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Replacing GB/T 13025.10-2003

General Test Method in Salt Industry - Determination of Ferrocyanide

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Standardization Administration of PRC.

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Foreword

This Part is one of the serial standards for general test method in salt industry; currently, the serial standards can be divided into the following 13 parts; other test method standards shall be supplemented in the follow-up work.

--- GB/T 13025.1 General Test Method in Salt Industry – Determination of Grain Size;

--- GB/T 13025.2 General Test Method in Salt Industry – Determination of Whiteness;

--- GB/T 13025.3 General Test Method in Salt Industry – Determination of Moisture;

--- GB/T 13025.4 General Test Method in Salt Industry – Determination of Insoluble Matter;

--- GB/T 13025.5 General Test Method in Salt Industry – Determination of Chloride Ion;

--- GB/T 13025.6 General Test Method in Salt Industry – Determination of Calcium and Magnesium;

--- GB/T 13025.7 General Test Method in Salt Industry – Determination of Iodide Ion;

--- GB/T 13025.8 General Test Method in Salt Industry – Determination of Sulfate;

--- GB/T 13025.9 General Test Method in Salt Industry – Determination of Lead;

--- GB/T 13025.10 General Test Method in Salt Industry – Determination of Ferrocyanide;

--- GB/T 13025.11 General Test Method in Salt Industry – Determination of Fluoride;

--- GB/T 13025.12 General Test Method in Salt Industry – Determination of Barium Ion;

--- GB/T 13025.13 General Test Method in Salt Industry – Determination of Arsenic.

This Part is Part 10 of GB/T 13025.

This Part was drafted as per the rules specified in GB/T 1.1-2009.

This Part replaces GB/T 13025.10-2003 *General Test Method in Salt Industry – Determination of Potassium Ferrocyanide*; this Part made the editorial medication to GB/T 13025.10-2003.

General Test Method in Salt Industry - Determination of Ferrocyanide

1 Scope

This Part of GB/T 13025 specifies the test method of determining the ferrocyanide in the edible salt.

The ferrous sulfate method in this Part is applicable to the determination of samples with a ferrocyanide content of 1mg/kg above; while the pyridine-pyrazolone method is applicable to the determination of samples with a ferrocyanide content of 1mg/kg below.

2 Normative References

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this document.

GB/T 6682-2008 Water for Analytical Laboratory Use - Specification and Test Methods

3 Ferrous Sulfate Method

3.1 Principle

Under acidic conditions, ferrocyanide reacts with ferrous sulfate t o form a white precipitate of ferrous ferrocyanide, which is oxidized by air to form Prussian blue and determined by spectrophotometry.

3.2 Reagents

3.2.1 Reagent specification

Unless otherwise stated, only analytically pure reagents and Class-III water specified in GB/T 6682-2008 are used in the analysis.

3.2.2 Sodium chloride

Take 50g of sodium chloride; place into high temperature furnace; burn it at 800°C for

30min; cool it off for later-use.

3.2.3 Sulfuric acid solution (1+20)

Take 5mL of concentrated sulfuric acid; slowly place it into 100mL of water; then mix well.

3.2.4 40g/L ferrous sulfate solution

Take 4g of ferrous sulfate ($FeSO_4•7H_2O$); dissolve it in 100mL of sulfuric acid solution (3.2.3); filter it; store in the brown reagent bottle at low temperature.

3.2.5 Potassium ferrocyanide standard stock solution (1mL of solution contains 1.0mg $[Fe(CN)_6]^{4-}$)

Accurately take 0.1993g of potassium ferrocyanide ($K_4[Fe(CN)_6]•3H_2O$); add a small amount of water to dissolve; dilute to 100mL.

3.2.6 Potassium ferrocyanide standard working solution (1mL of solution contains 50μg $[Fe(CN)_6]^{4-}$)

Pipette 5.00mL of potassium ferrocyanide standard stock solution (3.2.5); dilute to 100mL.

3.3 Instruments

3.3.1 Spectrophotometer.

3.3.2 General laboratory instruments.

3.4 Analytical procedures

3.4.1 Determination of the limit method

Pipette the potassium ferrocyanide standard working solution (3.2.6) corresponding to the limited amount into 50mL colorimetric tube; add 5g of sodium chloride (3.2.2); add water to dissolve; add 4mL of ferrous sulfate solution (3.2.4); add water to dilute to the scale; shake evenly; stand for 10min; take reagent blank as a reference at the wavelength of 670nm; then measure the absorbance. Take another 50mL colorimetric tube; take 5.0g of specimen into the colorimetric tube; add 40mL of water to dissolve (if the solution is turbid, filter with 0.45μm filter membrane); add 4mL of ferrous sulfate solution (3.2.4); add water to dilute to the scale; shake evenly; stand for 10min; then take reagent blank as a reference at the wavelength of 670nm; then measure the absorbance. If the absorbance of the specimen solution is lower than that of the standard solution, then it is qualified; otherwise, it is disqualified.

3.4.2 Determination of working curve method

4 Pyridine-Pyrazolone Method

4.1 Principle

The ferrocyanide is decomposed into cyanide under acidic conditions; reacts with chloramine T to form cyanogen chloride (CNCl); then blue dye is formed with pyridine-pyrazolone; then photometry is used to measure.

4.2 Reagent

4.2.1 Pyridine (GB/T 689)

4.2.2 Chloramine T solution (10g/L)

Take 1.0g of chloramine T; dissolve into 100mL of water.

4.2.3 Phosphate buffer solution

Take 17.8g of anhydrous disodium hydrogen phosphate and 17.0g of anhydrous potassium dihydrogen phosphate; add water to dissolve and dilute to 500mL.

4.2.4 Sodium hydroxide solution (0.1mol/L)

Take 1.0g of sodium hydroxide; dissolve into 250mL of water.

4.2.5 Acetic acid solution (1mol/L)

Pipette 11.5mL of acetic acid; add water to 200mL; mix evenly.

4.2.6 Tartaric acid solution (10.0g/L)

Take 20.0g of tartaric acid; dissolve into 200mL of water; filter it.

4.2.7 Standard solution

4.2.7.1 Standard stock solution

Take 10.0g of sodium chloride (3.2.2); add 100mL of water to dissolve; add 4.0mL of potassium ferrocyanide standard working solution (3.2.6); mix evenly; transfer into 500mL distillation flask; add 10mL of tartaric acid solution (4.2.6); quickly connect the condenser to start distillation. Use 100mL volumetric flask containing 10mL of sodium hydroxide solution (4.2.4) to collect the distillate; maintain the solution boiling in the distillation flaks for 10min. After cooling off, take off the volumetric flask; add water to the scale; shake evenly. 1mL of such solution contains 2.0μg $[Fe(CN)_6]^{4-}$.

4.2.7.2 Standard working solution

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