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Parameter validation of analytical methods of insecticide residue analyses in foods of animal origin, feed and water

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The study was conducted to examine the interrelationship and coherence of analytical parameters in method validation. Seventeen organochlorines and fifteen organophosphates were studied in food matrices of animal origin: egg, milk, fish, mutton, feed and water under six analytical procedures. An auto-system gas chromatography equipped with 63Ni electron capture detector (ECD), flame thermo ionization detector (FTD), split injector and DB-5 capillary column with a 30 m length was used. The inner column diameter and film thickness were 0.25 mm and 0.25 μm, respectively, at a maximum usable temperature of 325^{0}C. Recovery, sensitivity, linearity, precision and limits of detection (LOD) were tested. Compounds that fell out of the stipulated recovery, 70-120%, in a matrix have concurrently failed to meet the requirements for sensitivity (≥0.7), linearity (R2 >0.99) and precision (<0.2) in the same matrix. Highest LOD was recorded in those compounds and matrices. Different from the conventional point estimate, a new approach was introduced for setting upper and lower confidence limits of the LOD in quantitative analyses.

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