Japan Customs Analysis Methods

No. 121

Quantitative Analysis of Rice Powder in the Preparations of Rice Powder and Starch Derivatives

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1. Scope

This analytical method is applied for determining the contents of rice powder in preparations made up of rice powder and starch derivatives.

2. Outline of Test Method

This analytical method determines nitrogen in preparations composed of rice powder and starch derivatives by the Kjeldahl method and calculates the quantities of rice powder in the analytical samples by comparing the obtained amounts of nitrogen with the amounts of nitrogen in raw materials (rice powder and starch derivatives) [(1)].

Note 1) Qualitative analysis of raw material starch derivatives should be carried out in accordance with the Japan Customs Analysis Method No. 403.

3. Apparatus and Reagents

A quantitative instrument, such as automatic nitrogen/protein analyzer, deployed in Customs laboratories, is utilized.

Analytical reagents and methods for preparing solutions must follow descriptions of the instruction manual of analytical instrument used.

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

4. Procedure

Determination shall be carried out by the Kjeldahl method or using an automated combustion-type nitrogen analyzer (N coder).

4.1. Determination by Kjeldahl method

4.1.1. Digestion

Accurately weigh approximately 1 g of analytical sample, approximately 2 g of raw material starch derivatives and approximately 1 g of raw material rice powder onto medicine-wrapping-papers, respectively. Wrap the respective medicine-wrapping-papers and put them into separate digestion tubes. For preparation of a blank, put only a medicine-wrapping-paper in another digestion tube.

After putting one tablet of digestion accelerator into each of the digestion tubes above, add 10 mL of concentrated sulfuric acid. Furthermore, add 10 mL of a hydrogen peroxide solution little by little while paying attention to explosive boiling.

Set the digestion tubes on a Kjeldahl digestion apparatus. The conditions for digestion temperature and digestion time shall be adjusted to get the recovery of nitrogen from a standard tryptophan to be in the range of 98% to 100%.

An example of digestion conditions (temperature and time) considered is as follows:

(Primary) 200°C (20 min)
(Secondary) 420°C (110 min)

4.1.2. Distillation and Titration

Follow the standard procedure for operating analytical apparatus used.

4.2. Determination using N coder

For the sampling amount, handlings of apparatus, etc., follow the instruction manuals given by the manufacturer.

5. Determination of Moisture

Accurately weigh approximately 2 g each of the analytical sample, raw material starch derivatives and raw material rice powder into weighing bottles which have previously been dried to their constant weights, and dry them at 105°C for four hours. After cooling to a room temperature in a desiccator, weigh the bottles. Repeat drying at 105°C until the loss in weight does not exceed 2 mg per hour in the drying period.

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

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

Where–

W_0: Amount of sample collected (g)

W_1: Weight of sample after dried (g)

6. Calculation of Rice Powder Content

6.1. Calculation of the rice powder content on a dry basis

After determining the nitrogen contents and moisture contents of the analytical sample, raw material starch derivatives and raw material rice powder, calculate the rice powder content in the analytical sample on a dry basis from the following formula. Round off fractions to the first decimal place.

$$\%, \text{rice powder (dry base)}, \mathrm{Xd} = \frac{\mathrm{xs} - \mathrm{xp}}{\mathrm{xr} - \mathrm{xp}} \times 100$$

Where–

$\mathrm{xs} = \dfrac{100\mathrm{Ns}}{100 - \mathrm{Ws}}$: Nitrogen content (%) in analysis sample on a dry basis

$\mathrm{xr} = \dfrac{100\mathrm{Nr}}{100 - \mathrm{Wr}}$: Nitrogen content (%) in raw material rice powder

$\mathrm{xp} = \dfrac{100\mathrm{Np}}{100 - \mathrm{Wp}}$: Nitrogen content (%) in raw material starch derivative

Ns: Nitrogen content (%) in analytical sample

Nr: Nitrogen content (%) in raw material rice powder

Np: Nitrogen content (%) in raw material starch derivative

Ws: Moisture (%) in analytical sample

Wr: Moisture (%) in raw material rice powder

Wp: Moisture (%) in raw material starch derivative

6.2. Calculation of rice powder content

Using the percentage of rice powder in the analytical sample on a dry basis obtained according to 6.1., calculate the rice powder content from the following formula. Round off fractions to the first decimal place.

$$\%, \text{rice powder} = \frac{\mathrm{Xd}(100 - \mathrm{Wp})}{\mathrm{Xd}(100 - \mathrm{Wp}) + (100 - \mathrm{Wr})(100 - \mathrm{Xd})} \times 100$$

6. References

(1) Yamaguchi M., Kumazawa T., Sekikawa Y. (1995) Reports of the Central Customs Laboratory **34**: 21 (in Japanese).

(2) Onishi M., Suzuki M., Hayano M. (1992) Reports of the Central Customs Laboratory **31**: 71 (in Japanese).

(3) 澱粉糖関連工業分析法 (1991) 澱粉糖技術部会編.