Japan Customs Analysis Methods

No. 104

Analysis Method for Starch

(Issued in June 1999)
(Updated in May 2001)

1. Scope

This analysis method is applied to goods which require quantitative determination of starch content using the modified Ewers polarimetric method.

2. Outline of Test Method

This method determines starch content in flour and its preparations. The procedure is as follows.

(1) Sample preparation
(2) Measurement of total sugar content by saccharimeter.
(3) Measurement of sugar content of water soluble active substance by saccharimeter
(4) Calculation of starch content.

3. Apparatus

(a) Saccharimeter

The apparatus should be equipped with a transmitted light source of a sodium vapor lamp and a half-shadow angle of the Lippich polarizing system. The scale should conform to the International Sugar Scale adopted by the International Commission for Uniform Methods of Sugar Analysis (ICUMSA).

(b) Observation tube

A tube of 200 mm (tolerance ± 0.03 mm) with a swollen shape at one end, with an optically-inactive glass lid equipped with a gum washer, and a closing metal fitting for the glass lid.

(c) Pulverizer
(d) Sieve with 1 mm openings.

4. Reagents

All chemicals must be JIS special reagent grade or equivalent, unless otherwise specified.

(a) 1.128% hydrochloric acid aqueous solution

This hydrochloric acid solution is prepared such that 10 mL of the solution requires 30.94 mL of 0.1 N sodium hydroxide for neutralization. The indicator used for neutralization is methyl red (1% solution in 95% ethyl alcohol).

(b) 25% hydrochloric acid aqueous solution

Prepare this solution to obtain a specific gravity of 1.126 (20 °C).

(c) 4% sodium phosphotungstate aqueous solution
(d) 40% zinc sulfate aqueous solution
(e) 10% potassium ferrocyanide aqueous solution.
(f) Filter paper

Specified in JIS P 3801 Grade 2, 185 mm in diameter, or equivalent product.

5. Sample Preparation

Pulverize the sample to pass through a sieve with 1 mm openings.

6. Measurement Procedure

6.1. Measurement of Total Sugar Content

Mix the sample to obtain a homogenous mixture. Weigh 2.500 g (± 0.001 g) of the sample in a 200-mL Erlenmeyer flask. Add 25 mL of 1.128% hydrochloric acid aqueous solution. Mix until the hydrochloric acid solution has thoroughly infiltrated the sample. Add further 25 mL of 1.128% hydrochloric acid aqueous

solution. Connect the flask to a straight tube and vigorously shake in a boiling water bath for 1 minute. After heating in the boiling water bath for 15 minutes, remove the Erlenmeyer flask and immediately cool to 20 °C. Transfer the content in the flask to a 100-mL volumetric flask. Wash the Erlenmeyer flask with water at least three times and transfer all the washing liquid into the volumetric flask. Add 10 mL of 4% sodium phosphotungstate and mix [1]. Dilute to the volume with water at 20 °C and filter [2]. Discard the first 10 to 15 mL of the filtrate. Use approximately 30 mL of the filtrate to rinse the observation tube. Then, use the 200-mm observation tube to measure the sugar content in the filtrate at 20 °C [3]. The sugar content is designated as S.

Note 1)   Instead of 4% sodium phosphotungstate, 5 mL of 40% zinc sulfate and 5 mL of 10% potassium ferrocyanide may be added.

Note 2)   If precipitation occurs after adding 4% sodium phosphotungstate into the filtrate, repeat the procedure by increasing the initial volume of the 4% sodium phosphate.

Note 3)   Refer to Customs Analysis Method No. 101 "Determination of Sugar Content of Sugar" for the determination of sugar content.

6.2. Measurement of Sugar Content of Water Soluble Active Substance

Measure 12.500 g (± 0.001 g) of the sample in a beaker. Transfer this sample to a 250-mL volumetric flask with approximately 200 mL of water. Mix from time to time to extract for 1 hour at room temperature. Dilute the solution to volume with water and filter. Using a volumetric pipette, transfer 50 mL of the filtrate into a 200-mL Erlenmeyer flask. Add 2.1 mL of a 25% hydrochloric acid aqueous solution. Connect the flask to an air condenser and vigorously shake in a boiling water bath for 1 minute. After heating in the boiling water bath for 15 minutes, remove the Erlenmeyer flask and immediately cool to 20 °C. Transfer the content in the flask to a 100-mL volumetric flask. Wash the Erlenmeyer flask with water at least three times and transfer all the washing liquid into the volumetric flask. Add 10 mL of 4% sodium phosphotungstate and mix [1]. Dilute the solution to volume with water at 20 °C and filter [2]. Discard the first 10 to 15 mL of the filtrate.

Use approximately 30 mL of the filtrate to rinse the observation tube. Then, use the 200-mm observation tube to measure the sugar content in the filtrate at 20 °C [3]. The sugar content is designated as S'.

7. Calculation of Starch Content

Calculate the starch content in the sample from the following formula and express as percentage of dry weight.

$$A, \% = \frac{26.6 \times N \times (S - S')}{[\alpha D]} \times \frac{100}{100 - M}$$

Where –

A : starch content (%)

N : 26.0 (value using a Schmidt Haensch device)

$[\alpha D]$:

Oat starch	181.3
Barley starch	181.5
Wheat starch	182.7
Rye starch	184.0
Corn starch	184.5
White potato starch	185.7
Rice starch	185.9
Other starch	184

S : sugar content determined in 6.1.

S' : sugar content determined in 6.2.

M : moisture content determined in Note 4.

Round down to the nearest first decimal place.

Note 4)   The moisture content of the sample is determined as follows.

Accurately weigh 5 g (± 0.001 g) of the sample prepared in a weighing bottle. Dry in a hot forced air oven at 105 °C (±1 °C) to constant weight. Transfer the weighing bottle to a desiccator for 30 minutes to cool down before weighing. The weight reduced is taken as moisture content and is expressed as a percentage. Sampling of the sample is conducted simultaneously with the weighing of the sample for 6.1. and 6.2.

8. Temperature Correction

When sugar content is determined at a temperature other than 20 °C, temperature correction of the sugar content must be applied using the temperature

at the weighing of the sample according to the following formula.

$$St20, \% \ = \ Sa - 0.02 \times (\ 20 - Ta \ )$$

Where –

    $St20$: sugar content corrected at 20 °C

    Sa :   sugar content measured

    Ta :   temperature of sugar solution at the time
           of measurement