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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 29708-2013

**Determination of Sodium Pentachlorophenol residues in animal
derived food by Gas Chromatography-mass Spectrometric
method**

食品安全国家标准 动物性食品中五氯酚钠残留量的测定 气相色谱-质谱法

(English Translation)

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1 Scope

This standard specifies the sample preparation and gas chromatography-mass spectrometry in animal foods sodium pentachlorophenol residues detected.

This standard applies to porcine muscle, sodium pentachlorophenol residues in chicken liver and kidneys and muscle and liver tissue detection.

2 Normative references

The following documents for the application of this document is essential. For dated references, only applies to the version dated paper Pieces. For undated references, the latest edition (including any amendments) applies to this document.

GB/T 6682 Water for analytical laboratory specifications and test methods Principle 3 the sample of residual sodium pentachlorophenol, trichloroacetic acid precipitated protein, cyclohexane - ethyl acetate solution was extracted, acetic anhydride - pyridine derivative, Gas Chromatography - Mass Spectrometry, external standard.

4 Reagents and materials

The following reagents used, unless otherwise stated were of analytical reagent; water as a water line with GB/T 6682 provisions.

4.1

sodium pentachlorophenol Standard. content $\geq 98.0\%$.

4.2

potassium carbonate.

4.3

n-hexane.

4.4

cyclohexane.

4.5

ethyl acetate.

4.6

of acetic anhydride.

4.7

pyridine.

4.8

TCA.

4.9

concentrated hydrochloric acid.

4.10 5% trichloroacetic acid solution. Take acid solution 5g, dissolve and dilute to 100mL with water.

4.11 2.4mol/L hydrochloric acid solution. Take 20mL of concentrated hydrochloric acid was dissolved and diluted to 100mL with water.

4.12

cyclohexane - ethyl acetate. ethyl take 10mL, add cyclohexane 40mL, mix.

4.13

acetic anhydride - pyridine. acetic anhydride take 10mL, add pyridine 10mL, mix, now with the current.

4.14 0.2mol/L potassium carbonate solution. Take potassium carbonate 27.6g, dissolved

and diluted with water to 1000mL.

4.15 1mg/mL standard stock solution pentachlorophenol sodium. pentachlorophenol accurately weighed standard 10mg, in 10mL volumetric flask, and dissolved in water Diluted to the mark, formulated at a concentration of 1mg/mL of stock standard solution of sodium pentachlorophenol. The following stored at -20 °C, valid for three months.

4.16 2µg/mL sodium pentachlorophenol standard working solution. precise amount of 1mg/mL standard stock solution of sodium pentachlorophenol 200µL, in an amount of 100mL Flask was diluted with water to the mark, formulated at a concentration of 2µg/mL of sodium pentachlorophenol working standard solution, now with the current.

5 Apparatus

5.1

gas chromatograph mass spectrometer. with the EI source.

5.2

Analytical balance. a sense of volume 0.00001g.

5.3

Balance. a sense of the amount of 0.01g.

5.4

high-speed centrifuge.

5.5

oscillator.

5.6

vortex mixer.

5.7

tissue homogenizer.

5.8

Nitrogen blowing instrument.

5.9

rotary evaporator.

5.10

centrifuge tube. 50mL.

Preparation and Storage of sample 6

6.1 Preparation of the sample

Fresh or thawed take appropriate blank or test tissue, minced, and homogenized.

--- homogeneous test sample taken, as the feed try.

--- the blank sample were homogeneous, as a blank sample.

--- blank sample taken homogeneous, standard working solution is added an appropriate concentration, is added as a blank sample.

Save 6.2 sample

-20 °C less storage backup.

Determination Step 7

7.1 Preparation of matrix-matched standard curve

Accurately taken 2µg/mL sodium pentachlorophenol appropriate amount of working standard solution, prepared with water to a concentration of 2,5,10,20,50,100µg/L series Standard solution from each 1.0 mL, extracted separately dissolved, after drying 6 parts distilled white residue sample, mixed, dissolved with 2.4mol/L hydrochloric acid PH was adjusted to less than 2.0, add cyclohexane - ethyl acetate solution 5mL, vortexed, 8000r/min centrifugal 10min, the organic layer was pipetted and then Adding cyclohexane - ethyl acetate solution 5mL, the organic layer was twice vortex mixed, 8000r/min centrifugal 10min, combined, rotary evaporated at 45 °C dry. By derivatization steps, gas chromatography mass spectrometry. A characteristic ion mass chromatogram peak area for the vertical axis, the concentration control solution of Abscissa, the standard curve. Seeking regression equation and correlation coefficient.

7.2 Extract

Sample Weigh 5g ± 0.05g, in 50mL centrifuge tubes, add water 10mL, Vortex, 8000r/min centrifugal 10min, supernatant Solution, 5% trichloroacetic acid was added 5mL, Vortex, 8000r/min centrifugal 10min, the supernatant was adjusted to pH with 2.4mol/L hydrochloric acid solution to less than 2.0, add cyclohexane - ethyl acetate solution 5mL, vortexed, 8000r/min centrifugal 10min, the organic layer was pipetted, cyclohexyl plus Alkoxy - ethyl acetate solution 5mL, vortexed, 8000r/min centrifugal 10min, the two organic layers were combined, rotary evaporated to dryness at 45 °C.

7.3 Derivatized

The residue was added cyclohexane - ethyl acetate, 1.0 mL, was added a small amount of anhydrous sodium sulphate, was added acetic anhydride - pyridine solution, 100 L, sealed and 60 °C reaction 15min, cooled and 0.5 mL potassium carbonate solution was added,