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中华人民共和国出入境检验检疫行业标准

SN/T 1771—2006

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进出口粮谷中 T-2 毒素的测定 免疫亲和柱-液相色谱法

Determination of T-2 toxin in cereals for import and export—
Immunoaffinity column and liquid chromatography method

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行业标准
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Annex A
(informative)

Liquid chromatogram of the standard T-2 toxin derivative

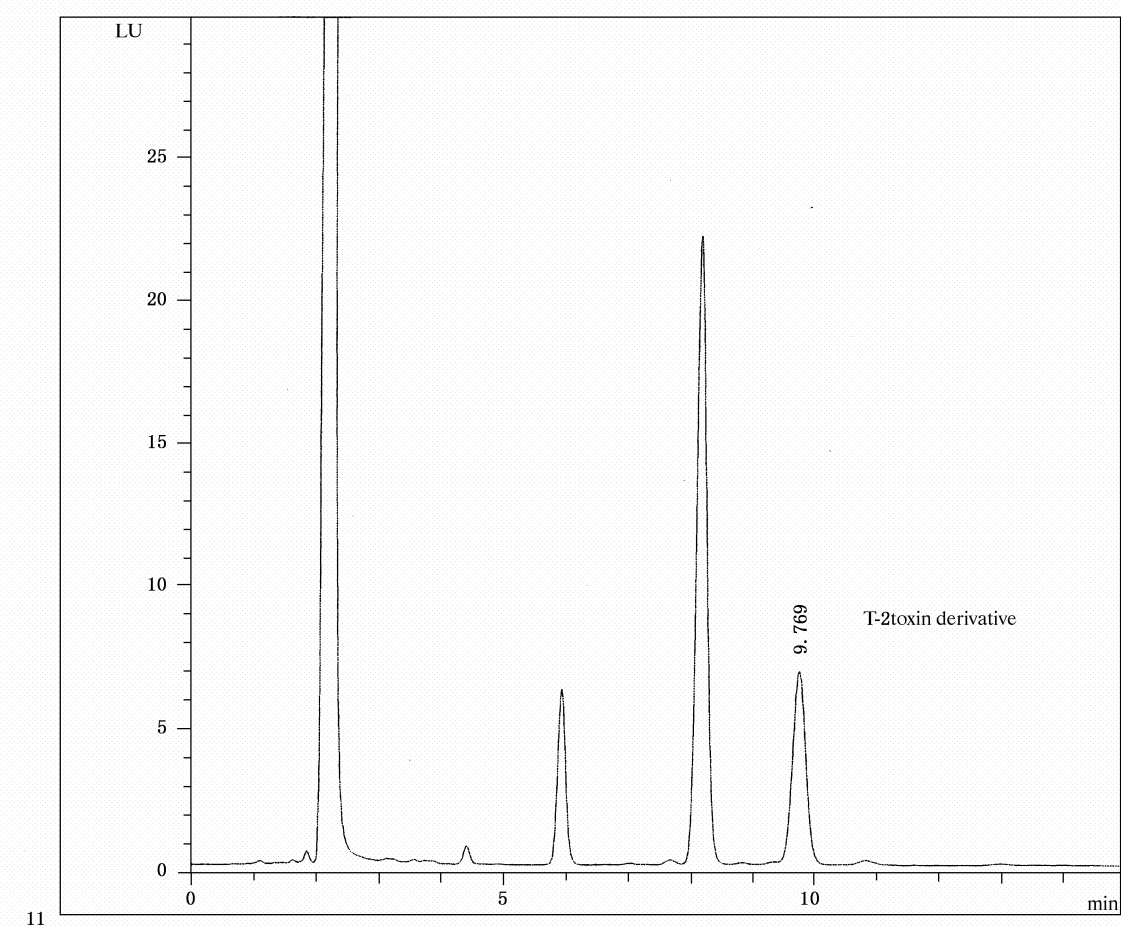


Figure A.1 Liquid chromatogram of T-2 toxin derivative

前 言

本标准的附录 A 为资料性附录。

本标准由国家认证认可监督管理委员会提出并归口。

本标准由中华人民共和国辽宁出入境检验检疫局、中华人民共和国上海出入境检验检疫局负责起草。

本标准主要起草人：卫锋、李军、隋凯、方晓明。

本标准系首次发布的出入境检验检疫行业标准。

3.4.2 Clean up

10 mL of diluted extract (equivalent to 1.0 g sample) were passed through the T-2 immunoaffinity column at a low-rate of about 1 drop/second, followed by 10.0 mL distilled water at one to two drops per second. T-2 toxin was then eluted with 1.0 mL methanol and collected in a clean glass tube. The eluted extract was evaporated under a stream of N₂ at 50°C in heating block and the dried residue derivatized with 1-AN as reported below.

3.4.3 Derivatization

Fifty microlitres of DMAP solution followed by 50 μL of 1-AN reagent were added to the residue. The vial was closed and mixed by vortex for 1 min. The mixture was left to react for 15 min at 50°C in a heater block and then cooled in ice for 10 min. The whole volume of the reaction mixture was dried under a stream of N₂ at ca. 50°C and reconstituted with 1 000 μL mobile phase. Twenty microlitres of reconstituted reaction mixture were injected into the chromatographic apparatus by a full loop injection system.

Note: Annotation: Extract was diluted appropriately for upper content of T-2 toxin in sample, ensuring the content of T-2 toxin was under the maximum adsorption of T-2 toxin immunoaffinity column.

3.4.4 Determination

3.4.4.1 HPLC operating condition

- Chromatographic column: ZORBAX Eclipse XDB-C₁₈ 4.6 × 150 mm, 5 μm;
- Mobile phase: acetonitrile-water (80 + 20, V/V);
- Flow rate: 1 mL/min;
- Detection wavelength: excitation wavelength: 381 nm; emission wavelength: 470 nm;
- Injection volume: 20 μL.

3.4.4.2 HPLC determination

According to the approximate concentrate of T-2 toxin derivative in the sample solution, select the standard working solution with similar peak area to that of sample solution. The responses of T-2 toxin derivative in the standard working solution and sample solution should be within the linear range of the instrumental detection. The standard working solution should be randomly injected in-between the injections of the sample solution of equal volume. Under the above chromatographic condition, the retention time of T-2 toxin derivative is ca 9.8 min. For HPLC chromatogram of standard, see figure A. 1 in annex A.

3.4.5 Blank test

The operation of the blank test is the same as that described in the method of determination but with omission of the sample addition.

进出口粮谷中 T-2 毒素的测定 免疫亲和柱-液相色谱法

1 范围

本标准规定了进出口粮谷中 T-2 毒素检验的制样和免疫亲和柱-液相色谱测定方法。本标准适用于进出口玉米、小麦中 T-2 毒素的检验。

2 制样

2.1 试样制备

将样品按四分法缩分至 1 kg, 全部磨碎并通过 20 目筛, 混匀, 均分成两份作为试样, 分别装入洁净的容器内, 密封, 标明标记。在抽样和制样的操作过程中, 应防止样品受到污染或发生残留物含量的变化。

2.2 试样保存

将试样于 -5°C 以下避光保存。

3 测定方法

3.1 方法提要

试样中的 T-2 毒素用甲醇-水提取后, 提取液经免疫亲和柱净化, 浓缩、衍生、定容后, 用配有荧光检测器的液相色谱仪进行测定, 外标法定量。

3.2 试剂与材料

除另有规定外, 所用试剂均为分析纯, 水为重蒸馏水。

3.2.1 甲醇。

3.2.2 乙腈: HPLC 级。

3.2.3 甲苯: HPLC 级。

3.2.4 甲醇+水(8+2): 取 80 mL 甲醇加 20 mL 水。

3.2.5 0.325 mg/mL 4-二甲基氨基吡啶(DMAP)溶液: 准确称取 0.032 5 g 于 100 mL 容量瓶, 甲苯稀释至刻度。

3.2.6 0.3 mg/mL 1-氰酸苈(1-Anthroylnitrile, 1-AN)溶液: 准确称取 0.030 0 g 于 100 mL 容量瓶, 甲苯稀释至刻度。

3.2.7 T-2 毒素标准品: 纯度 ≥ 98%。

3.2.8 T-2 毒素标准溶液: 准确称取适量的 T-2 毒素标准品, 用乙腈配成浓度为 0.5 mg/mL 的标准储备液。按所需要的浓度进行稀释, 作为标准工作溶液使用。

3.3 仪器与设备

3.3.1 液相色谱仪配有荧光检测器。

3.3.2 粉碎机。

3.3.3 高速均质器。

3.3.4 氮吹仪。

3.3.5 空气压力泵。

3.3.6 玻璃纤维滤纸。