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SN

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

SN/T 3137-2012

Determination of deoxynivalenol, 3-acetyl-deoxynivalenol, 15-O-acetyl-4-deoxynivalenol and their metabolite in food for export. LC-MS/MS method

出口食品中脱氧雪腐镰刀菌烯醇、3-乙酰脱氧雪腐镰刀菌烯醇、15-乙酰脱氧雪腐镰刀菌烯醇及其代谢物的测定 液相色谱-质谱/质谱法

(English Translation)

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Foreword

This standard was drafted in accordance with GB/T 1.1-2009 given rules.

This standard is proposed and managed by the National Certification and Accreditation Administration Committee.

This standard was drafted. People's Republic of China Shaanxi Exit Inspection and Quarantine, Shenzhen People's Republic of China Entry-Exit Inspection and Quarantine Bureau.

1 Scope

This standard provides the food for export deoxynivalenol fungi alcohol, 3-acetyl deoxynivalenol fungi alcohol, 15-acetyl deoxynivalenol sickle Enol knife bacteria and their metabolites to epoxy deoxynivalenol bacteria alcohol residues LC - MS/MS detection method.

This standard applies to wheat, rice, corn, beer, biscuits deoxynivalenol fungi alcohol, 3-acetyl deoxynivalenol fungi alcohol, 15- acetyl deoxynivalenol bacteria alcohol and chicken, liver, kidney, milk, eggs metabolites to epoxy deoxynivalenol fungi alcohol residues Determination of the amount of the stay and confirmed.

2 Method summary

Sample of deoxynivalenol fungi alcohol, 3-acetyl deoxynivalenol fungi alcohol, 15-acetyl deoxynivalenol bacteria enolase and their representatives Epoxide metabolite go deoxynivalenol fungi alcohol extracted with acetonitrile, defatted with hexane and after solid phase extraction (SPE), liquid chromatography - mass Spectrum/MS detection, external standard.

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3 Reagents and materials

Unless otherwise indicated, the reagents used were of analytical grade water for ultra-pure water.

3.1
acetonitrile. HPLC grade.

3.2
Methanol. HPLC grade.

3.3
hexane. pesticide grade.

3.4
sodium chloride.

3.5
anhydrous sodium sulfate. After burning 650 °C 4h, placed in a desiccator.

3.6
acetonitrile saturated n-hexane solution. n-hexane in the amount of 200mL 250mL separating funnel, adding a small amount of acetonitrile, shaken vigorously for a few minutes Bell, still hierarchical, discarding the lower acetonitrile layer, that is, too.

3.7
methanol - water. Measure 20mL of methanol was added 80mL of water, mix well.

3.8
standards. deoxynivalenol bacteria enolase (deoxynivalenol, DON), a purity of greater than or equal to 96.0%; 3-acetyl deoxynivalenol Fusarium enol (3-acetyldeoxynivalenol, 3-Ac-DON), a purity of greater than or equal to 94.0%, 15-acetyl deoxynivalenol bacteria ene Alcohol (15-O-acetyl-4-deoxynivalenol, 15-Ac-DON), a purity of greater than or equal to 94.0%. Standard information Table A.1.

3.9
to epoxy deoxynivalenol bacteria enolase (deepoxy-deoxynivalenol, DOM-1) in acetonitrile. a concentration of 50μg/mL.

3.10
standard stock solution. Weigh accurately were a certain amount of DON, 3-Ac-DON and 15-Ac-DON standard (3.8), dissolve with acetonitrile Solution, dubbed 0.5mg/mL standard stock solution stored in the dark at less -18 °C.

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3.11

intermediate standard solution. accurate Pipette each standard stock solution (3.9,3.10) 1mL to 10mL brown volumetric flask, constant volume with acetonitrile to the mark, to the following -18 °C stored.

3.12

Standard working solution. Depending on needs, accurately draw a certain amount of intermediate standard solution (3.11), were formulated with a suitable concentration of acetonitrile

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