Japan Customs Analysis Methods

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Quantitative Analysis of Salt in Salted Meat

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1. Scope

This analysis method is applied to meat and edible meat offal, salted or 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 determines the concentration of salt in salted meat by the Volhard method according to the following procedures:

(1) Standardization of standard solutions (determination of the factors)
(2) Preparation of test samples.
(3) Titration.

3. 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).

(1) 0.1 mol/L silver nitrate standard solution (1)

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

[Determination of factor]

Heat sodium chloride at 500 to 650 ºC for 4 to 5 hours. Dissolve exactly 5.8454 g of it in water to make a 1,000 mL solution. Take 25.0 mL of the solution in a 200 mL-Erlenmeyer flask. After adding 25 mL of water into the flask, add 1 mL of 5% potassium chromate solution as an indicator and shake thoroughly to mix. Titrate it with the 0.1 mol/L silver nitrate standard solution by the use of 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)

The factor is obtained 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 standard solution (mL)

V2: amount 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.

(2) 5% potassium chromate solution

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

(3) 0.5 mol/L silver nitrate solution

Dissolve 85 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.

(4) 5% potassium permanganate solution

Dissolve 25 g of potassium permanganate in water to make a 500 mL solution. Store the solution in a brown bottle.

(5) Saturated solution of ammonium iron (III) sulfate

Add ammonium iron (III) sulfate in 100 mL of water and saturate.

(6) 0.1 mol/L ammonium thiocyanate standard solution

Dissolve approximately 8 g of ammonium thiocyanate in water to make a 1,000 mL solution and store in a brown bottle.

(7) Diethyl ether

(8) Sucrose

4. Determination of the factor of 0.1 mol/L ammonium thiocyanate standard solution

Accurately take 25.0 mL of 0.1 mol/L silver nitrate solution in a 250 mL-Erlenmeyer flask. Add 50 mL of water, 2 mL of nitric acid(2) and 2 mL of saturated ammonium iron (III) sulfate solution as an indicator to the flask. Titrate the solution with the 0.1 mol/L ammonium thiocyanate standard solution while shaking vigorously.(3),(4) The end point of the titration occurs when the supernatant shows pale red even after leaving it to stand for about 30 seconds. 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.

Use the following equation to obtain the factor of 0.1 mol/L ammonium thiocyanate solution.

$$F = \frac{V2 \times f}{V1}$$

Where —

F : factor of 0.1 mol/L ammonium thiocyanate standard solution

V1 : titration value of 0.1 mol/L ammonium thiocyanate standard solution (mL)

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

V2 : amount of 0.1 mol/L silver nitrate standard solution used (mL)

Round off fractions to the third decimal place.

Note 2) It is added to decolorize ammonium iron (III) sulfate of yellow-brown.

Note 3) Titration will finish before the correct end point if the flask is not shaken vigorously.

Note 4) Titration should be carried out at the liquid temperature of 25°C or below.

5. Preparation of Test samples and Titration

Cut off a suitable portion weighing 2.5 – 3 g from the sample as a specimen,(5) put it in a 300 mL Erlenmeyer flask and measure its weight accurately. Accurately add 10 mL of 0.5 mol/L silver nitrate solution.(6) Further add 15 mL of nitric acid and boil it slowly by heating on a sand saucer or a heat plate until the specimen decomposes.(7) After the decomposition of the specimen, while continuing to boil, add 15 mL of 5 % potassium permanganate solution three times. When the solution almost loses its color after once turning to yellow, add 25 mL of water and again boil for 5 min. After cooling, add water to the solution to make it about 150 mL in total. Then add 25 mL of diethyl ether, shake well and use it as test solution.

To the test solution, add 5 mL of saturated ammonium iron (III) sulfate solution as an indicator and then perform the same procedures as those for the determination of factor described in 4. Conduct a blank test separately, in the same manner. In the blank test, however, when potassium permanganate is added, sucrose is also added to eliminate the color.

Note 5) In case of lump meat samples, specimens are usually cut away from a center portion of them. However, as for samples not appropriate to apply general sampling methods, for example, due to inhomogeneous distributions of salt, specimens may be collected from the surface layers or the inner layers of the samples in the same manner as necessary. The collected specimens should be analyzed individually

without being mixed. Furthermore, in collecting specimens, fatty layers should be removed.

Note 6) If over 10 % of salt is contained, a larger amount of the solution should be added.

Note 7) Generally, 10 minutes will be sufficient.

6. Calculation of Salt concentration

$$\text{Salt},\% = \frac{(V0 - V1) \times F \times 0.005845 \times 100}{S}$$

Where —

V0 : titration volume of 0.1 mol/L ammonium thiocyanate standard solution for blank (mL)

V1 : titration volume of 0.1 mol/L ammonium thiocyanate standard solution for test sample (mL)

F : factor of 0.1 mol/L ammonium thiocyanate solution

S : amount of collected sample (g)

Round off fractions to the nearest tenth.

7. References

(1) AOAC 24,007 (1975).

(2) AOAC 50,027 (1975).

(3) AOAC 18,031 (1975).

(4) Methods of Analysis in Health Science, Kanehara-shuppan (1973) (in Japanese).

(5) 高木誠司 「定量分析の実験と計算」共立出版 (1969).