

研究论文

气相色谱-质谱法同时检测动物组织中多种激素类兽药的残留量

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摘要 建立了不同动物基质(肌肉、肝脏、肾脏)中己烷雌酚、己烯雌酚、己二烯雌酚、还原尿睾酮、表睾酮、雌酮、雌二醇、炔雌醇和雌三醇激素残留量的气相色谱-质谱联用(GC-MS)检测方法。以乙腈为提取溶剂,固相萃取柱净化,微波辅助衍生化,用双(三甲基硅烷基)三氟乙酰胺(BSTFA)与甲基氯硅烷(TMCS)(体积比为99:1)的硅烷化试剂在吡啶存在下进行衍生化反应。实验结果表明,9种激素的检出限为0.1~1 μg/kg。3种动物基质中9种激素的平均回收率为68.8%~93.1%,实验室内相对标准偏差(RSD)为4.1%~22.3%,实验室间RSD为3.1%~17.9%。方法的技术指标满足动物组织中激素类兽药残留分析的要求。

关键词 [气相色谱-质谱法](#) [固相萃取](#) [微波辅助衍生](#) [激素](#) [兽药](#) [多残留检测](#) [动物组织](#)

Determination of hormone multi-residues in animal tissues by gas chromatography-mass spectrometry

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Abstract

A method of gas chromatography-mass spectrometry (GC-MS) for the simultaneous determination of nine sex hormone residues, such as hexestrol, diethylstilbestrol, dienestrol, etiocholan-3 α -ol-17-one, epitestosterone, estrone, estradiol, ethinylestradiol and estriol, in animal tissues was developed. The sex hormones were extracted with acetonitrile, then cleaned-up with a C18 solid-phase extraction (SPE) column. The microwave-assisted derivatization of the target components with N,O-bis(trimethylsilyl)trifluoroacetamide (BSTFA) and trimethylchlorosilane (TMCS) (99:1, v/v) using pyridine as solvent was performed, and then the derivatives were analyzed by GC-MS. The limits of detection were 0.1~1 μg/kg for all hormones, and the limits of quantification were 0.2~2 μg/kg. The average recoveries of sex hormones were 68.8%~93.1%. The relative standard deviations (RSDs) of inter- and intra-assay were 4.1%~22.3% and 3.1%~17.9%, respectively. The real sample tests showed this method can be used for the sensitive and accurate determination of multi-sex hormones residues in biological samples.

Key words [gas chromatography-mass spectrometry \(GC-MS\)](#) [solid-phase extraction \(SPE\)](#) [microwave-assisted derivatization](#) [hormones](#) [veterinary drugs](#) [multi-residue determination](#) [animal tissues](#)

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