

技术交流

微波萃取-GPC净化-GC/MS法检验血中氨基甲酸酯和沙蚕毒素类农药

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摘要 利用微波萃取、凝胶色谱仪净化浓缩联用仪和气相色谱质谱联用仪技术, 分析血中氨基甲酸酯和杀蚕毒素类农药的检验方法。结果表明, 农药经V(丙酮):V(二氯甲烷):V(环己烷)=4:3:3的混合溶剂微波辅助萃取, 凝胶色谱自动净化浓缩至2 mL, 待GC/MS/SIS测定, 各类农药的回收率为68%~91%, 检测限为0.001~0.47 mg·L⁻¹, 部分药物的线性相关系数为0.990 0~0.997 6。该方法操作简便、机械化程度高、处理批量大、重现性好、空白干扰小, 可用于医疗、卫生、法庭科学实际案例的药物毒物筛选。

关键词 气相色谱-质谱法(GC/MS) 微波萃取 凝胶色谱净化 氨基甲酸酯和杀蚕毒素类农药 血样

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Determination of Carbamate Pesticides and Kill Silkworm Toxin in Human Blood Using Microwave Extraction-PTV-GC/MS with Gel Permeation Chromatography Clean-Up

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Abstract Carbamate pesticides and kill silkworm toxin in human blood were analyzed by microwave extraction-PTV-GC/MS and gel permeation chromatography (GPC) clean-up. The results showed that pesticide residues were extracted from blood with solvents by microwave assisted extraction, extracts were cleaned up by GPC, and analyzed by gas chromatography with mass selective detection model. Programmed temperature vaporization(PTV) large volume injector(LVI)was applied to inject the sample. The rates of recovery range from 68% to 91%. The detection limits of pesticides are 0.001-0.47 mg·L⁻¹. The method can be used for simultaneous determination of pesticide residues in blood. It is sensitive and simple, and meets the determinative requirement of pesticide residues.

Key words GC/MS microwave extraction gel permeation chromatography (GPC) clean up carbamate pesticides and kill silkworm toxin blood

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