

ICS 67.020
CCS C 53



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

GB 29705-2013

**Determination of Cypermethrin, Fenvalerate and Deltamethrin
residues in aquatic products by Gas Chromatographic method**

食品安全国家标准 水产品中氯氰菊酯、氰戊菊酯、溴氰菊酯多残留的测定 气相
色谱法

(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 aquatic cypermethrin, fenvalerate and deltamethrin residues sample preparation and gas chromatographic method.

This standard applies to edible tissue of fish and shrimp detecting cypermethrin, fenvalerate and deltamethrin residues.

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 specimens remaining cypermethrin, fenvalerate and deltamethrin, dehydrated with sodium chloride, extracted with acetonitrile, hexane degreasing, C18 column purification, Determined by gas chromatography, 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

cypermethrin, fenvalerate, deltamethrin standard. content $\geq 99.0\%$.

4.2

Acetonitrile. chromatographically pure.

4.3

Methanol. HPLC grade.

4.4

hexane. chromatography.

4.5

Benzene. chromatography.

4.6

ethyl acetate. chromatography.

4.7

chloroform. HPLC grade.

4.8

Sodium chloride. excellent pure, 650 °C burning 4h, cooled, dried and stored for use.

Aqueous methanol 4.9. Methanol 100mL, dissolved and diluted to 300mL with water.

4.10

n-hexane solution in acetonitrile. take 10mL, was dissolved with n-hexane and diluted to 110mL.

4.11

C18 solid phase extraction column. 200mg/3mL, or equivalent person.

4.12

alumina SPE. 200mg/3mL, or equivalent person.

4.13 100 μ g/mL cypermethrin, fenvalerate and deltamethrin standard stock solution. Weigh accurately cypermethrin, fenvalerate and bromine Cyhalothrin each standard 10mg, brown in 100mL flask, dissolve and dilute benzene, formulated at a concentration of 100 μ g/mL of Mixed standard stock solution. 2 °C ~ 8 °C storage period of three months.

4.14 5µg/mL cypermethrin, fenvalerate and deltamethrin working standard solutions. precise amount of 100µg/mL cypermethrin, pentyl cyanide Permethrin and deltamethrin 500 L standard stock solution, brown in 10mL volumetric flask, dilute to the mark with benzene, formulated at a concentration of 5µg/mL of cypermethrin, fenvalerate and deltamethrin working standard solutions. 2 °C ~ 8 °C storage period of three months.

5 Apparatus

5.1

Gas chromatograph. 63Ni with an electron capture detector.

5.2

Analytical balance. a sense of volume 0.00001g.

5.3

Balance. a sense of the amount of 0.01g.

5.4

homogenizer.

5.5

rotary evaporator.

5.6

centrifuge.

5.7

speed shaker.

5.8

Nitrogen blowing instrument.

5.9

SPE.

5.10

heart-shaped bottle. 125mL.

5.11

stoppered centrifuge tube. 10mL, 50mL.

Preparation and Storage of sample 6

6.1 Preparation of the sample

Take appropriate blank or fresh or thawed fish and shrimp test edible 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 $5\text{g} \pm 0.05\text{g}$, in 50mL glass centrifuge tube with stopper, acetonitrile was added 15mL, shaking 5min, sodium chloride 1.5g vibrator Swing 2min, 4000r/min centrifugal 5min, supernatant to another glass stoppered centrifuge tube, the residue was added acetonitrile 15mL, extraction was repeated Once, twice supernatants were combined, acetonitrile, n-hexane solution was added 10mL, shaking mixing 2min, 4000r/min centrifugal 5min, the upper layer was removed Hexane solution, acetonitrile, n-hexane solution was added 10mL, extraction was repeated once, the upper n-hexane was discarded. The acetonitrile layer was taken to heart-shaped flask, to 45 °C water bath rotary evaporated to dryness, the residue was dissolved in methanol 2mL, 4 mL water was added, mixed and set aside.

7.2 Purification and concentration

5mL C18 column successively with methanol, 5mL of chloroform, methanol and aqueous methanol 5mL 5mL activation, taking stock solution through the column, the control Flow rate of 1 drop/s. 5mL rinsed with aqueous methanol, dried stream, Benzene was added 4mL, collecting eluate standby.

5mL activated alumina column with acetonitrile, the eluent through the column to take backup. Benzene was added 1mL elution, is repeated three times. Three eluent to merge 10mL graduated centrifuge tube, with the volume of benzene to 5mL, gas phase chromatography.