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National food safety standard - Determination of Cyanide in Foods

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1 Scope

This Standard specifies the method for the determination of cyanide in foods.

The first method "spectrophotometry" is applicable to the determination of cyanide in distilled liquor and its integrated alcoholic beverages, edible alcohol, tapioca flour, packaged drinking water and mineral water.

The second method "gas chromatography" and the third method "gas chromatography - mass spectrometry" are applicable to the determination of cyanide in liquor, edible alcohol, grain, packaged drinking water and mineral water.

The fourth method "ion chromatography" is applicable to the determination of cyanide in distilled liquor and its integrated alcoholic beverages, edible alcohol, packaged drinking water, mineral water and beverages (based on almonds).

The fifth method "flow injection / continuous flow - spectrophotometry" is applicable to the determination of cyanide in distilled liquor and its integrated alcoholic beverages, edible alcohol, packaged drinking water and mineral water.

Method I - Spectrophotometry

2 Principle

Heat the specimen under alkaline conditions to remove high-boiling-point organic matters,

or heat and distill it under acidic conditions. After using sodium hydroxide solution to dissolve or absorb it, under the condition of pH = 7.0, use chloramine T to convert cyanide to cyanogen chloride, then, react it with isonicotinic acid-pyrazolone to generate a blue dye, which can be quantitatively compared with the standard series.

3 Reagents and Materials

Unless it is otherwise specified, the reagents used in this Method are all analytically pure, and the water is Grade-3 water specified in GB/T 6682.

3.1.1 Absolute ethanol (C2H6O).

distillation device (for water cooling, a condenser is required to adjust the temperature to 10 ?C ~ 15 ?C for distillation), insert the lower end of the condenser tube under the liquid level of a 100 mL volumetric flask containing 10 mL of sodium hydroxide solution (20 g/L) (if the room temperature is higher than 25 ?C, use an ice pack to cool it down, so that the temperature of the distillate is between 20 ?C ~ 25 ?C), and perform distillation. When the distillate collected is nearly 100 mL, use water to reach a constant volume to the scale. Accurately draw 1.00 mL of the solution into a 10 mL colorimetric tube, add 2 g/L of sodium hydroxide solution to 5 mL, shake it well, and use it as a specimen solution.

5.1.1.2 Add 2 drops of phenolphthalein indicator solution to the specimen solution, add acetic acid solution to adjust it, until the red color fades, then, use 20 g/L sodium hydroxide solution to adjust it to nearly red color. Add 2 mL of phosphate buffer solution, then add 0.2 mL of chloramine T solution, shake it well and let it stand for 3 min. Add 2 mL of isonicotinic acid-pyrazolone solution, add water to reach a constant volume to the scale. Add a stopper and shake to evenly mix it. Place it in a constant-temperature water bath at 37 ?C for 40 min and reserve it for testing.

5.1.2 Distilled liquor and its integrated alcoholic beverages, edible alcohol

5.1.2.1 Accurately draw 1.00 mL of specimen (for sesame-flavor liquor, Maotai-flavor liquor and special-flavor liquor specimens, draw 0.10 mL) into a 25 mL beaker, add 5 mL of sodium hydroxide solution (2 g/L), and leave it for 10 min. Then, place it on an electric heating plate at 160 ?C ~ 180 ?C and heat it at low temperature, until about 1 mL of the solution remains.

Remove it and place it at room temperature. Use 2 g/L sodium hydroxide solution to transfer it to a 10 mL colorimetric tube with a stopper. Finally, add 2 g/L sodium hydroxide solution to 5 mL.

5.1.2.2 If the liquor specimen is turbid or colored, take 25.0 mL of specimen into a 250 mL distillation flask, add 100 mL of water, add a few glass beads, and dropwise add a few drops of methyl orange indicator. Insert the lower end of the condenser tube under the liquid level of the colorimetric tube containing 10 mL of sodium hydroxide solution (20 g/L),

add 1 g ~ 2 g of tartaric acid, the solution changes from orange to orange-red. Quickly connect the distillation device to perform distillation, and collect nearly 50 mL of the distillate, then, use water to reach a constant water of 50 mL, and evenly mix it. Take 2.00 mL of the distillate and operate in accordance with 5.1.2.1.

5.1.2.3 Same as 5.1.1.2.

5.1.3 Packaged drinking water and mineral water

5.1.3.1 Measure-take 250 mL of water specimen (when the cyanide content exceeds 20 ?g, take an appropriate amount of water specimen and add water to 250 mL) and place it into a 500 mL distillation device. Add 1 ~ 2 drops of methyl orange indicator, then, add 5 mL of zinc acetate solution, and add 1 g ~ 2 g of tartaric acid, the solution changes from orange to orange-red.

Quickly connect the distillation device, insert the lower end of the condenser tube under the liquid level of a 50 mL colorimetric tube with a stopper containing 5.0 mL of sodium hydroxide solution (20 g/L). By adjusting the temperature, control the distillation speed at 2 mL/min ~ 3 mL/min, collect nearly 50 mL of the distillate, then, use water to reach a constant water of 50 mL, and evenly mix it. Take 10.0 mL of the distillate and place it in a 25 mL colorimetric tube with a stopper.

5.1.3.2 Add 5.0 mL of phosphate buffer solution to the colorimetric tube containing the specimen, place it in a constant-temperature water bath at 37 ?C, then, add 0.25 mL of chloramine T solution, add a stopper to mix it, leave it for 5 min. Then, add 5.0 mL of isonicotinic acid-pyrazolone solution, add water to 25 mL and mix it well. Place it in a constant-temperature water bath at 37 ?C for 40 min and reserve it for testing.

5.2 Drawing of Standard Working Curve

5.2.1 Standard curve of tapioca flour Respectively and accurately draw 0 mL, 0.40 mL, 0.80 mL, 1.20 mL, 1.60 mL and 2.00 mL of cyanide ion standard solution (1.0 ?g/mL) into a 10 mL colorimetric tube [equivalent to the mass of cyanide (CN?): 0 ?g, 0.40 ?g, 0.80 ?g, 1.20 ?g, 1.60 ?g and 2.00 ?g], add 2 g/L sodium hydroxide solution to 5 mL. In accordance with 5.1.1.2, conduct synchronous operation with the sample below. Take the prepared standard curve solution to be tested, use a 1 cm cuvette, and take 2 g/L sodium hydroxide solution as the blank solution to adjust the zero point. At a wavelength of 638 nm, determine the absorbance; take the mass of cyanide ions as the x-coordinate and the absorbance of cyanide ions as the y-coordinate, and draw a standard curve.

5.2.2 Standard curve of distilled liquor and its integrated alcoholic beverages, edible alcohol Respectively and accurately draw 0 mL, 0.40 mL, 0.80 mL, 1.20 mL, 1.60 mL and 2.00 mL of cyanide ion standard solution (1.0 ?g/mL) into a 50 mL beaker [equivalent to the mass of cyanide (CN?): 0 ?g, 0.40 ?g, 0.80 ?g, 1.20 ?g, 1.60 ?g and 2.00 ?g], add 5 mL of sodium hydroxide solution (2 g/L) and leave it for 10 min. In accordance with 5.1.2.1 and 5.1.2.3,