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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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GB 31658.18-2022

National Food Safety Standard - Determination of Triazine Residues in Animal Foods - High Performance Liquid Chromatography

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Issued by: National Health Commission of the People's Republic of China, State Administration for Market Regulation

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Table of Contents

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https://chinastandards.org/standards/gb-31658-18-2022

10.3 Precision

foreword

This document is in accordance with the provisions of GB/T 1.1-2020 "Guidelines for Standardization Work Part 1.Structure and Drafting Rules for Standardization Documents"

drafting.

This document is published for the first time.

National Food Safety Standards

Determination of triazine residues in animal food by high performance liquid chromatography

1 Scope

This document specifies the sample preparation and high performance liquid chromatography detection methods for the detection of triazine residues in animal foods.

This document is applicable to the detection of triazine residues in muscle, liver, kidney tissue, milk and goat milk of cattle and sheep.

2 Normative references

The content in the following documents constitutes the essential provisions of this document through normative references in the text. Among them, the dated reference documents,

Only the version corresponding to the date applies to this document; for undated references, the latest version (including all amendments) applies to this document

document.

GB/T 6682 Analytical laboratory water specifications and test methods

3 Terms and Definitions

This document does not have terms and definitions that need to be defined.

4 principles

The residual triazine in the sample was extracted with acetonitrile water (the residual triazine in milk was extracted with acetic acid acetonitrile solution), weak cation exchange solid phase extraction

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https://chinastandards.org/standards/gb-31658-18-2022

The column was purified, determined by high performance liquid chromatography-ultraviolet method, and quantified by external standard method.

5 Reagents and materials

The reagents used in the following are all analytical reagents unless otherwise specified; the water is the first-class water in accordance with the provisions of GB/T 6682.

5.2 Solution preparation

5.2.1 60% acetonitrile solution. Take 60mL of acetonitrile, add 40mL of water, and mix well.

5.2.2 0.5% acetic acid in acetonitrile solution. Take 2.5 mL of glacial acetic acid, dilute to 500 mL with acetonitrile, and mix well.

5.2.3 5% ammonia solution. take 5mL of ammonia water, add 95mL of water, and mix well.

5.2.4 Acetic acid methanol solution (pH 7.0). Take 6 mL of glacial acetic acid, add 94 mL of methanol, mix well, adjust the pH to 7.0 +/- 0.05 with ammonia water.

5.2.5 0.02mol/L ammonium formate solution (pH4.0). take 1.26g of ammonium formate, add 900mL of water to dissolve, adjust the pH to 4.0+/-0.0 with formic acid

0.05, dilute with water to 1000mL.

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5.3 Standards

Diacetamide triazine amidine (Diminazene acetic acid, C14H15N7.2C4H7NO3, CAS number. 908-54-3), content >= 91%.

5.4 Preparation of standard solution

5.4.1 Standard stock solution. Take an appropriate amount of diacetamide triazine standard (equivalent to about 10 mg of triazine), weigh it accurately, add water to dissolve

and dilute to a 10mL brown volumetric flask, and prepare a standard stock solution of triazine with a concentration of 1mg/mL. Store in the dark at 4°C, and the validity period is

1 month.

5.4.2 Standard working solution. Accurately measure 1mL of 1mg/mL standard stock solution, put it in a 100mL brown volumetric flask, dilute with water to the mark,

Prepare the triazine standard working solution with a concentration of 10 μg/mL, store it in the dark at 4°C, and have a validity period of 7 d.

6 Instruments and equipment

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https://chinastandards.org/standards/gb-31658-18-2022

6.1 High performance liquid chromatography. equipped with ultraviolet detector or diode array detector.

7.1.1 Organization

Take an appropriate amount of fresh or thawed blank or test tissue, mince it, and homogenize it.

a) Take the homogeneous test sample as the test sample;

b) Take a homogeneous blank sample as a blank sample;

c) Take a homogeneous blank sample, add a standard solution of appropriate concentration, and add a sample as a blank.

7.1.2 Milk

Take an appropriate amount of fresh or thawed blank or test milk sample, and mix well.

a) Take the uniformly mixed test sample as the test sample;

b) Take a uniformly mixed blank sample as a blank sample;

c) Take a uniformly mixed blank sample, add a standard solution of appropriate concentration, and add a sample as a blank.

7.2 Storage of samples

Store below -18°C.

8.1.1 Organization

Take 2g of the sample (accurate to +/-0.05g), put it in a 50mL polypropylene centrifuge tube, add 18mL of 60% acetonitrile solution, vortex for 1min, and store it on ice.

Adjust the pH to 5.5+/-0.1 with acetic acid, shake for 10 min, centrifuge at 10,000 r/min for 5 min, take the supernatant, and dilute it with 60% acetonitrile solution to

20.0mL, mix well, take 2.0mL (muscle samples take 10.0mL), set aside.

8.1.2 milk

Take 5g of the sample (accurate to +/-0.05g), put it in a 50mL polypropylene centrifuge tube, add 10mL of 0.5% acetic acid acetonitrile solution accurately, vortex

1 min, shake for 10 min, centrifuge at 10000r/min for 10 min, remove all the supernatant, and set aside.

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