

研究论文

高效液相色谱-二极管阵列/荧光检测器串联法同时测定精油中的七种性激素

王小芳*, 曾文芳, 王菁, 任韧

杭州市疾病预防控制中心, 浙江 杭州 310006

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摘要 建立了高效液相色谱法(HPLC)-二极管阵列检测器(DAD)/荧光检测器(FLD)串联技术同时测定精油中7种性激素(雌二醇、雌三醇、雌酮、睾酮、甲基睾酮、孕酮、己烯雌酚)的方法。样品先用正己烷溶解后,用90%的甲醇水溶液提取,弃去正己烷层,下层清液再用正己烷脱脂、净化2次,目标化合物以水-甲醇-乙腈(体积比为50:30:20)为流动相,经XTerra RP18色谱柱(250 mm×4.6 mm, 5 μm)分离,用DAD-FLD串联法进行检测。雌二醇、雌三醇、雌酮、己烯雌酚的DAD检测波长为197 nm,睾酮、甲基睾酮、孕酮的DAD检测波长为240 nm。雌二醇、雌三醇、雌酮同时用FLD定性定量,激发波长为280 nm,发射波长为310 nm。7种性激素分离效果良好并消除了样品中杂质峰的干扰。7种性激素除孕酮的回收率为79.5%以外,其余组分的平均回收率均在93%以上;相对标准偏差为0.90%~1.89%;检出限为0.010~1.0 mg/L。该方法简便、准确,可用于同时测定精油中的7种性激素。

关键词 [高效液相色谱法](#) [二极管阵列检测](#) [荧光检测](#) [性激素](#) [精油](#)

Simultaneous determination of seven sexual hormones in essential oil by high performance liquid chromatography with diode array and fluorescence detectors

WANG Xiaofang*, ZENG Wenfang, WANG Jing, REN Ren

Hangzhou Center for Disease Control and Prevention, Hangzhou 310006, China

Abstract

An efficient method for analyzing seven sexual hormones (estradiol-17β, estriol, estrone, testosterone, methyl-testosterone, progesterone and diethylstilbestrol) in essential oil by high performance liquid chromatography was developed. The samples were dissolved in n-hexane, then extracted with 90% methanol solution. The n-hexane layer was discarded and the methanol layer was cleaned-up twice with n-hexane. The target compounds were separated on an XTerra RP18 column (250 mm×4.6 mm, 5 μm) using water-methanol-acetonitrile (50:30:20, v/v/v) as mobile phase in isocratic mode, and detected by a diode array detector (DAD) and a fluorescence detector (FLD). The flow rate was 1.0 mL/min. Estriol, estradiol-17β, estrone, diethylstilbestrol were detected at 197 nm; progesterone, testosterone, methyl-testosterone were detected at 240 nm; estriol, estradiol-17β, estrone were detected with the fluorescence detector simultaneously, the excitation and emission wavelengths were 280 nm and 310 nm, respectively. The average spiked recoveries for seven sexual hormones were above 93.0% except that of progesterone was 79.5%. The relative standard deviations of seven sexual hormones were from 0.90% to 1.89%. The linear ranges of the determination were from 0.010 mg/L to 1.0 mg/L. The method is simple and accurate for simultaneously analyzing the seven sexual hormones in essential oil.

Key words [high performance liquid chromatography \(HPLC\)](#) [diode array detection \(DAD\)](#) [fluorescence detection \(FLD\)](#) [sexual hormones](#) [essential oil](#)

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通讯作者 王小芳 wxfana@163.com

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