

水產藥品歐索林酸於三種鱸形目魚種殘留試驗分析

動物用藥品檢定分所

詹勳隆 助理研究員

摘要

本研究目的在於建立歐索林酸藥物殘留試驗以推算出石斑魚、黑鯛及金目鱸之停藥期，以供作水產動物用藥新增藥品及使用規範檢討參考依據。依據衛福部公告試驗方法，以電灑離子化離子源，在正離子採用模式下的高效液相層析串聯質譜法分析歐索林酸在組織中的殘留量。在正常分析條件下，其檢量線線性範圍為 0.005-1 $\mu\text{g/mL}$ ，線性迴歸決定係數(R^2)為 0.99996，定量極限為 0.005 $\mu\text{g/mL}$ ，偵測極限為 0.003 $\mu\text{g/mL}$ 。回收率以添加 10-100 $\mu\text{g/kg}$ 進行評估，三種鱸形目肌肉(含皮)回收率範圍為 72.19-93.60 %，而肝臟添加回收率範圍為 82.67-99.30 %；天內精密度於肌肉(含皮)添加 10-100 $\mu\text{g/kg}$ 濃度相對標準偏差(RSD)範圍為 1.31-9.20 %，肝臟範圍為 1.47-6.83 %；天間精密度於肌肉(含皮)添加 10-100 $\mu\text{g/kg}$ 濃度 RSD 範圍為 4.34-13.31 %，肝臟分別為 2.32-8.80 %。殘留試驗以歐索林酸低、高劑量 30、60 mg/kg 連續投予 5 天後，分別於 1、3、5、7、10、14、21 天等採樣，檢測其肌肉(含皮)、肝臟殘留之歐索林酸濃度。黑鯛、金目鱸及石斑魚高、低劑量組肌肉(含皮)、肝臟於投藥後分別於第 5 天、第 7 天及第 10 天殘留濃度均低於偵測極限(0.003 $\mu\text{g/kg}$)，故以檢測不到殘留量的時間再加上二分之一的安全期間即為停藥期，建議本藥物於黑鯛、金目鱸及石斑魚藥期分別為 8 天、11 天與 15 天。

Determination of oxolinic acid residues in three species of Perciformes

Hsun-Lung Chan

Abstract

The purpose of this study was to assess oxolinic acid residues in grouper (*Epinephelus coioides*), silver perch (*Lates calcarifer*) and black seabream (*Acanthopagrus schlegelii*) and generate a basis for determining withdrawal periods, which will be used as references during the review of newly approved aquatic animal drugs and their proper administration. Based on the standardized tests established by the Ministry of Health and Welfare, oxolinic acid residues in fish tissue were analyzed by ultra-performance liquid chromatography combined with electrospray ionization triple quadrupole tandem mass spectrometry (UPLC–MS/MS) and quantification was achieved by using multiple reaction monitoring (MRM). Under standard analytical conditions, calibration curves of oxolinic acid showed good linearity within the range of 0.005–1 µg/mL, with square correlation coefficients of 0.99996, and limits of detection (LOD) and limits of quantification (LOQ) of 0.003 µg/g and 0.005 µg/g, respectively. At spiked levels of 10–100 µg/kg oxolinic acid, muscle tissue (including skin) displayed average recovery rates of 72.19–93.60% while liver recovery rates ranged from 82.67–99.30%, in the three species of Perciformes. The intra-assay relative standard deviations (RSDs) of the muscle and liver assays were 1.31–9.20% and 1.49–6.83% respectively, while the inter-assay RSDs were 4.34–13.31% and 2.32–8.80%, respectively. We measured oxolinic acid residues in these three fish species which were orally administered low and high oxolinic acid doses of 30 and 60 mg/kg respectively, for five days. Muscle and liver samples from fish were both collected at definitive time points on the 1st, 3rd, 5th, 7th, 10th, 14th and 21st days. The results showed that oxolinic acid residue levels in muscle and liver tissues in fish administered with low and high doses were both under the LOD (0.003 µg/g) by the 5th day in black seabream, the 7th day in silver perch, and by the 10th day in grouper. The withdrawal time is calculated as the time it takes for oxolinic acid residue to be below the LOD from the time of oral administration, plus half of this time. These results thus suggest recommended withdrawal periods of 8 days for black seabream, 11 days for silver perch, and 15 days for grouper.