

Method development validation for corticoids in animal feed samples by liquid chromatography using a monolithic column

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Abstract-

A LC method for corticosteroids (CC) determination in poultry feed using a Chromolith column and UV detection has been developed and validated. The method development involved the optimization of different hydro-organic mobile phases using methanol or ACN as organic modifiers, flow rate, and temperature. The optimum separation was achieved at 40 degrees C using ACN/water (21:79 v/v) as mobile phase and 3 mL/min flow rate, allowing the separation to baseline of four out of seven CC in about 10 min. Prior to LC, a sample preparation procedure previously assayed for anabolics was used. It includes a leaching process, saponification of the esters from fatty acids, and SPE. Method validation was carried out according to the EU criteria established for quantitative screening methods. The extraction efficiencies, decision limits (CC α), and detection capabilities (CC β) for these compounds were in the ranges of 86-92%, 27-36 microg/kg, and 33-43 microg/kg, respectively. The repeatability and the within-laboratory reproducibility at 1, 1.5, and 2 CC β concentration levels were smaller than 9.0, 5.0, and 4.2% and 9.4, 6.4, and 4.9%, respectively. The CV values of the robustness test were less than 3.8% and the accuracy was in the range of 98-103%. The proposed method was applied to other feed with satisfactory results.

Index Terms- Animal feed; Corticoids; Monolithic column

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