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DETERMINATION OF PESTICIDES AND MYCOTOXINS IN FEED. APPLICABILITY OF AN ANALYTICAL METHOD IN ROUTINE ANALYSIS.

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In routine analysis it is often necessary to introduce minor changes in protocols or extend the applicability of previously validated methods to similar matrices. The SANTE guidelines contemplate on-going validation as a quality control process to verify analytical performance through a reduced number of experiments¹.

In this work, a validated QuEChERS method for the determination of pesticides and mycotoxins in corn and alfalfa was used for the analysis of a large number of different types of feed samples (cereals, pastures, silages). Briefly, 2 g of sample were hydrated with 10 ml H₂O (2% CH₂O₂), followed by extraction and partition with 10 ml acetonitrile, MgSO₄/NaCl (4:1). An aliquot of the extract was then cleaned-up using d-SPE with MgSO₄, C18 and PSA, prior to analysis by UHPLC-MS/MS².

A modification of the clean-up step had to be introduced due to the large number of samples to be processed and availability of sorbent salts at the time of analysis. Thus, for cereal-based samples only MgSO₄/C18 was used while MgSO₄/PSA was used for pasture-based samples.

Following the concepts of the on-going validation, the mean recovery (Rec%) was evaluated at the same concentration level from validation (100 µg/kg). Precision was evaluated as repeatability and intermediate precision, repeating the test 2 and 3 consecutive days for corn and alfalfa, respectively. The recovery results obtained after the modifications were compared with those from the initial validation through statistical t-tests. Finally, an additional quality control test was done by applying the methodology to a European Union Proficiency Test (EUPT CF18-2014) in wheat flour.

As a result, 80% of the studied compounds (117 pesticides and 2 mycotoxins) showed recoveries in the 60-140% range accepted for routine analysis by the SANTE guide, with RSDs <20% in both matrices.

From the statistical tests, the Rec% of more than 85% of the studied compounds were not affected by the changes introduced. Moreover, for compounds such as acephate, chlorpyrifos methyl, clethodim, fenthion and metribuzin in corn, and methomyl, metribuzin, thiabendazole, profenofos, spinosad D and terbuphos in alfalfa their Rec% improved with respect to the initial validation. Finally, the results from the EUPT test were compared to the declared concentration through a z-score, verifying the aptitude of the method for the determination of azoxystrobin, epoxiconazole, linuron, pyraclostrobin and carbendazim.

Keywords: Feed, Pesticide, On-going validation

References: 1. SANTE/12682/2019-https://ec.europa.eu/food/sites/food/files/plant/docs/pesticides_mrl_guidelines_wrkdoc_2019-12682.pdf.

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