# 全自动 QuEChERS 样品制备系统结合超高效液相色谱-串联质谱法测定花椒中 20 种农药残留

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**摘要**:建立了全自动 QuEChERS 样品制备系统结合超高效液相色谱－串联质谱法测定花椒中 20 种农药残留分析方法。方法利用全自动 QuEChERS 样品制备系统涡旋振动和离心功能，将 QuEChERS 方法中样品提取和分散固相萃取净化完美结合；优化了全自动 QuEChERS 样品制备系的操作参数，在多反应监测（MRM）模式下检测，基质外标法定量。从方法学验证角度对全自动 QuEChERS 法和手动 QuEChERS 法进行了比较。结果表明，该方法中 20 种农药在 0.005-0.2mg/L 浓度范围内呈现良好的线性关系，相关系数（$R^2$）均大于 0.995，检出限范围为 0.4~3.0 μg/kg，定量限范围为 1.8~9.6 μg/kg，方法加标回收率为 62.5~111.2%，RSD 小于 11.8%（n=6）。该方法验证结果与手动 QuEChERS 法结果对比，结果无明显差异，应用该设备进行食用林产品中农药残留检测进行有大大提高检测效率和降低劳动强度。

**关键词**：QuEChERS；液相色谱-串联质谱；农药残留；花椒

## Automated QuEChERS Sample Preparation System Coupled with Ultra High Performance Liquid Chromatography-Tandem Mass Spectrometry for Determination of 20 Pesticide Residues in *Zanthoxylumbungeanum Maxim*

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**Abstract:**An analytical method was developed for the determination of 20 pesticide residues in *Zanthoxylumbungeanum Maxim*. using an automated QuEChERS sample preparation system coupled with ultra high performance liquid chromatography-tandem mass spectrometry. The automated QuEChERS system integrates vortex vibration and centrifugation functions, seamlessly combining the extraction and dispersive solid phase extraction cleanup steps of the QuEChERS method. The operating parameters of the automated system were optimized. Analytes were

detected in multiple reaction monitoring (MRM) mode and quantified by matrix-matched calibration. The automated and manual QuEChERS methods were compared from a methodological perspective. The results showed that the 20 pesticides exhibited good linearity ($R^2$ > 0.995) over a concentration range of 0.005-0.2 mg/L. The limits of detection were in the range from 0.4-3.0 μg/kg and limits of quantification were in the range from 1.8 - 9.6 μg/kg. The average recoveries ranged from 62.5% to 111.2% with relative standard deviations (RSD) less than 11.8% (n=6) at three spiked levels. No significant differences were observed between the automated and manual QuEChERS methods. The application of the automated system greatly improved the efficiency and reduced the labor intensity of pesticide residue analysis in edible forest products.

**Keywords:**QuEChERS; pesticide residue; liquid chromatography-tandem mass spectrometry ; *Zanthoxylumbungeanum Maxim*.

花椒不仅是中国传统的香辛料[1]，近些年在医药、防腐、防虫、杀菌及工业等方面具有重要的使用价值[2-4]。近几年来花椒在国内种植面积越来越大，病虫害状况也日趋严重[5,6]，但至目前为止仍没在花椒上登记使用的农药品种，导致农药滥用严重。最新版《食品安全国家标准食品中农药最大残留限量》（GB 2763-2021）[7]，仅规定花椒中 8 种农药的最大残留限量值（MRL），只规定胺苯磺隆、敌敌畏和杀虫畏三种农药为正式限量值，其它 5 种均为临时限量值。花椒中化学成分复杂[8, 9]，含挥发油、生物碱、酰胺、脂肪酸、木脂素、甾醇等种类丰富的化合物，这些物质对花椒中农药残留测定产生严重的基质效应，从而影响农药残留定性定量的稳定性和准确性。因此，找到一种花椒中农药残留测定既快速又准确的前处理方法十分重要。QuEChERS 作为一种快速高效前处理技术，极大简化了样品前处理过程，目前国际国内农药多残留检测的首选前处理方法[10-14]。传统手动 QuEChERS 方法操作劳动强度大，且由于人员操作差异会加大实验结果的不稳定性。因此，本实验采用全自动 QuEChERS 样品制备系统进行样品前处理，结合双层结构的 QuEChERS 整合管，利用仪器的三维立体震荡整合离心技术，实现一次完成提取和净化两个步骤，操作简便、方便且快捷，20 种目标农药的加标实验，得到了满意实验结果。

# 1 仪器与试剂

Waters I Class/Xevo TQS-micro 超高效液相色谱-串联质谱仪（配有电喷雾（ESI）源及