

K3 Screening and Quantitation of Pesticides in Biological Specimens

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Learning Overview: After attending this presentation, attendees will be able to understand: (1) the ongoing potential of Gas Chromatography (GC) coupled with Mass Spectrometry (MS) in the detection of common pesticides, (2) suitable single-step Liquid-Liquid Extraction (LLE) procedures, and (3) the current misuse of different types of pesticides used in Pakistan. The objective of this study was to develop a simultaneous screening and quantitation method for the detection of pesticides by using a suitable solvent combination (for LLE) in the absence of a color test.

Impact on the Forensic Science Community: This presentation will impact the forensic science community by providing the results of the case studies regarding the use of pesticides for suicidal and homicidal purposes. Rapid GC/MS run with Selected Ion Monitoring (SIM) and scan mode for the analysis of common pesticides has not been previously reported and may be highly valued in clinical and forensic settings.

Pesticides are a common source of oral and inhaled poisoning in Pakistan. Cases have been reported regarding accidental and/or intentional poisoning of some commonly used pesticides. Rapid identification of pesticides in biological samples is not only necessary for early treatment decisions but also important for forensic toxicology cases.¹ Analytical methods devised for the determinations of pesticides in food and plant items are not directly useful for biological matrices as sample clean up pours are a major hindrance.²⁻¹⁰ Despite the benefits of Solid Phase Extraction (SPE), Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS), pressurized liquid extraction and Solid Phase Microextraction (SPME), LLE is still the most commonly used extraction technique for the past two decades.¹¹

Basified postmortem samples (2mL) were extracted in a single step by using ethyl acetate and dichloromethane (5mL of 1:1 v/v). Caffeine was used as the internal standard in this study. All extracts were reconstituted with ethyl acetate and hexane in (1:1 v/v), and 1 μ L was injected in GC/MS. Electron Impact Mass Selective Detector (MSD) was used on Selected Ion Monitoring (SIM) and scan mode whereby DB-5ms capillary column (30m x 250 μ m x 0.25 μ m) was installed in GC. The method was validated for screening and quantitation of carbofuran, atrazine, chlorpyrifos, buprofezin, bifenthrin, pyriproxyfen, lambda-cyhalothrin, cypermethrin, deltamethrin, acephate, acetamiprid, and imidacloprid.

The method was validated by using spiked synthetic blood samples with five-point calibration (calibration range 0.50mgL–3.75mg/L). Limit Of Detection (LOD) ranged from 0.202mgL–0.657mg/L, while the Limit Of Quantitation (LOQ) varied from 0.50mgL–1.00mg/L. Accuracy (82.0%–115.20%) and precision (as %CV; 2.14%–17.50%) at three different concentration levels (1.0mgL, 2.5mgL, and 3.75mg/L of pesticides) were within acceptable ranges (80%–120% and < 20%, respectively). Postmortem samples of homicide and suicide cases submitted were analyzed for the presence and quantitation of pesticides. Various cases were found to be positive for the stated pesticides in whole blood, gastric contents, and liver tissues.

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Pesticides, Homicides, Suicides