Japan Customs Analysis Methods

No. 109

Quantitative Analysis of reducing sugars in dextrin

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1. Scope

This analysis method is applied to starch degradation products which are imported as dextrin and which require the determination of their "reducing sugar content, expressed as dextrose in dry substances," as referred to in Note 2 to Chapter 35 in Customs Tariff Law (Appendix Table—Customs Tariff Schedule).

2. Outline of Test Method

This analysis method is applied for determining reducing sugars, e.g. dextrose and maltose, in dextrin (starch degradation products), expressed as dextrose. The procedure is as follows.

(1) Determination of moisture content.
(2) Determination of reducing sugars by the Lane-Eynon method.
(3) Calculation of the content (%) of reducing sugars, expressed as dextrose.

3. Reagents

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

3.1. Preparation of reagents

(1) Standard invert sugar solution

Accurately weigh 4.75 g of sucrose and transfer with 90 ml of water to a 500 mL volumetric flask. Add 5 mL of hydrochloric acid (specific gravity, 1.18) to the flask and store for three days at room temperature (20–30°C). Then, dilute the solution to volume with water and keep in a cool dark place. Transfer one 50 mL portion of the solution to a 200 mL volumetric flask, neutralize with 1 mol/L sodium hydroxide aqueous solution using phenolphthalein as an indicator, and dilute to volume with water.

Use the solution as standard invert sugar solution for the standardization of Fehling's solution.

(2) 1% Methylene Blue solution

Dissolve 1 g of methylene blue in water to make 100 mL.

(3) Fehling's Solutions

Solution A: Dissolve 34.639 g of copper sulfate ($CuSO_4 \cdot 5H_2O$) in water to make exactly 500 mL, leave it for two days, and then filter.

Solution B: Dissolve 173 g of potassium sodium tartrate ($KNaC_4H_4O_6 \cdot 4H_2O$) and 50 g of sodium hydroxide in water to make exactly 500 mL, leave it for two days, and then filter.

3.2. Standardization of Fehling's Solution

Put 5.0 mL of Fehling's Solution A and 5 mL of Fehling's Solution B in a 200 mL Erlenmeyer flask containing a few glass beads and add from a 50 mL burette 19.5 mL of the standard invert sugar solution. After boiling on an electric stove (heater) for two minutes, add four drops of the methylene blue solution to the flask, and complete titration within a total boiling time of three minutes by dropwise addition of the standard invert sugar solution—without preventing boiling—until the blue color disappears. Repeat titration twice and calculate the mean of three parallel titrations. (1)

Obtain the *factor* of the Fehling's Solution from the following formula:

$$Factor^{(2)} = \frac{20.36}{A}$$

Where– A: Volume (mL) of the standard invert sugar solution required

Note 1) Use the mean value of three parallel titrations as the volume of the standard invert sugar solution required; Duplicate titrations must agree to within 0.1 mL of each other.

Note 2) Calculate *factor* by rounding off fractions to the third decimal place; the *factor* must be within a range of 1± 0.02.

4. Preparation of sample

Prepare analysis samples according to either of the following procedures, depending on their properties.

4.1. Solid samples

Grind into powder (or crystal-like powders). Crush any lumps and mix well.

4.2. Liquid samples

When crystals or solids are present in a sample, place the sample in a sealed container and immerse it in a water bath at a temperature of 60–70°C to melt them. After melting, shake the container vigorously to mix and cool to room temperature.

5. Procedure

5.1. Determination of moisture content

Determine the moisture content in samples according to either of the following procedures, depending on the types of the samples.

5.1.1. For solid samples

Accurately weigh about 2 g of the sample homogenized in 4. into a tared weighing bottle. Dry it in a vacuum oven at a temperature of 70°C for four hours, cool to room temperature in a desiccator and weigh. Repeat vacuum drying until the loss in weight does not exceed 2 mg per hour in the drying period.

5.1.2. For liquid samples

Place about 15 g of sea sand,[3] as a drying agent, and a short glass rod in a weighing bottle, and dry in an oven at a temperature of 105°C to a constant weight. Add a known amount of the sample homogenized in 4., equivalent to about 2 g of the dry matter—add a small amount of water until the sample is entirely soaked if necessary—and heat in a steam water bath, stirring occasionally, until most of the water is evaporated. Dry the sample in the weighing bottle in a drying oven at a temperature of 105°C, stirring occasionally, until the sample is almost dried. Transfer the weighing bottle with the sample to a vacuum oven and dry at 70°C for four hours. Cool to room temperature in a desiccator and weigh. Repeat vacuum drying until the loss in weight does not exceed 2 mg per hour in the drying period.

Note 3) Celite (diatomaceous earth) can be used in place of sea sand. In that case, use about 5 g of Celite as drying agent.

5.1.3. Calculation of moisture content

Calculate from the following formula the moisture content in the sample. Round off fractions to the second decimal place.

$$\%, \text{moisture} = \frac{W_0 - W_1}{W_0} \times 100$$

Where–

W_0: Weight (g) of sample collected.

W_1: Weight (g) of sample after drying.

5.2. Determination of reducing sugar content

5.2.1. Preparation of test solution

Accurately weigh about 10 g[4] of the sample homogenized in 4., and dissolve in water. Transfer the solution to a 500 mL volumetric flask and dilute to volume with water. Use the solution as test solution.

Note 4) This is a guideline for the sampling of dextrin whose concentration of reducing sugar, expressed as dextrose, is about 10%.

5.2.2. Titration

Put 5.0 mL of Fehring's Solution A and 5 mL of Fehring's Solution B in a 200 mL Erlenmeyer flask containing a few glass beads. Add 15 mL of the test solution prepared in 5.2.1 by mean of a 50 mL burette.

Titrate with the test solution in the same manner as in the procedure in 3.2., as a preliminary titration.

Subsequently, put 5.0 mL of Fehring's Solution A and 5 mL of Fehring's Solution B in another 200 mL Erlenmeyer flask. Add from a 50 mL burette the test solution within 1 mL of the anticipated end point from the result of the preliminary titration above. Titrate in the same manner as in the preliminary titration above.

Multiply by the *factor* of the Fehling Solution the volume (mL) of the test solution required. Using the corrected value, obtain the concentration of reducing sugar, expressed as dextrose, by referring to the appended Lane-Eynon Table (Dextrose).

5.2.3. Calculation of reducing sugar content

Calculate from the following formula the content of reducing sugar, expressed as dextrose, in the sample in a dried state. Round off fractions to the first decimal place.

$$\%, \mathrm{DE} = \frac{\mathrm{Ds}}{2(100 - \mathrm{M})\mathrm{S}} \times 100$$

Where–

DE (%): Content (%) of reducing sugar, expressed as dextrose, in the sample in a dried state.

Ds: Concentration (mg/100mL) of dextrose in test solution, obtained by reference to the appended Lane-Eynon Table (Dextrose).

M: Moisture content (%) of sample.

S: Weight (g) of sample collected.

6. References

(1) ISO 5381:1983 "Starch hydrolysis products–Determination of water content–Modified Karl Fischer method"

(2) ISO 5377:1981 "Starch hydrolysis products–Determination of reducing power and dextrose equivalent–Lane and Eynon constant titre method"

(3) AOAC (1980)

(4) 日本食品工業学会食品分析法編集員会編「食品分析法」光琳 (1982)

(5) 中村道徳、鈴木繁男 編「澱粉化学ハンドブック」 朝倉書店 (1976)

(6) 浜口栄次郎、桜井芳人 編「シュガーハンドブック」 朝倉書店 (1964)

Appendix

Lane-Eynon Table (Dextrose)

(all figures relate to anhydrous dextrose)

mL of sugar solution required	Saccharides:
	dextrose, mg/100mL
15	327
16	307
17	289
18	274
19	260
20	247.4
21	235.8
22	225.5
23	216.1
24	207.4
25	199.3
26	191.8
27	184.9
28	178.5
29	172.5
30	167
31	161.8
32	156.9
33	152.4
34	148
35	143.9
36	140
37	136.4
38	132.9
39	129.6
40	126.5
41	123.6
42	120.8
43	118.1
44	115.5
45	113
46	110.6
47	108.4
48	106.2
49	104.1
50	102.2