

研究论文

固相萃取-气相色谱/质谱法同时测定化妆品中的邻苯二甲酸酯和对羟基苯甲酸酯

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摘要 采用超声协助甲醇提取、固相萃取净化、气相色谱/选择离子质谱联用法,同时测定化妆品中8种邻苯二甲酸酯和4种对羟基苯甲酸酯。该方法线性范围广、重现性好、快速简便、干扰小。样品的加标回收率为80%~100%;含量检测的相对标准偏差小于10%;方法的检出限为0.1~5.0 $\mu\text{g}/\text{kg}$ 。用该方法对15种实际样品中的12种残留物进行定量检测,结果表明除了一种样品中不含待测物外,其余样品均检测到3~7种待测物。其中以对羟基苯甲酸甲酯、对羟基苯甲酸丙酯、邻苯二甲酸丙酯、邻苯二甲酸环己酯和邻苯二甲酸乙基庚基酯为主。

关键词 [气相色谱/质谱法](#); [固相萃取](#); [邻苯二甲酸酯](#); [对羟基苯甲酸酯](#); [化妆品](#)

分类号

Simultaneous Determination of Phthalates and Parabens in Cosmetic Products by Gas Chromatography/Mass Spectrometry Coupled with Solid Phase Extraction

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Abstract

Studies have been carried out on the simultaneous determination of 8 phthalates, i.e. di-ethyl phthalate (DEP), di-propyl phthalate (DPP), di-isobutyl phthalate (DIBP), di-butyl phthalate (DBP), benzyl butyl phthalate (BBP), di-cyclohexyl phthalate (DCHP), di-(2-ethylhexyl) phthalate (DEHP), di-octyl phthalate (DOP) and 4 parabens, i.e. methylparaben (MPB), ethylparaben (EPB), propyl paraben (PPB), and butyl paraben (BPB) by gas chromatography in combination with mass spectrometry (GC/MS) in electron ionisation mode (EI) with selected-ion monitoring (SIM) acquisition method. The phthalates and parabens in 15 cosmetic products, including hair sprays, perfumes, deodorants, cream, lotion, etc. were determined. The determination of the samples were performed after sonication-assisted extraction with methanol, cleaned up with an LC-C18 column (3 mL) and analyzed by GC/MS method. The base peak (m/z 149) of the phthalates and the base peak (m/z 121) of the parabens were selected for the screening studies. The characteristic ions, m/z 121, 149, 177, 222 for DEP; m/z 149, 191, 209 for DPP; m/z 57, 149, 223 for DIBP; m/z 104, 149 for DBP; m/z 91, 132, 149, 206 for BBP; m/z 55, 149, 167 for DCHP; m/z 113, 149, 167, 279 for DEHP; m/z 149, 279 for DOP; m/z 65, 93, 121, 152 for MPB; m/z 93, 121, 138, 166 for EPB; m/z 93, 121, 138, 180 for PPB; and m/z 93, 121, 138, 194 for BPB were chosen for quantitative studies. These techniques are capable to detect phthalates and parabens at the level of 0.1-5.0 $\mu\text{g}/\text{kg}$. Overall recoveries were 80%-100% with relative standard deviations (RSDs) less than 10%. Only one of the 15 examined samples was free from phthalates and parabens. The rest 14 samples were found to contain at least 3 or more of these phthalates and/or parabens. The predominant phthalates detected in the studied

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samples were MPB, PPB, DPP, DCHP and DEHP. The residue levels were at 1.42-4278 mg/kg.

Key words [gas chromatography/mass spectrometry \(GC/MS\)](#)
[solid phase extraction \(SPE\)](#) [phthalates](#) [parabens](#) [cosmetic products](#)

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