

LC-MS/MS による豚肉および豚肉加工食品中の
クレンブテロール残留分析法

(2015年9月30日受付)

(2016年1月18日受理)

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Determination of clenbuterol residue in pork muscle and processed pork food by LC-MS/MS

(Received September 30, 2015)

(Accepted January 18, 2016)

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Abstract

An analytical method that uses LC-MS/MS was developed for the analysis of clenbuterol residue in pork muscle and processed pork food. The method was evaluated by conducting recovery tests according to the guidelines for validation of analytical methods by the Ministry of Health, Labour and Welfare of Japan. Samples were initially extracted with acetonitrile, anhydrous sodium sulfate, and sodium chloride. The extracts were subjected to dispersive solid-phase extraction (dsPE) based on C18 substrate and PSA cartridge column SPE. PSA effectively removed residual fatty acids and other interferences remaining in the extracts. Clenbuterol in the purified solution was determined quantitatively using LC-MS/MS. The recoveries of clenbuterol fortified at 0.05 ng/g in pork muscle and processed pork food were examined. For pork muscle and processed pork food measured by the matrix calibration curve method, accuracy (trueness) was 93.4% and 92.4%, repeatability was 3.0% for both, and within-laboratory reproducibility was 8.4% and 13.2%, respectively. For pork muscle and processed pork food measured by the internal standard method, accuracy (trueness) was 100.1% and 97.2%, repeatability was 5.1% and 2.8%, and within-laboratory reproducibility was 5.7% and 3.7%, respectively. These results indicate that both the matrix calibration curve method and the internal standard method meet the target values stated in the guidelines. Whereas the analytical method developed in this study is adequate for measuring current regulation levels of residues, in the examination of various types of processed food, such as processed pork food that requires different matrices, quantification by the internal standard method is likely to be more practical and desirable.

Keywords : クレンブテロール、 β 作動薬、動物用医薬品、妥当性評価、液体クロマトグラフ・タンデム型質量分析計

clenbuterol, β -agonist, veterinary drug, single-laboratory validation study, LC-MS/MS

I はじめに

動物用医薬品は、家畜や家禽、養殖魚等に対して病気の予防や治療または成長促進を目的に使用される医薬品である

が、使用に際して、法律で定められている用法用量や休薬期間を遵守しなかった場合、畜水産食品中への移行、残留が食品衛生上問題となっている^{1,2)}。 β 作動薬であるクレンブテロール (Fig. 1) は、平滑筋にある β 受容体を選択的に刺