色谱 » 2010, Vol. 28 » Issue (12) :1189-1191 DOI: 10.3724/SP.J.1123.2010.01189

亲水作用色谱-串联质谱法测定化妆品中三聚氰胺残留量

梅少博, 侯晋*, 张文国, 倪鹏, 殷烈, 丁黎

南通出入境检验检疫局, 江苏 南通 226005

Determination of melamine in cosmetics by hydrophilic interaction chromatography-tandem mass spectrometry

MEI Shaobo, HOU Jin*, ZHANG Wenguo, NI Peng, YIN Lie, DING Li

Nantong Entry-Exit Inspection and Quarantine Bureau, Nantong 226005, China

摘要 建立了化妆品中三聚氰胺的亲水作用色谱-质谱联用（HILIC-MS/MS）检测方法。样品经三氯乙酸溶液提取（脂溶性样品再经正己烷萃取)后,用混合型阳离子交换(MCX)反相固相萃取柱富集净化,用5 mmol/L乙酸铵水溶液(含0.1%甲酸)和乙腈作为流动相,以梯度洗脱方式在ZIC-HILIC色谱柱上实现分离,以电喷雾离子源正离子(ESI+)模式进行质谱分析。三聚氰胺在0.02～0.5 mg/L范围内呈良好线性关系,相关系数为0.9985;方法的检出限(LOD,信噪比(S/N)≥3)为5.0 μg/kg,定量限(LOQ,S/N＞10)为20.0 μg/kg;在0.01～0.1 mg/kg添加浓度范围内,三聚氰胺的平均回收率为84.7%～93.4%,相对标准偏差为4.5%～8.4%。该方法能满足化妆品中三聚氰胺残留量的检测。

关键词： 亲水作用色谱-串联质谱联用法 三聚氰胺 残留 化妆品

Abstract： A method for the determination of melamine in cosmetics was developed by hydrophilic interaction chromatography-tandem mass spectrometry (HILIC-MS/MS). The sample was extracted in turn by aqueous solution of trichloroacetic acid and hexane, and then cleaned-up by the mixed-mode cation exchange (MCX) solid-phase extraction (SPE) cartridge. A ZIC-HILIC column was used for the separation by gradient elution, and an electrospray ion trap mass spectrometer was used in the positive ion mode. The linear range of melamine was from 0.02 mg/L to 0.5 mg/L with a correlation coefficient of0.9985. The limit of detection (LOD, S/N≥3) and limit of quantification (LOQ, S/N＞10) were 5.0 μg/kg and 20.0 μg/kg, respectively. The average recoveries and the relative standard deviations ranged from 84.7% to 93.4% and from 4.5% to 8.4%, respectively, in spiked samples at the concentrations from 0.01 mg/kg to 0.1 mg/kg. The method is suitable for the determination of melamine in cosmetics.

Keywords： hydrophilic interaction chromatography-tandem mass spectrometry (HILIC-MS/MS) melamine residue cosmetics

Received 2010-08-25; published 2010-12-27

Fund:

江苏检验检疫系统重点科研资助计划项目(No. 2007KJ54).

Corresponding Authors: 侯晋,高级工程师,博士,研究方向为微量物质分析. Tel: (0513)83517973, E-mail: houjinnt@yahoo.com.cn. Email: houjinnt@yahoo.com.cn.

引用本文:

梅少博, 侯晋*, 张文国, 倪鹏, 殷烈, 丁黎.亲水作用色谱-串联质谱法测定化妆品中三聚氰胺残留量[J] 色谱, 2010,V28(12): 1189-1191

MEI Shaobo, HOU Jin*, ZHANG Wenguo, NI Peng, YIN Lie, DING Li.Determination of melamine in cosmetics by hydrophilic interaction chromatography-tandem mass spectrometry[J] Chinese Journal of Chromatography, 2010,V28(12): 1189-1191

链接本文:

http://www.chrom-china.com/CN/10.3724/SP.J.1123.2010.01189 或 http://www.chrom-china.com/CN/Y2010/V28/I12/1189