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Determination of zearalenone in food - High performance liquid chromatographic method with immunoaffinity column clean-up

食品中玉米赤霉烯酮的测定

免疫亲和层析净化高效液相色谱法

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Table of contents

Foreword		3
1	Scope	4
2	Normative references	4
3	Method summary	4
4	Reagents and materials	5
5	Instruments and equipment	5
6	Analysis procedures	6
7	Result calculation	8
8	Recovery	8
9	Repeatability	8
Δn	nendix A (Informative) Liquid chromatogram of zearalenone standard	a

Foreword

Appendix A of this standard is an informative appendix.

This standard was proposed by the National Food Industry Standardization Technical Committee (SAC/TC 64).

This standard shall be under the jurisdiction of the National Food Industry Standardization Technical Committee Food General Detection Technology Technical Committee Branch (SAC/TC 64 /SC 8)

The drafting organizations of this standard: Qingdao Product Quality Supervision and Inspection Institute, the People's Republic of China Shandong Entry-Exit Inspection and Quarantine Bureau, Clover Technology Group Inc., Tsingtao Brewery Co., Ltd.

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Determination of zearalenone in food - High performance liquid chromatographic method with immunoaffinity column clean-up

1 Scope

This standard specifies the method for the determination of zearalenone in foods by high performance liquid chromatographic method with immunoaffinity column clean-up.

This standard is applicable to the determination of zearalenone in food and grain products, alcohol, soy sauce, vinegar, sauce and sauces.

The limit of detection of this method is 20 μ g/kg for food and grain products; 20 μ g/kg for alcohol; AND 50 μ g/kg for soy sauce, vinegar, sauce and sauces.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard; however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-2008, ISO 3696:1987, MOD)

3 Method summary

USE the extraction solution to extract the zearalenone from the test specimen; after making it be purified by immunoaffinity column, USE the high performance chromatography fluorescence detector to determine it and the external standard method to quantify it.

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5.5 Glass syringe: 10 mL.

5.6 Test screen: 1 mm aperture.

5.7 Air pressure pump.

6 Analysis procedures

6.1 Sample preparation and extraction

- **6.1.1** Food and grain products: GRIND samples; USE high-speed universal mill to grind the hard grain and MAKE it pass the test sieve (5.6); DO not grind it into powder. WEIGH 50 g of ground test specimen (accurate to 0.01 g) and PLACE it into a 100 mL volumetric flask; ADD 5 g of sodium chloride; USE the extraction solution (4.3) to make its volume reach to the mark; MIX it uniformly; TRANSFER it into a homogeneous cup; MAKE it subjected to high-speed stirring and extraction for 2 min. USE the quantitative filter paper to filter it; TRANSFER 10.0 mL of filtrate into a 50 mL volumetric flask; ADD water to make its volume reach to the mark; MIX it uniformly; USE glass fiber filter paper to the filtrate is clean; COLLECT the filtrate A into a clean container.
- **6.1.2** Alcohols: TAKE 20 g of alcohol samples (the alcohol containing carbon dioxide is refrigerated at 4 °C for 30 min, filtered or degassed ultrasonically prior to use) or other alcohol samples containing no carbon dioxide (accurate to 0.01 g) into a 50 mL volumetric flask; USE acetonitrile to make its volume reach to the mark; SHAKE it uniformly; TRANSFER 10.0 mL of filtrate into a 50 mL volumetric flask; ADD water to make the volume reach to the mark; MIX it uniformly; USE glass fiber filter to filter it until the filtrate is clean; COLLECT the filtrate B into a clean container.
- **6.1.3** Soy sauce, vinegar, sauce and sauce products: WEIGH 25 g (accurate to 0.01 g) of uniformly mixed test specimen; USE acetonitrile to make the volume reach to 100.0 mL; MAKE it subjected to ultrasonic extraction for 2 min; USE the quantitative filter paper to filter it; TRANSFER 10.0 mL of filtrate into a 50 mL volumetric flask; ADD water to make the volume reach to the mark; MIX it uniformly; USE glass fiber filter to filter it until the filtrate is clean; COLLECT the filtrate C into a clean container.

6.2 Purification

CONNECT the immunoaffinity column to under the glass syringe (5.5); accurately PIPETTE 10.0 mL of the filtrate A or B or C as mentioned in 6.1 into the glass syringe. CONNECT the air pressure pump to the glass syringe; ADJUST the pressure so that the solution can pass through the immunoaffinity

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