#### 研究论文

快速溶剂萃取-高效液相色谱法测定含脂羊毛中残留的除虫脲和杀铃脲 樊苑牧\*,黄绍棠,俞雪钧,顾晓俊,裘亚钧,陈树兵,房科腾,陈俊 宁波出入境检验检疫局,浙江,宁波 315012

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摘要 建立了快速溶剂萃取—高效液相色谱 (ASE-HPLC)测定含脂羊毛中除虫脲、杀铃脲残留量的方法。以正己烷饱和的乙腈为萃取剂,在80 °C、10.34 MPa条件下用快速溶剂萃取仪提取样品中的目标物,提取液经冷冻除脂、浓缩及Waters Plus Silica柱净化后,采用Waters Atlants dC18色谱柱分离,以乙腈和水为流动相进行梯度洗脱,二极管阵列检测器于254 nm处检测。结果表明,在0.1~10.0 mg/L范围内,除虫脲、杀铃脲的峰面积与其质量浓度的线性关系良好,相关系数均大于0.999 9,定量检出限  $(S/N \ge 10)$  分别为0.05,0.04 mg/kg。该方法具有操作简便、快速、灵敏度高等特点,完全能满足含脂羊毛中除虫脲、杀铃脲残留初筛检测的要求。

关键词 快速溶剂萃取 高效液相色谱法 除虫脲 杀铃脲 含脂羊毛 杀虫剂

# Determination of diflubenzuron and triflumuron residues in greasy wool by accelerated solvent extraction technique and high performance liquid chromatography

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#### Abstract

A method for the determination of diflubenzuron and triflumuron residues in greasy wool was developed by high performance liquid chromatography (HPLC) coupled with accelerated solvent extraction (ASE). The diflubenzuron and triflumuron residues were extracted with acetonitrile saturated with n-hexane at 80 °C and 10.34 MPa. The extract was pretreated by a series of procedures such as freezing-lipid filtration, concentration and purification by solid-phase extraction prior to the determination with HPLC. The target analytes were separated on a Waters Atlants dC18 column (250 mm×4.6 mm, 5  $\mu$ m), gradiently eluted with acetonitrile and water as the mobile phases and detected by a photodiode array detector (DAD) at 254 nm. The linear ranges were 0.1~10.0 mg/L. There were good linearity between the peak areas and concentrations in the linear range for the analytes, and the correlation coefficients of diflubenzuron and triflumuron were higher than 0.999 9. The limits of quantification for diflubenzuron and triflumuron were 0.05 and 0.04 mg/kg (S/N≥10), respectively. The method is simple, rapid, sensitive and suitable for preliminary screening of diflubenzuron and triflumuron residues in greasy wool.

Key words <u>accelerated solvent extraction (ASE)</u> <u>high performance liquid chromatography (HPLC)</u> <u>diflubenzuron</u> <u>triflumuron</u> <u>greasy wool</u> <u>pesticide</u>

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