

Method development and validation for melamine and its derivatives in rice concentrates by liquid chromatography. Application to animal feed samples

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Abstract-

An isocratic LC method for the determination of melamine and its degradation products (ammelide, ammeline, and cyanuric acid), used to increase the apparent protein content of rice protein concentrate, has been developed. Method development involved optimization of different RP columns, aqueous mobile phases, pH, phosphate concentration, and temperature. The optimum separation of these compounds was achieved using a Luna CN column (30 degrees C), 5 mmol L⁻¹ sodium phosphate (pH 5.0) as mobile phase, 1 mL min⁻¹ flow-rate, UV absorbance-DAD detection at 220 nm, and resorcinol as internal standard; this enabled separation of these compounds with baseline resolution (values in the 2.1-10.1 range) in about 8 min. Prior to HPLC, the developed sample preparation procedure consisted in a leaching process using the above mentioned mobile phase. Method validation was carried out in rice protein concentrates in accordance with the European Commission decision 2002/657/EC criteria. For this purpose, eight mandatory performance characteristics for the conventional validation approach were determined: calibration graphs, extraction efficiencies, decision limits, detection capabilities, precision (repeatability and within-laboratory reproducibility), accuracy, selectivity, and robustness. The extraction efficiencies for these compounds were in the range 99-100% and the within-laboratory reproducibility at 1.0, 1.5, and 2.0 detection capabilities concentration levels were smaller than 5, 4, and 3%, respectively. Finally, the proposed method was successfully applied to the analysis of other rice protein concentrates and several animal feed samples.

Index Terms- Rice protein concentrate; Animal feed; Melamine; Cyanuric acid; Ammelide; Ammeline; Liquid chromatography;

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