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

GB 29706-2013

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

食品安全国家标准 动物性食品中氨苯砒残留量的测定 液相色谱-串联质谱法

(English Translation)

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1 Scope

This standard specifies the animal food dapsone Residues sample preparation and liquid chromatography - tandem mass spectrometry.

Residues in muscle and liver tissue dapsone is applicable to the porcine and bovine.

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.

Specifications and test methods for laboratory water GB/T 6682 analysis.

Principle 3

Dapsone remaining in a sample, extracted with acetonitrile, hexane degreasing, MCX, purified by liquid chromatography - tandem mass spectrometry, the external standard method Quantitative.

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

dapsone Standard. Content $\geq 98\%$.

4.2

Acetonitrile. chromatographically pure.

4.3

n-hexane.

4.4

concentrated ammonia.

4.5

hydrochloric acid.

4.6

MCX SPE. 60mg/3mL, or equivalent person.

4.7 1mol/L hydrochloric acid solution. Take 8.4mL of concentrated hydrochloric acid was dissolved and diluted to 100mL with water.

4.8 0.1mol/L hydrochloric acid solution. 0.84mL take concentrated hydrochloric acid, dissolve and dilute to 100mL with water.

4.9 5mmol/L ammonium acetate solution. Take 0.385 g ammonium acetate, dissolved and diluted to 1000mL with water, membrane filtration.

4.10 5% ammoniated acetonitrile. concentrated aqueous ammonia Take 5mL, dissolved in acetonitrile and diluted to 100mL.

4.11 1mg/mL dapsone standard stock solution. Weigh accurately dapsone 10mg, in 10mL volumetric flask, dissolved in methanol and diluted to the mark with Degree, formulated as dapsone stock standard solution at a concentration of 1mg/mL of. -20°C dark preservation, valid for six months.

4.12 10 μg /mL dapsone standard working solution. precise amount of 1mg/mL standard stock solution 0.1 mL dapsone, in 10mL volumetric flask, Dilute with methanol, formulated as dapsone standard working concentration of 10 μg /mL of. -20°C dark preservation, valid for six months.

4.13 100ng/mL dapsone standard working solution. precise amount of 10 μg /mL dapsone

working standard solution 0.1mL, in 10mL volumetric flask , Diluted with methanol to scale, formulated at a concentration of 100ng/mL dapson the standard working solution. -20 °C from light, valid 6 months.

5 Apparatus

5.1

liquid chromatography - tandem mass spectrometry. equipped with an ESI source.

5.2

Analytical balance. a sense of volume 0.00001g.

5.3

Balance. a sense of the amount of 0.01g.

5.4

centrifuge.

5.5

nitrogen blow device.

5.6

vortex mixer.

5.7

SPE.

5.8

ultrasonic cleaning machine.

5.9

homogenizer.

5.10

filter. 0.22μm.

5.11

centrifuge tube. 50mL.

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 feed try.

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

--- 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

Sample Weigh $2\text{g} \pm 0.05\text{g}$, a centrifuge tube, acetonitrile was added 10mL, vortexed 2min, n-hexane was added 5mL, vortexed 1min, ultrasonic 5min, 5000r/min centrifugal 10min, discard the upper layer of n-hexane solution. Acetonitrile was taken to another centrifuge tube, was added 1mol/L Hydrochloric acid solution 10mL, Vortex, spare.

7.2 Purification

MCX column washed with 5mL methanol and 5mL water activation, taking stock solution through the column, controlling the flow rate of less than 1mL/min, with 0.1mol/L 5mL methanol and 5mL solution of hydrochloric acid rinsed, drained, eluting with 5% ammoniated 5mL acetonitrile, dry nitrogen at 40 °C water bath, mobile phase the residue was dissolved 1.0mL, Vortex for liquid chromatography - tandem mass spectrometry.

7.3 Preparation of standard curve

The precise amount of 100ng/mL working standard solution amount dapsons, diluted with methanol, concentrations were formulated and 0.5,1,2,5,10 20µg/L standard solution series. For liquid chromatography - tandem mass spectrometry. A characteristic ion mass chromatogram peak area for the vertical axis, control solution Concentration as the abscissa, the standard curve, seeking regression equation and correlation coefficient.

7.4.1 LC Conditions

7.4.1.1

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

7.4.1.2

mobile phase. acetonitrile 5mmol/L ammonium acetate (50 50, volume ratio).

7.4.1.3

Column temperature. 40 °C.