

ICS 67.020

CCS C 53



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

GB 29703-2013

**Determination of Sodium nifurstyrenate residues in animal
derived food by Liquid Chromatography-tandem Mass
Spectrometric method**

食品安全国家标准 动物性食品中呋喃苯烯酸钠残留量的测定 液相色谱-串联质谱
法

(English Translation)

Issued on: September 16, 2013

Implemented on: January 1, 2014

**Issued by: Ministry of Agriculture of the People's Republic of China,
National Health and Family Planning Commission of the People's
Republic of China**

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1 Scope

This standard specifies the animal food furan benzene acid sodium Residues sample preparation and liquid chromatography - tandem mass spectrometry.

Muscle and liver tissue This standard applies to pigs and chickens furan residual amounts of benzene acid sodium 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 Residual benzene in a sample furanyl acid sodium, 0.5% formic acid - ethyl acetate extraction, solid-phase extraction (SPE), liquid chromatography - tandem mass spectrometry ESI positive ion mode measurement, external standard.

4 Reagents and materials

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

4.1

furanyl benzene sodium acid standard. content of $\geq 98.0\%$.

4.2

methanol.

4.3

n-hexane.

4.4

Acetonitrile. chromatographically pure.

4.5

ethyl acetate.

4.6

formic acid.

4.7

HLB solid phase extraction column. 60mg/3mL, or equivalent person.

4.8 0.5% formic acid solution. Take acid 0.5mL, dissolve and dilute to 100mL with water.

4.9 0.5% formic acid - ethyl acetate solution. Take 200 mL of ethyl acetate, 0.5% formic acid solution was added 10mL, mix, now with the current.

4.10 1mg/mL sodium furan benzene acid standard stock solution. Weigh accurately furan-benzene standard acid sodium 10mg, in 10mL flask, Dissolved in dimethyl sulfoxide and diluted to the mark, formulated at a concentration of 1mg/mL of benzene, furan, sodium acid standard stock solution. $-20\text{ }^{\circ}\text{C}$ save less, Valid for six months.

4.11 10 μg /mL sodium acid benzyl furan-standard working solution. precise amount of 1mg/mL sodium furan benzene acid standard stock solution 1.0mL, in 100mL volumetric flask, dissolved in mobile phase and dilute to volume, formulated at a concentration of 10 μg /furan benzene acid sodium working standard solutions mL.

$-20\text{ }^{\circ}\text{C}$ below, valid for six months.

5 Apparatus

5.1

liquid chromatography - tandem mass spectrometry. electrospray ionization with (ESI).

5.2

Analytical balance. a sense of volume 0.00001g.

5.3

Balance. a sense of the amount of 0.01g.

5.4

freezing high-speed centrifuge.

5.5

oscillator.

5.6

vortex mixer.

5.7

homogenizer.

5.8

Nitrogen blowing instrument.

5.9

rotary evaporator.

5.10

centrifuge tube. 50mL.

5.11

eggplant-shaped bottle.

5.12

filter. 0.22 μ m.

Preparation and Storage of sample 6

6.1 Preparation of the sample

Fresh or frozen blank or test tissue, minced, and homogenized.

--- the test sample taken after homogenization, as the test material.

--- blank sample taken after homogenization, as a blank material.

--- blank sample taken after homogenization, adding a suitable concentration of the standard working solution, is added as a blank sample.

Save 6.2 sample

Or less at -20 °C.

Determination Step 7

7.1 Extract

Take Sample $2g \pm 0.05g$, in 50mL centrifuge tubes, add 0.5% formic acid - ethyl acetate solution 15mL, vortexed, shaken 5min, 8000r/min centrifugal 10min, the ethyl acetate layer eggplant-shaped flask, the residue was added 0.5% formic acid - ethyl acetate solution 10mL, heavy Re-extracted once, twice with ethyl acetate layers were combined, rotary evaporated at 45 °C to dryness, treated with 50% aqueous acetonitrile and the residue was dissolved 3mL, go Centrifuge tube with 3mL washed with 50% aqueous acetonitrile eggplant-shaped flask, two washings were combined, hexane was added 3mL, shaking after mixing, 5000r/min centrifugal 5min, the lower layer was taken and set aside.

7.2 Purification

HLB column eluted sequentially with methanol and 3mL 3mL water activation, taking stock solution through the column. 6mL water rinsed, drained, washed 3mL of acetonitrile was added Off, the eluate was collected, dried under nitrogen 50 °C, the mobile phase the residue was dissolved 1.0mL, 1.5mL transferred to centrifuge tube, 10000r/min Centrifugal 10min, supernatant, membrane filtration, liquid chromatography - tandem mass spectrometry.

7.3 Preparation of standard curve

They were the precise amount of 10μg/mL sodium acid benzyl furan-standard working fluid amount, diluted with mobile phase to prepare a concentration of 0.2,0.5,2, 5 and 8μg/L series of standard solutions, the liquid chromatography - tandem mass spectrometry. A characteristic ion mass chromatogram peak area for the vertical axis, standard solutions Concentration as the abscissa, the standard curve.

7.4.1 LC Conditions

7.4.1.1

Column. C18 (150mm x 2.1mm, particle size 5μm), or equivalent person.