

特殊医学用途全营养配方食品中

黄曲霉毒素 B₁ 检验方法研究

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摘要: 目的: 依据 GB 5009.22-2016《食品安全国家标准食品中黄曲霉毒素 B 族和 G 族的测定》第三法 高效液相色谱-柱后衍生法对特殊医学用途全营养配方食品中黄曲霉毒素 B₁ 含量进行检测, 对黄曲霉毒素 B₁ 响应值偏低的风险问题进行分析研究。

本次实验应用国标参考条件, 对标准曲线低浓度点、检出限浓度点、定量限浓度点上机检测后, 发现主峰响应值偏低, 很难进行有效积分, 直接影响检验结果准确度。通过控制变量、调整检测参考条件等方法, 对检验过程进行分析研究, 对检验方法进行有效验证。最终得到提高黄曲霉毒素 B₁ 灵敏度及响应值的重要条件: 调节流动相比例已达到色谱主峰有效积分效果; 增加样品上机检测的进样量; 增加过免疫亲和柱用的样液体积, 以减少样品稀释倍数。三个条件同时满足时, 所测得的结果准确且灵敏度高, 精密度重复性良好。

关键词: GB 5009.22; 高效液相色谱; 黄曲霉毒素 B₁; 检出限; 定量限; 灵敏度; 信噪比

Abstract: Objective: To detect the content of aflatoxin B₁ in full nutrition formula food for special medical purposes according to the third method of high performance liquid chromatography post column derivation in GB 5009.22-2016 National Food Safety Standard Determination of Aflatoxin Groups B and G, and analyze and study the risk of low response value of aflatoxin B₁.

In this experiment, the national standard reference conditions were applied to detect the low concentration point, detection limit concentration point and quantitative limit concentration point of the standard curve on the computer. It was found that the main peak response value was low, so it was difficult to conduct effective integration, which directly affected the accuracy of the test results. The inspection process is analyzed and studied by means of controlling variables, optimizing the reference conditions for inspection, and the inspection method is effectively verified. Finally, the important conditions to improve the sensitivity and response value of aflatoxin B₁ on the instrument were obtained: adjust the proportion of mobile phase to achieve the effective integration effect of the main chromatographic peak; Increase the sample injection

volume of the sample for on-line detection; Increase the volume of the sample solution used for the immunoaffinity column to reduce the dilution ratio of the sample. When the three conditions are met at the same time, the measured results are accurate, with high sensitivity and good precision repeatability.

Key words:GB 5009.22; High performance liquid chromatography; Aflatoxin B₁; Detection limit; Limit of quantitation; Sensitivity; Signal-to-noise ratio.

1 绪论

1.1 研究对象

黄曲霉毒素 B₁ 的毒性高于氰化钾毒性，而且具有极强的致癌性、致突变性及致畸性。黄曲霉毒素 B₁ 耐热，280℃才可裂解，故一般烹调加工温度下难以破坏。黄曲霉毒素的污染可导致严重的婴幼儿急性、慢性中毒和癌症，导致生长障碍、肝脏损伤和肝癌等脏器损伤，甚至死亡^[1]。特殊医学用途全营养配方食品是为了满足进食受限、消化吸收障碍、代谢紊乱或特定疾病状态人群对营养素或膳食的特殊需要，专门加工配制而成的配方食品^[2]。其配方及基质复杂，严格控制原料的质量安全，同时按照 GB 29922-2013 食品安全国家标准 特殊医学用途配方食品通则的要求对特殊医学用途全营养配方食品黄曲霉毒素 B₁ 进行重点检测，保障终端产品安全。本文旨在对黄曲霉毒素 B₁ 检验方法进行研究，对标准曲线低浓度、检出限浓度、定量限浓度在仪器上灵敏度及响应值低不易检出、难积分的风险问题进行分析验证，并得出有效结论。

1.2 立题依据

高效液相色谱-柱后衍生法检测食品中的黄曲霉毒素 B₁，适用于谷物及其制品、豆类及其制品、坚果及籽类、油脂及其制品、调味品、婴幼儿配方食品和婴幼儿辅助食品^[3]。研究表明，高效液相色谱法是当前在黄曲霉毒素定量检测方面有着相当应用广度的方法。此种方法将样品中的黄曲霉毒素 B₁ 经柱层析分离后，通过测量色谱峰的面积实现定量检测。高效液相色谱法在实际应用过程中表现出灵敏、准确并且重复性好等多方面优点，并且适合食品中四种黄曲霉毒素含量的检测^[4]。依据 GB 5009.22-2016《食品安全国家标准食品中黄曲霉毒素 B 族和 G 族的测定》为特殊医学用途全营养配方食品中黄曲霉毒素 B₁ 检测方法应用提供有效依据。对检测方法的线性关系、回收率、检出限、定量限、精密度进行验证，结果均符合 GB/T27404-2008 实验室质量控制规范 食品理化检测^[5]的要求。