

Method of Test for Sodium Ferrocyanide in Salt

1. Scope

This method is applicable to the determination of sodium ferrocyanide, potassium ferrocyanide, and calcium ferrocyanide in salt.

2. Method

After dissolution and coloring, analytes are determined by spectrophotometer.

2.1. Equipment

2.1.1. Spectrophotometer: with the visible wavelength range.

2.2. Chemicals

Ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), sodium chloride and sulfuric acid, reagent grade;

Deionized water, resistivity $\geq 18 \text{ M}\Omega \cdot \text{cm}$ (at 25°C);

Sodium ferrocyanide [$\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}$], reference standard.

2.3. Apparatus and materials:

2.3.1. Volumetric flask: 50 mL and 100 mL.

2.3.2. Centrifuge tube: 50 mL, PP.

2.3.3. Membrane filter: $0.45 \mu\text{m}$, Nylon.

2.4. Reagents:

2.4.1. Ferrous sulfate solution

Dissolve 3 g of ferrous sulfate with 80 mL of deionized water. Add 1 mL of sulfuric acid and dilute with deionized water to 100 mL. Prepare before use.

2.5. Standard solution preparation

Accurately weigh equivalent to 100 mg of sodium ferrocyanide reference standard into a 100 mL volumetric flask. Dissolve and dilute with deionized water to volume as the standard stock solution. Store in a refrigerator and protect from light. When to use, mix appropriate volume of the standard stock solution, and dilute with deionized water to $100 \mu\text{g/mL}$ as the standard solution.

2.6. Standard curve

Dissolve 10 g of sodium chloride with 40 mL of deionized water into each 50-mL volumetric flask, and add 0.2~5 mL of standard solution separately as the standard curve solutions. Add 2.5 mL of ferrous sulfate solution and dilute with deionized water to 50 mL. Mix thoroughly and allow to stand for 30 minutes. Determine the absorbance with spectrophotometer at 720 nm wavelength. Establish the standard curve of sodium ferrocyanide by the absorbance vs.

the added concentrations of standard solution (0.4~10 µg/mL).

2.7. Sample solution preparation

Transfer about 10 g of sample accurately weighed into a centrifuge tube and dissolve with 40 mL of deionized water. Filter with a membrane filter. Collect the filtrate. Rinse the centrifuge tube with 5 mL of deionized water and filter with a membrane filter. Combine the filtrate as the sample solution.

2.8. Assay

Add 2.5 mL of ferrous sulfate solution into the sample solution and dilute to 50 mL with deionized water. Mix thoroughly and allow to stand for 30 minutes. Determine the absorbance at 720 nm wavelength. Calculate the amount of sodium ferrocyanide in the sample by the following formula:

$$\text{The amount of sodium ferrocyanide in the sample (mg/kg)} = \frac{C \times V}{M}$$

C: the concentration of sodium ferrocyanide in the sample solution calculated by the calibration curve (µg/mL)

V: the final make-up volume of the sample (50 mL)

M: the weight of the sample(g)

Remark

1. Limit of quantification (LOQ) of this method is 2 mg/kg.
2. Further validation should be performed when interfering compounds are found in the samples.

References

Ministry of Health, Labour and Welfare. 2002. Regarding the ministerial ordinance to partially revise the Food Sanitation Law Enforcement Regulations and the matter to partially revise the standards for foods, additives, etc. Ministry of Health, Labor and Welfare administrative information (August 1, 2002, No.0801001). Tokyo, Japan.

<http://www.ffcr.or.jp/Zaidan/mhwinform.nsf/ab440e922b7f68e2492565a700176026/16f9c057678d235849256c0f000688b4?OpenDocument>