_1}{m \times V/250 \times 1\,000} \times 100 \qquad \cdots\cdots(1)$$

Where:

X — The reducing sugar content in the sample (calculating a certain kind of reducing sugar), g/100g;

m_1 — The alkaline copper tartrate solution (A solution and B solution each half) which is equal to the mass of a certain kind of reducing sugar, mg;

8.1 Copper sulfate ($CuSO_4 \cdot 5H_2O$).

8.2 Sodium hydroxide (N_aOH).

8.3 Sodium potassium tartrate ($C_4H_4O_6KNa \cdot 4H_2O$).

8.4 Ferric sulfate [$Fe_2(SO_4)_3$].

8.5 Hydrochloric acid (HC1).

8.6 Alkaline copper tartrate A solution: Take 34.639g of copper sulfate ($CuSO_4 \cdot 5H_2O$). Add some water to dissolve it. Then add 0.5mL of sulfuric acid. Dilute it with water to 500mL. And filtrate it with refined asbestos.

8.7 Alkaline copper tartrate B solution: Take 173g of potassium sodium tartrate, 50g of sodium hydroxide. Add a moderate amount of water to dissolve it. And dilute it to 500mL. Filtrate it with refined asbestos. And store the solution in a glass bottle with rubber plug.

8.8 Sodium hydroxide solution (40g/L): Take 4g of sodium hydroxide. Add water to dissolve and dilute it to 100mL.

8.9 Ferric sulfate solution (50g/L): Take 50g of ferric sulfate. Add 200mL of water to dissolve it. Slowly add 100mL sulfuric acid into it. And dilute it to 1000mL after cooling.

8.10 Hydrochloric acid (3mol/L): Take 30mL of hydrochloric acid and dilute it with water to 120mL.

8.11 Permanganate standard solution [$c(1/5KMnO_4)$=0.1000mol/L].

8.12 Refined asbestos: Before using the asbestos, soak it into the hydrochloric acid (3mol/L) for 2d-3d. Wash it with water. Add sodium hydroxide solution (400g/L) to soak it for 2d-3d. Discard the solution. Then use hot alkaline copper tartrate B solution to soak it for several hours. Wash it with water. Then put it into hydrochloric acid (3mol/L) for several hours. Keep washing it with water till it is not acidic. Add water and shake it. Make it form tiny soft fiber of pulpiness. Put it into water and store it in a glass bottle. That is to be used to fill Gooch crucible.

9 Instruments

9.1 25mL Gooch crucible or G4 vertical melting crucible.

alkaline copper tartrate A solution and 25mL of B solution. Cover a watch glass over the beaker. Heat it. Ensure it to boil within 4min. Keep boiling for another 2min accurately. While it is hot, leach it with Gooch crucible or G4 vertical melting crucible which are laid with asbestos. Wash the beaker and sedimentation with 60ºC water till the water is not alkaline. Put the Gooch crucible and vertical melting crucible back to the 400mL beaker. Add 25mL of ferric sulfate solution and 25mL water. Use glass rod to stir it to completely dissolve cuprous oxide. Use permanganate standard solution [$c(1/5KMnO_4)$=0.1000mol/L] to drop it to a reddish color as its end.

At the same time, absorb 50mL of water. Add alkaline copper tartrate A solution, B solution, ferric sulfate solution and water of which the amounts are same as the determined samples. Conduct blanking test according to the same method.

10.3 Result calculation

The mass of the reducing sugar in test sample is similar to that of the cuprous oxide. it shall be calculated according to formula (3).

$$X = (V - V_0) \times c \times 71.54 \qquad \cdots\cdots(3)$$

Where:

X — The mass of the reducing sugar which is similar to that of the cuprous oxide, mg;

V — The sample solution volume of the consumed permanganate standard solution in the determination, mL;

V_0 — The sample solution volume of the consumed permanganate standard solution of blanking, mL;

C — Concentration of potassium permanganate standard solution, mol/L;

71.54 — The mass that 1mL of 1.000mol/L permanganate solution is equivalent to the cuprous oxide, mg.

Obtain the cuprous oxide mass according to the calculation of the formula. Check Table 1. Then calculate the reducing sugar content in the test sample. Calculate according to formula (4).

$$X = \frac{m_3}{m_4 \times V/250 \times 1\,000} \times 100 \qquad \cdots\cdots(4)$$

Where:

X — Content of reducing sugar in the test sample, g/100g;

m_3 — The mass of reducing sugar obtained from the table, mg;

m_4 — Sample mass (volume), g or mL;

V — The volume of the sample solution in the determination, mL;

250 — The total volume of the sample after processing, mL.

When the reducing sugar content is ≥10g/100g, the result calculated shall be kept three significant digits. When the reducing sugar content is <10g/100g, the result calculated shall be kept two significant digits.

421.1	199.1	213.3	290.3	206.3		469.5	225.1	240.3	324.9	232.9
422.2	199.7	213.9	291.1	206.9		470.6	225.7	241.0	325.7	233.6
423.3	200.3	214.5	291.9	207.5		471.7	226.3	241.6	326.5	234.2
424.4	200.9	215.1	292.7	208.1						
425.6	201.5	215.7	293.5	208.7		472.9	227.0	242.2	327.4	234.8
426.7	202.1	216.3	294.3	209.3		474.0	227.6	242.9	328.2	235.5
						475.1	228.2	243.6	329.1	236.1
427.8	202.7	217.0	295.0	209.9		476.2	228.8	244.3	329.9	236.8
428.9	203.3	217.6	295.8	210.5		477.4	229.5	244.9	330.8	237.5
430.1	203.9	218.2	296.6	211.1		478.5	230.1	245.6	331.7	238.1
431.2	204.5	218.8	297.4	211.8		479.6	230.7	246.3	332.6	238.8
432.3	205.1	219.5	298.2	212.4		480.7	231.4	247.0	333.5	239.5
433.5	205.1	220.1	299.0	213.0		481.9	232.0	247.8	334.4	240.2
434.6	206.3	220.7	299.8	213.6		483.0	232.7	248.5	335.3	240.8
435.7	206.9	221.3	300.6	214.2						
436.8	207.5	221.9	301.4	214.8		484.1	233.3	249.2	336.3	241.5
438.0	208.1	222.6	302.2	215.4		485.2	234.0	250.0	337.3	242.3
						486.4	234.7	250.8	338.3	243.0
439.1	208.7	232.2	303.0	216.0		487.5	235.3	251.6	339.4	243.8
440.2	209.3	223.8	303.8	216.7		488.6	236.1	252.7	340.7	244.7
441.3	209.9	224.4	304.6	217.3		489.7	236.9	253.7	342.0	245.8

11 Precision

Under the repeatability condition, the absolute different value obtained between the 2 independent determination results must not exceed 10% of the arithmetic mean value.

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