ESTABLISHMENT AND APPLICATION OF A ROBUST MASS SPECTRAL LIBRARY FOR SCREENING POTENTIAL CONTAMINANTS IN FEED

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National monitor plans have been launched in many countries for routine survey of contaminants in food. For instant, Chinese MOA issued annual plans to monitor contaminants in animal borne food and feed. Through the targeted contaminants are changed year by year, they are quite limited to compared to potential contaminants, especially for abuse drugs and additives. There is a growing interest from researchers to screen for and identify non-targeted compounds in food, including metabolites and degradates, but also completely unexpected contaminants. Several versions of LC/MS/MS instrument and tools have being developed to face the challenging task. The detected compounds are identified based on empirical calculation of the molecular formula, interpretation of MS/MS fragments and mass spectral library searching. The acquired high resolution and accurate mass spectra are further used to quantify the amounts of detected and identified pollutants in a large set of practical food samples. 100 samples of finished pig feed were collected in nationwide areas with higher price or doubtful advertisement. AB TripleTOF™5000 LC/MS/MS system was used to screen for unexpected feed additives. LC-MS/MS data is processed using principal components analysis (PCA) to find combinations of variables, in this case based on retention times, mass signals and intensities, that explain most of the variance present in a data set. Every sample has a score and every variable has a loading for each principal component, scores and loadings of two or three principal components are plotted in 2D or 3D to visualize analytical results. The scores plot clearly differentiates samples originating from different sample types. Some feeds arrange in the top part of the scores plot. The corresponding loading plot assists to find characteristic marker ions for this grouping. The loading plot indicates mass 254.5 Da at 12.6 min. The high resolution and accurate mass spectra of the detected molecular ion and the automatically collected MS/MS spectrum were further processed using the Formula Finder of PeakView™Software to calculate empirical formulas for the molecular ion and detected fragment ions. Accurate mass information, the isotopic pattern and logic on distribution of chemical elements were used. The information gained from data processing was used to characterize the structure of the identified contaminant and identify the molecule as Dihydropyridine (1,4-dihydro-3,5-dicarbetboxy-2,6- dimethylpyridine, 1,4-Dihydro-2,6-dimethyl-3,5-pyridinedicarboxylic acid diethyl ester). After identification of the additive, a quantitation method of detecting dihydropyridine in feed was developed with an analytical standard by LC-MS/MS with SIM mode. 13 of 100 collected samples were detected for this additive with level from 120ppm to 278ppm.

Keywords: *non targeted contaminant, LC-Ms/Ms, feed, library*