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

GB/T 21926-2008

**Determination of 2-dodecylcyclobutanone in irradiated foods
containing fat -- GC/MS method**

辐照含脂食品中2-十二烷基环丁酮测定 气相色谱-质谱法

(English Translation)

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Foreword

Instead of the standard GB/T 21926-2008 "irradiated food containing fat 2-dodecyl-azetidinone by Gas Chromatography/Mass Spectrometry." as compared with the present standard GB/T 21926-2008, the main changes are as follows.

- standard name changed to "Food Safety Standard irradiated food containing lipid-2-dodecyl cyclobutanone identified by gas chromatography -Mass Spectrometry ";
- Improved silica gel column chromatography.

National Food Safety Standard Identification of lipid-containing irradiated food 2-dodecyl cyclobutanone GC -Mass spectrometry

1 Scope

This standard specifies the use of 2-dodecyl-azetidinone lipid-containing food irradiation identified by gas chromatography - mass spectrometry.

This standard applies to the identification of irradiated food fat content greater than 1%.

Principle 2

After extraction using n-hexane fat sample cyclobutanone and 2-dodecyl out together, purification by silica gel column chromatography, concentrated gas Chromatography - mass spectrometer 2-dodecyl cyclobutanone, external standard.

3 Reagents and materials

Unless otherwise indicated, the reagents used in this method are analytically pure water three water GB/T 6682 provisions.

3.1 Reagent

3.1.1

hexane (C₆H₁₄).

3.1.2

anhydrous sodium sulfate (Na₂SO₄). 650 °C burning 4h, cool in a desiccator, stored for use.

3.1.3

ether (C₄H₁₀O).

3.1.4

nitrogen (N₂). purity ≥99.999%.

3.1.5

helium (He). purity ≥99.999%.

3.2 Material

Silicone. 250μm ~ 500μm, column chromatography. Before use, the activated silica gel placed in an oven at 100 deg.] C 12h, cool in a desiccator. in Stoppered Erlenmeyer flask was added 4% (mass ratio) of water deactivation of the conical flask shaken until uniformly dispersed silica. The deactivated silica gel in a desiccator Ping Heng 12h, available within a week.

3.3 Standard

2-dodecyl cyclobutanone standard (C₁₆H₃₀O). purity ≥99%.

3.4 Standard Preparation

3.4.1 2-dodecyl azetidinone standard stock solution Accurately weighed amount of 2-dodecyl cyclobutanone standard (accurate to 0.1 mg), was dissolved with n-hexane, formulated at a concentration of 100μg/mL of Standard stock solution, -20 °C storage, 3 months effective.

3.4.2 2-dodecyl azetidinone standard working solution Standard stock solution was diluted with hexane (100μg/mL), were formulated as 0.10μg/mL, 0.25μg/mL, 0.50μg/mL, 1.00 Standard working solution levels μg/mL, 2.50μg/mL, 5.00μg/mL of, -20 °C storage 1 month

effective, but appear abnormal peaks You should consider re-formulated.

4 Instruments and equipment

4.1

GC-MS. with a less polar (e.g., DB-5) and suitable properties or specifications of the capillary column, length. 30m, inner diameter. 0.25mm, film Thickness. 0.25 μ m.

4.2

Soxhlet extractor.

4.3

Nitrogen blowing instrument.

4.4

electric mixer.

4.5

Ovens.

4.6

temperature water bath.

4.7

rotary evaporator.

4.8

Electronic balance. a sense of the amount of 0.1mg.

4.9

Glass chromatography columns. column length.200mm ~ 300mm, inner diameter. 20mm.

b) inlet temperature. 250 °C;

c) Interface temperature. 280 °C;

d) Ion source. EI source, 70eV;

e) Measurement mode. SIM (the SIM); ion monitoring (m/z). 55,98,112, quantitative ion (m/z). 98;

f) Carrier gas. Helium, flow rate 1.0mL/min;

g) Injection volume. 1.0 μ L;

h) Injection mode. splitless.

6 Expression analysis results

Standard working solution and the volume of the sample solution and the like is inserted into the sample measured parameters, external standard. In the chromatographic conditions, 2-dodecyl cyclobutanone Selected ion chromatogram of the standard sample shown in Figure A.1.2- dodecyl cyclobutanone retention time of approximately 17.1 min, mass spectrum is shown in Figure A.2.

2-dodecyl cyclobutanone peaks detected if the sample liquid retention time retain the 2-dodecyl cyclobutanone standard peaks Same time (relative deviation $\leq 2s$), which spectrum is consistent with the 2-dodecyl cyclobutanone standard spectrum, after deducting the background monitor select Measuring ions m/z 55,98 and 112 both occur, and the relative abundance of m/z 55,112 and the base peak m/z 98 and the ratio of standard 2-dodecyl cyclobutanone Registration deviation relative abundance ratio not exceeding 20%, can be detected in the sample confirmed 2-dodecyl cyclobutanone, indicated the sample had received irradiated deal with.

Appendix A

2-dodecyl cyclobutanone standard chromatogram and mass spectrum A.1 2-azetidinone-dodecyl selected ion chromatogram of the standard 2-dodecyl azetidinone standards selected ion chromatogram shown in Figure A.1.

FIG A.1 2- azetidinone-dodecyl selected ion chromatogram of the standard

A.2 2- azetidinone-dodecyl standard full scan mass spectrum 2-dodecyl azetidinone standard full-scan mass spectra shown in Figure A.2.

FIG A.2 2- azetidinone-dodecyl standard full scan mass spectrum