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

GB 29707-2013

**Determination of marker residues of Amitraz in milk by Gas
Chromatographic method**

食品安全国家标准 牛奶中双甲脒残留标志物残留量的测定 气相色谱法
(English Translation)

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1 Scope

This standard specifies the sample preparation and gas chromatographic method for the detection of residues in milk amitraz marker.

This standard applies to methyl aniline residues in milk amitraz markers 2,4.

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 amitraz, extracted with aqueous sodium hydroxide, hydrolysis, extraction, derivatization heptafluorobutyric anhydride, gas chromatography - electron capture detection Detection measurement method, 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

pairs formamidine reference. content $\geq 99\%$; 2,4- dimethylaniline reference. content of $\geq 95\%$.

4.2

Sodium hydroxide. pure class.

Sodium bicarbonate 4.3. excellent pure.

Anhydrous sodium sulfate 4.4. excellent pure.

4.5

heptafluorobutyric anhydride. chromatography.

4.6

hexane. chromatography.

4.7 1mol/L sodium hydroxide solution. take sodium hydroxide 40g, dissolved and diluted with water to 1000mL.

4.8

alkaline aqueous solution. water 1000mL, with 1mol/L sodium hydroxide solution was adjusted to pH 9.0.

4.9

with saturated sodium bicarbonate solution. Take bicarbonate 10.35g, dissolved and diluted with water to 1000mL.

4.10 1mg/mL, amitraz and 2,4-dimethylaniline standard stock solution. Weigh accurately amitraz and 2,4-dimethylaniline reference substance 10mg, respectively 10mL volumetric flask, dissolve and dilute with n-hexane to the mark, formulated as amitraz standard stock concentration of 1mg/mL of and 2,4-dimethyl aniline solution standard stock solution. 2 °C ~ 8 °C storage period of three months.

4.11 10 μ g/mL working standard solutions. precise amount of 1mg/mL standard stock solution of amitraz and 2,4-dimethylaniline standard stock 1.0 mL each of the appropriate amount of liquid, in 100mL volumetric flask, diluted to volume with hexane, formulated at a concentration of 10 μ g/mL of the working standard liquid. 2 °C ~ 8 °C from light, one week period.

5 Apparatus

5.1

Gas chromatograph. 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

centrifuge.

5.5

vortex mixer.

5.6

homogenizer.

5.7

Ovens.

5.8

polypropylene centrifuge tube.

5.9

stoppered centrifuge tube.

5.10

round-bottomed flask.

Preparation and Storage of sample 6

6.1 Preparation of the sample

An appropriate amount of fresh or frozen or blank test milk and mix well.

--- 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 Preparation of standard curve

The precise amount of 2,4-dimethylaniline appropriate amount of standard stock solution, diluted with hexane, and formulated to a concentration of 10,20,50,100,200 400ng/mL of the standard solution series, from each 2.0 mL, the processing according to the derivatization step, gas phase chromatography. To the measured peak area for the vertical take Standard, the standard concentration corresponding to abscissa, the standard curve. Seeking regression equation and correlation coefficient.

7.2 Extraction and hydrolysis

Sample Weigh $5g \pm 0.05g$, polypropylene centrifuge tube, the aqueous alkaline solution was added 10mL, Vortex, 4200r/min centrifugal 10min, the supernatant was further polypropylene centrifuge tube, the aqueous alkaline solution added to the residue 10mL, extraction was repeated once, the two supernatants were combined liquid. Oven temperature was allowed to stand at 70 deg.] C in 50min, cooled, hexane was added 10mL, Vortex, standing 5min, 4200r/min centrifugal 10min, the upper n-hexane solution in a round bottom flask, was added 5 mL of n-hexane extraction was repeated once underlayer, the two hexane layer were combined, in 45 °C transferred evaporated to dryness, the residue was dissolved with n-hexane 2.0mL standby.

7.3 Derivatives and purify

Stock solution taken in a stoppered centrifuge tube, add 10 L heptafluorobutyric anhydride, mixed, at 60 deg.] C thermostat 90min, 30min intervals therebetween removed Ultrasound 5min. After standing derived 10min, saturated sodium bicarbonate solution was added 2mL, mixing, the organic layer was taken, dried over anhydrous sodium sulfate was added to 2g Water, gas phase chromatography.

7.4.1 Chromatographic conditions

7.4.1.1

Column. Rtx-1 capillary column (30m x 0.25mm), or equivalent person.

7.4.1.2

Column temperature. programming, see Table 1.

Table 1 Temperature Programmed Table

Heating rate

°C/min