

同位素稀释-超高效液相色谱-串联质谱法同时测定精油中的7种雌性激素

黄百芬¹, 韩铮², 徐小民¹, 蔡增轩¹, 姜维¹, 任一平^{1*}

1. 浙江省疾病预防控制中心, 浙江 杭州 310051; 2. 浙江大学, 浙江 杭州 310058

Simultaneous determination of 7 female sex hormones in essential oil by high performance liquid chromatography-tandem mass spectrometry with isotope dilution

HUANG Baifen¹, HAN Zheng², XU Xiaomin¹, CAI Zengxuan¹, JIANG Wei¹, REN Yiping^{1*}

1. Zhejiang Provincial Center for Disease Prevention and Control, Hangzhou 310051, China; 2. Zhejiang University, Hangzhou 310058, China

摘要	参考文献	相关文章
----	------	------

Download: PDF (197KB) HTML 1KB Export: BibTeX or EndNote (RIS) Supporting Info

摘要 建立了采用同位素稀释-超高效液相色谱-串联质谱同时快速测定精油中7种雌性激素(雌三醇、雌二醇、雌酮、炔雌醇、己烯雌酚、己烷雌酚、己烯雌酚)的方法。样品中雌性激素用乙酸乙酯-正己烷(2:98, v/v)溶液提取后,经硅胶固相萃取小柱净化,通过ACQUITY UPLCTM BEH SHELDED RP18色谱柱(100 mm×2.1 mm, 1.7 μm)、以水-乙腈作流动相梯度洗脱对7种雌性激素进行分离,采用串联质谱在负离子扫描方式下通过多反应监测(MRM)模式进行定性定量分析。以雌三醇-D3、雌二醇-D3、己烯雌酚-D6为内标,有效减少了样品基质的影响。该方法对精油中7种雌性激素的检出限(LOD)为0.3~7 μg/kg,定量限(LOQ)为1~20 μg/kg。待测物与内标物定量离子的峰面积比值与待测物的质量浓度在20~500 μg/L范围内呈良好的线性关系,相关系数(r²)均大于0.997;在20~500 μg/kg范围内3个水平的加标平均回收率为88.5%~114.8%,日内精密性(以相对标准偏差计)(n=6)为4.8%~18.9%。应用该方法对浙江杭州地区不同超市或美容院随机采集的12份精油样品进行测定的结果显示,有1份样品含有雌二醇和雌酮,其余11份样品均未检出雌性激素。

关键词: 同位素稀释 超高效液相色谱-串联质谱 雌性激素 精油

Abstract: A reliable ultra-high performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) method for the simultaneous determination of 7 female sex hormones (estriol, estradiol, estrone, ethinyloestradiol, dienestrol, hexestrol, diethylstilbestrol) in essential oil was developed. The sample was extracted by ethylacetate-normal hexane solution (2:98, v/v) and the extract was purified by a silica solid phase extraction-based clean-up column. Then, the analytes were separated on an ACQUITY UPLC BEH SHELDED RP18 column (100 mm×2.1 mm, 1.7 μm) in gradient elution with the mobile phases of water and acetonitrile. The separated compounds were detected with a Waters Xevo TQ MS tandem quadrupole mass spectrometer operated in negative electro-spray ionization using multiple reaction monitoring mode. Estriol-D3, estradiol-D3 and diethylstilbestrol-D6 were used as the internal standards to reduce the matrix effects. The limits of detection and quantitation for the 7 female sex hormones in essential oil were 0.3~7 μg/kg and 1~20 μg/kg, respectively. Good linear relationships and high correlation coefficients (r²≥0.997) were obtained in the mass concentration range of 20~500 μg/L. The average recoveries were 88.5%~114.8% and the intra-assay relative standard deviations were 4.8%~18.9% at the spiked levels of 20~500 μg/kg. Finally, a total of 12 samples randomly collected from different supermarkets in Zhejiang Province were screened for the 7 female sex hormones by the proposed method. The results showed that only one sample contained estradiol and estrone.

Keywords: isotope dilution ultra-high performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) female sex hormones essential oil

Received 2010-09-15; published 2011-01-21

Fund:

浙江省医药卫生科技计划项目(No. 2008A026).

Corresponding Authors: 任一平,教授级高级工程师,研究方向为色谱-质谱分析技术. Tel: (0571)87115261, E-mail:

renyiping@263.net. Email: renyiping@263.net

引用本文:

黄百芬¹, 韩铮², 徐小民¹, 蔡增轩¹, 姜维¹, 任一平^{1*}.同位素稀释-超高效液相色谱-串联质谱法同时测定精油中的7种雌性激素[J] 色谱, 2011,V29(01): 20-25

HUANG Baifen¹, HAN Zheng², XU Xiaomin¹, CAI Zengxuan¹, JIANG Wei¹, REN Yiping^{1*}.Simultaneous determination of 7 female sex hormones in essential oil by high performance liquid chromatography-tandem mass spectrometry with isotope dilution[J] Chinese Journal of Chromatography, 2011,V29(01): 20-25

链接本文:

http://www.chrom-china.com/CN/10.3724/SP.J.1123.2011.00020 或 http://www.chrom-china.com/CN/Y2011/V29/I01/20

Service

- ▶ 把本文推荐给朋友
- ▶ 加入我的书架
- ▶ 加入引用管理器
- ▶ Email Alert
- ▶ RSS

作者相关文章

- ▶ 黄百芬
- ▶ 任一平
- ▶ 韩铮
- ▶ 徐小民
- ▶ 蔡增轩
- ▶ 姜维