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腌制食品中敌敌畏和敌百虫检测的前处理方法研究

Study on the pretreatment methods for determination of dichlorvos and trichlorfor in pickled foods

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中文摘要:

采用超高效液相色谱-串联质谱法(UPLC-MS/MS),对腌制食品中敌敌畏和敌百虫的前处理方法进行研究。方法 样品用含0.1%甲酸的乙腈溶液超声提取后,分别经分散固相萃取(d-SPE)或凝胶渗透色谱(GPC)净化；采用BEH C_{18}色谱分离柱,以乙腈-0.1%甲酸水溶液梯度洗脱,正离子多反应监测模式测定,外标法定量。结果 敌敌畏和敌百虫在一定浓度范围内呈良好线性,r均＞0.999。d-SPE净化方法对敌敌畏和敌百虫的检出限均为1.0 μg/kg,定量限为2.0 μg/kg；而GPC净化方法的检出限和定量限分别为0.5和1.0 μg/kg。以空白样品(选取火腿和腊肉为代表性基质)进行2、10、20和40 μg/kg水平的加标回收试验,d-SPE和GPC两种净化方法对敌敌畏和敌百虫的平均回收率均分别在81.7%～104.2%之间和72.4%～89.8%之间,RSD均＜7.8%。采用d-SPE和GPC两种净化方法对阳性样品的测定结果无明显差异。结论 本方法快速、准确、灵敏度高,适用于火腿和腊肉等腌制食品中非法添加敌敌畏和敌百虫的监测。对采集的8份火腿、腊肉和咸鱼干样品进行检测,1份火腿样品检出痕量敌敌畏。

Abstract:

The pretreatment methods for determination of dichlorvos and trichlorfor in pickled foods were developed using ultra high performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS). MethodsSamples were ultrasonically extracted with acidified acetonitrile and cleaned up with dispersivesolid phase extraction (d-SPE) or gel-permeation chromatography (GPC) respectively. The extract components were separated and gradient eluted on a BEH C_{18} column with acetonitrile-0.1% formic acid solutions, and external standard calibration was used for quantification. ResultsThe linearity was satisfying within certain range of concentration, and the coefficients of determination (r) were above 0.999 for dichlorvos and trichlorfor. The limits of detection (LODs) and the limits of quantitation (LOQs) were 1.0 and 2.0 μg/kg for d-SPE cleanup, 0.5 and 1.0 μg/kg for GPC cleanup, respectively. The recoveries of the spiked samples at the levels of 2,0, 20, and 40 μg/kg ranged from 81.7% to 104.2% for d-SPE cleanup, and from 72.4% to 89.8% for GPC cleanup, with the relative standard deviations (RSDs)less than 7.8%. There was no significant difference between the results obtained by the two kinds of cleanup.ConclusionThe proposed method is rapid, simple, sensitive and specific enough for the supervision of the illegal use of dichlorvos and trichlorfor in pickled foods. Dichlorvos was detected in 1 out of 8 pickled foods collected from local market.

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