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Quantitative multi-method for the detection of veterinary drugs in distillers grains by liquid chromatography high-resolution accurate mass spectrometry

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Abstract Distillers grain (DG) is an important co-product of ethanol production. Ethanol production process uses only the starch portion of the corn and all the remaining nutrients, protein, fat, minerals, and vitamins are concentrated into distillers grains, a valuable feed supplement for livestock. The use of veterinary drugs is helpful to limit harmful bacterial growth during the early part of the fermentation process. This can lead to possible contaminants in the by-products that are used in the food and feed industry, resulting in a major concern for the development of bacterial resistance in both humans and animals. To facilitate the detection of veterinary drugs in DGs, a liquid chromatography-high resolution mass spectrometry (LC-HRMS) method was developed including a wide range of chemical families, such as ionophore and non-ionophore coccidiostats, banned coccidiostats, macrolides, tetracyclines, nitroimidazoles, amphenicols, quinolones, sulphonamides, tranquilizers, non-steroidal anti-inflammatory drugs and benzimidazoles. Following a simple and fast extraction step with a mixture of organic solvents, the extract was directly injected into the LC-Orbitrap mass analyzer. The identification of residues is based on accurate mass measurement. The high mass resolution of 50.000 full width at half maximum and corresponding narrow mass windows permitted a very selective and sensitive detection of the analytes in such a complex matrix. A single-laboratory validation procedure was constructed evaluating specificity, sensitivity, linearity, precision and accuracy. The method showed satisfactory analytical performance for quantification and allowed the determination of the compounds at low concentration. The proposed multi-method demonstrated that liquid chromatography utilized with an Orbitrap mass spectrometer is a powerful analytical technique, suitable for official residue control of a variety of veterinary drugs in distillers grains ensuring feed safety policies.



Keywords multi-method; veterinary drugs; distillers grains; validation; liquid chromatography-high resolution mass spectrometry; orbitrap;

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