Japan Customs Analysis Methods

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Quantitative Analysis of Salt in Vegetables (and Fruits) Preserved in Brine

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1. Scope

This quantitative analytical method is applied to vegetables or fruits, preserved in brine, for which their salt concentrations are required. (The salt concentration means the mass percentage of sodium chloride in a sample.).

2. Outline of Test Method

This method is applied for the determination of salt in vegetables or fruits preserved in brine by means of the Mohr method or a potentiometric titration method according to the following procedure:

(1) Standardization of 0.1 mol/L silver nitrate standard solution (determination of factor).
(2) Preparation of samples.
(3) Titration.

3. Apparatus

A potentiometric automatic titration apparatus equipped with a platinum electrode in combination with a reference electrode.

4. Reagents

All chemicals must be JIS special reagent grade or equivalent, unless otherwise specified. All solutions for titration can be a commercial standard solution for volumetric analysis as long as it is a JA.5 solution for titration of JIS K 8001 or standardized on the Japanese Pharmacopoeia for its strength (factor).

(a) 0.1 mol/L silver nitrate standard solution

Dissolve approximately 17 g of silver nitrate in water to make a 1,000 mL solution. Store the solution in a brown bottle and in a dark place.

(b) 5% potassium chromate solution

Dissolve 5 g of potassium chromate in water to make a 100 mL solution.

(c) 0.05 mol/L silver nitrate standard solution

Dissolve about 8.5 g of silver nitrate in water to make a 1,000 mL solution. Store the solution in a brown bottle and in a dark place.

(d) 1% Tween 20 (polyoxyethylene sorbitan monolaurate) solution

Dissolve 10 g of Tween 20 in water to make a 1,000 mL solution.

(e) 40% nitric acid

Dissolve 40 mL of concentrated nitric acid in water to make a 100 mL solution.

5. Mohr Method

5.1. Standardization of 0.1 mol/L silver nitrate standard solution

Heat sodium chloride at 500 to 650°C for 4 to 5 hours. Accurately weigh 5.8454 g of it and dissolve in water to make a 1,000 mL solution. Take 25.0 mL of the solution in a 200 mL-Erlenmeyer flask. Next, add 1 mL of 5% potassium chromate solution as an indicator after adding 25 mL of water, and then shake thoroughly to mix them. Titrate it with the 0.1 mol/L silver nitrate standard solution using a brown burette. The end-point can be detected by the appearance of a permanent red

precipitate over about 15 seconds after the color of the solution changes from yellow to red brown. Repeat titration twice and calculate the mean value as the titration value. Nevertheless, the difference between the two titration values must be 0.2 mL or less.(1)

Obtain the factor of the 0.1 mol/L silver nitrate standard solution from the following equation.

$$F=V2/V1$$

Where —

F : factor of 0.1 mol/L silver nitrate standard solution

V1 : titration volume of 0.1 mol/L silver nitrate solution (mL)

V2 : volume of sodium chloride solution used (mL)

Round off fractions to the third decimal place.

Note 1) The end point can be clearly identified when titration is conducted under cooling with ice.

5.2. Preparation of samples

Take an appropriate amount of analysis sample from the container and crush it with a mixer or the like. Accurately weigh about 5 g of the crushed sample,(2) add water, transfer to a 200 mL-volumetric flask and dilute to the volume with water. Shake it thoroughly and leave to stand for a while. Then filter the liquid and use the filtrate as analyte.

The filtrate (analyte) must be neutral (pH6.5 to 10.5). If not, it should be neutralized with sodium hydrogen carbonate, acetic acid, etc.

Note 2) Prepare the analyte so that the chlorine content is about 0.1 g / 50 mL.

5.3. Titrimetric operation

Put 50 mL of the analyte obtained in 5.2 above in a 200 mL-Erlenmeyer flask, add 1 mL of 5% potassium chromate solution as an indicator, and titrate in the same manner as that mentioned in 5.1 “Standardization of 0.1 mol/L silver nitrate standard solution ” above.

5.4. Calculation of salt content

$$\text{Salt},\% = \frac{V \times F \times 0.005845 \times 200 \times 100}{50 \times S}$$

Where —

V : titration volume of 0.1 mol/L silver nitrate solution (mL)

F : factor of 0.1 mol/L silver nitrate solution

S : amount of sample collected (g)

Round off fractions to the first decimal place.

6. Potentiometric Titration Method

6.1. Standardization of 0.05 mol/L silver nitrate standard solution

Heat sodium chloride at 500 to 650°C for 4 to 5 hours. Accurately weigh 2.9227g of it and dissolve in water to make a 1,000 mL solution. Obtain the factor of the 0.05 mol/L silver nitrate standard solution by a potentiometric titration against the sodium chloride solution.

6.2. Preparation of test substance

Take an appropriate amount of analysis sample from the container and crush it with a mixer or the like. Accurately weigh about 2 g of the sample, add water and transfer to a 500 mL volumetric flask. After stirring the liquid with a stirrer for one to two hours, sediment solids by leaving to stand for a while or filter the liquid.

6.3. Titrimetric operation

Using a whole pipette, transfer 25 mL of the supernatant or filtrate obtained in 6.2 above,(3) into a titration beaker. Add 2 to 3 mL each of 40% nitric acid and 1% Tween 20 solution into the titration beaker and dilute with water until a volume suitable for measurement. Conduct titration with an automatic potentiometer.

Round off fractions to the first decimal place.

Note 3) If the concentration of salt in the analyte is lower than 15%, increase the volume of the analyte collected using a whole pipette.

7. References

(1) Ikeda H., Yamakami K., Tomita K. (1998) Report of the Central Customs Laboratory, Japan **38**: 7 (in Japanese).