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技术交流

液相色谱-串联质谱法测定饲料中黄曲霉毒素的研究

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摘要 建立了液相色谱 串联质谱法测定饲料中6种黄曲霉毒素（包括黄曲霉毒素B_1、B_2、G_1、G_2、M_1、M_2）的方法。试样中的黄曲霉毒素经84%乙腈溶液超声提取后，用正己烷脱脂，过霉菌毒素多功能固相萃取柱净化，氮气吹至干，用流动相溶解后进行液相色谱 串联质谱法测定。方法的最低检出限为0.2 μg•kg^{-1}，定量限均为0.5 μg•kg^{-1}；标准工作液在0.5～100 μg•L^{-1}的范围内线性良好；加标浓度在1.0～100 μg•kg -1 范围内，饲料中黄曲霉毒素的回收率在66.1%～108%之间，批内相对标准偏差≤17.8%，能满足相关法规要求。

关键词 液相色谱-串联质谱法（LC-MS/MS） 黄曲霉毒素 饲料

分类号

Determination of Aflatoxins in Feeds by Liquid Chromatography-Tandem Mass Spectrometry

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Abstract

Totally 6 Aflatoxins in feeds were determined by high performance liquid chromatography combined with electrospray ionization triple quadrupole tandem mass spectrometry(HPLC-MS/MS) under the multiple reaction monitoring (MRM) mode, and especially focused on the optimization of extraction, clean-up, HPLC separation and MS/MS parameters of analytes. The Aflatoxins B_1、B_2、G_1、G_2、M_1 and M_2 were extracted by 84% of acetonitrile aqueous solution, purified by passing through multifunctional cartridges. The elution was evaporated by nitrogen blow and dissolved by mobile phase, and then assayed by HPLC-MS/MS. The limits of detection(LOD) are 0.2 μg•kg^{-1}, and the limits of quantitation(LOQ) are 0.5 μg•kg^{-1}. The calibration curves are linear between 0.5 μg•L^{-1} and 100 μg•L^{-1} for Aflatoxins B_1、B_2、G_1、G_2、M_1 and M_2. Recoveries of those mycotoxins in feeds are 66.1% —108%, and inter-relative standard deviation（n=5） is less than 17.8% at spiked level of 1.0—100 μg•kg^{-1}. The method is simple, sensitive and reliable for feed safety.

Key words high performance liquid chromatography-tandem mass spectrometry(HPLC-MS/MS) _ Aflatoxins _ feeds

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