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Automation in Pesticide Residue Analysis in Foods: A Step toward Smarter Laboratories and Green Chemistry

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WHY AUTOMATION?

Worldwide, food testing laboratories are confronted with large sample loads of fresh and processed commodities, and any delay in evaluating perishable items may undermine their shelf life and commercial value. Because of the growing awareness of food safety, supply chain stakeholders must strictly adhere to the applicable food legislation and obtain all necessary certifications to promote international trade. Newer tools for managing a safe food supply chain are therefore warranted.

To comply with trade regulations, pesticide residue testing in foods is essential. Regulatory compliance analysis of multiclass pesticides requires numerous sample preparation steps and instrumental (GC-MS/MS and LC-MS/MS) runs that are labor-intensive and time-consuming. Over the past couple of decades, laboratory automation has evolved from moving samples to miniaturization through robotics. In simple terms, automation is the process of substituting human interactions with robotic interventions controlled by computer applications. Dilution, derivatization, pH adjustment, liquid extraction, mixing, centrifugation, and evaporation are all examples of potentially automated steps. While many laboratories lack automated systems due to their prohibitively high costs, automation in food testing has attracted widespread attention because of its ability to offer robust data in a timely manner.[1] In this Viewpoint, we make the case for automation in pesticide residue analysis, arguing that it provides numerous benefits to food testing laboratories by enabling them to deliver the highest-quality test results for ensuring food safety and promoting business.

FOOD ROBOTICS: MICRO-SPE

In QuEChERS and similar methods of residue analysis, dispersive solid phase extraction (dSPE) is used to clean a sample extract (in acetonitrile or ethyl acetate). Choosing a matrix-specific dSPE sorbent mixture is challenging because it requires several manual optimizations. Because the composition of the sorbent mixture in dSPE varies by food type, optimization and validation of a cleanup step take time. During the post-extraction steps, analytical bias caused by decomposition and evaporation loss of analytes can occur. Using micro-SPE (Figure 1), the method efficiency and laboratory throughput can be significantly increased. This automated technology cleans raw extracts using micro-SPE cartridges that are compatible with a variety of commodities, including spices[2,3] and fatty matrices.[4] Using an x,y,z-robotic autosampler, loading the raw extract onto a cartridge and its subsequent elution have become simple and require less human involvement. Notably, sample cleaning occurs simultaneously with analysis (known as "prep-ahead" mode), saving time. Internal standards and analyte protectants can be added either before or after cleanup to provide a platform for calibration, quantitation, and recovery calculation. Meticulous workflow documentation ensures traceability and GLP (good laboratory practices) compliance. Due to the reduced level of solvent consumption, the low level of exposure of analysts to solvents, and the lack of evaporation steps, micro-SPE is truly a green analytical technique.[5]

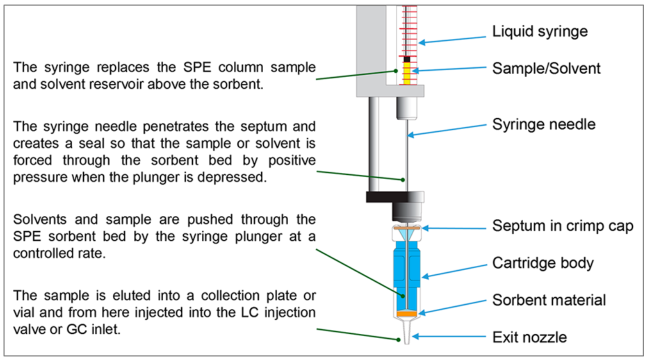

Figure 1. Micro-SPE automated operation for pesticide cleanup (courtesy ITSP Solutions Inc.).

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RELIEVE THE BURDEN OF MANPOWER

Training and retaining laboratory technicians to run manual processes can be a challenge. Residue testing by untrained personnel can jeopardize the laboratories' long-term viability and reputation. In such situations, automation ensures a technique's flawless performance. When used in conjunction with a preprogrammed procedure, an automated system saves time and alleviates the workload of staff. While the manufacturer's recommended workflow is preferred, it can also be customized locally. A robotic sample preparation system (with the appropriate configuration and user interface) has defined parameter settings for performing a workflow. By standardizing procedures and analytical operations, it assists in resolving quality-related noncompliances. The customized automated workflows are validated to meet GLP requirements for accuracy and precision. Because the processing parameters and error components are documented on a regular basis, the workflow can be traced for quality assurance/quality control (QA/QC) purposes.

Miniaturization. With the advent of the QuEChERS method in the new millennium, the sample size in residue analysis has significantly decreased.[6] Additionally, it has resulted in a decrease in solvent use and analysis time. Homogenization of samples of fruits and vegetables with liquid nitrogen has drastically reduced the test sample size.[7] Because automation works effectively with smaller samples, micro-SPE is a fit-for-purpose cleanup option, promoting eco-friendly chemistry and global business harmony.

DIGITAL TRANSFORMATION

Digitization and automation have significantly improved data quality and traceability, QA/QC, and laboratory logistics. Remote network access is often practiced for data processing and operating and monitoring instrument performances online. Aside from creating a paperless laboratory, data networking helps optimize and record operational services. Laboratory information management systems (LIMS) improve a laboratory's performance through digital transformation. LIMS ensures data transparency and eliminates time-consuming corrective actions. Data storage in a cloud allows distributed access with process safety, especially in high-throughput laboratories around the world.

Rapid and Accurate Preparation of Solutions. Preparing multicompound calibration standards is a critical and time-consuming task in a food laboratory, where any unidentified or uncorrected errors might lead to catastrophic consequences. When liquid handling procedures are carried out manually, errors can occur. The SANTE analytical quality control document specifies several options for minimizing such errors,[8] which can largely be achieved through a LIMS database that stores batch numbers, expiration dates, concentrations, sourcing channels, delivery times, and safety information for all registered analytes. Through bidirectional communication with a robotic preparation platform (Figure 2), the database plans the composition of routinely prepared working standard mixtures[9] and helps in their preparation. A comprehensive data recording system complements it by avoiding human data entry errors and ensuring ongoing calibration traceability. These computational gains thus meet the demands of contemporary food testing laboratories quite effectively.

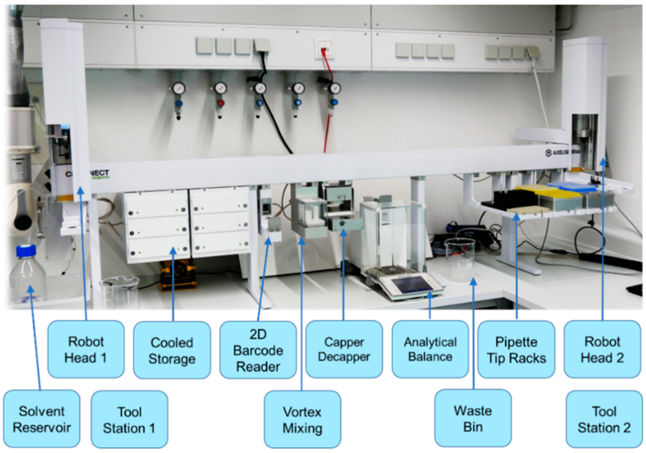

Figure 2. Automatic multicompound working standard preparation with a dual-head x,y,z robot (courtesy Axel Semrau GmbH).

Smart Consumables. Two-dimensional barcode labeling (Figure 3) or active data interchange via a memory chip is used to ensure the traceability of vials and consumables. Consumable component (e.g., syringe) failures can drastically slow the processing of a large sample series. Syringes for robotic sampling must thus be maintained or replaced on a regular basis. As illustrated (Figure 4), inserting a memory chip into a syringe plunger encodes and tracks the syringe's unique identifier and enables a robot to read the syringe's usage data for preventative maintenance. These digital solutions assist in documenting equipment maintenance cycles and quality control records. A user-friendly preventative maintenance cycle significantly improves workflow safety, although routine inspections of consumables for large sample series (e.g., septa, columns, syringes, solvents, and reagents) are required.

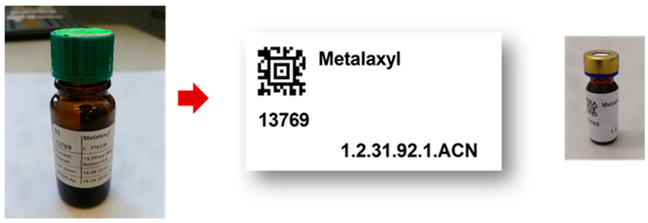

Figure 3. Standard two-dimensional barcode labeling by the LIMS system (courtesy Axel Semrau GmbH).

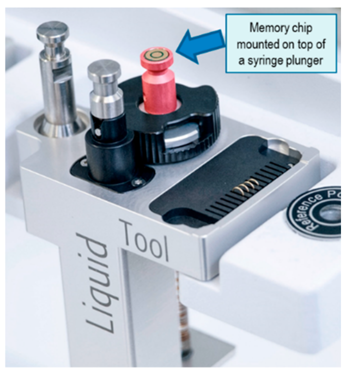

Figure 4. Memory chip in a syringe plunger (courtesy CTC Analytics AG).

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Compared to manual steps, automation reduces solvent volumes required for analysis and lowers reagent and waste removal costs. Another tangible benefit is that it minimizes or eliminates exposure of laboratory personnel to hazardous chemicals, thereby supporting public health. As practitioners, we advise accelerating digitization and implementing automated residue analysis workflows in routine laboratories.

RETROSPECTIVE DATA ANALYSIS BY HIGH-RESOLUTION ACCURATE MASS SPECTROMETRY (HRAM-MS)

The sheer number of pesticides complicates a multiresidue analysis program as it includes all possible analytes in a target list. The HRAM full-scan mass spectra recording enables nontargeted identification of unknown compounds.[10] Following this, additional analytes can be identified looking at the data sets.[11,12] The digitization of the full MS data thus paves the way for retrospective data analysis and confirmation.

LIMITATIONS OF AUTOMATION

In certain tasks, robots lack the dexterity of a human hand. Numerous standard methods require manual scaling of the sample size and volume of chemicals (solvent and reagents). Additionally, automated sample weighing and transport are difficult due to the variety of requirements and, therefore, require human involvement. Robots can handle only small tubes, pipettes, and flasks and cannot handle large separatory funnels or evaporative devices.[13] In addition, robots are subject to mechanical and electrical breakdowns and require a continuous power supply. These obstacles, however, do not preclude automation from being used in food testing laboratories.

CONCLUSION

We have discussed how automated equipment can run continuously, ensuring rapid turnaround times to meet global demand. Downscaling enables improved GLP compliance, cross-border procedure alignment, meaningful data exchange, and systematic auditing. It generates precise and timely quantitative data at a low cost. Additionally, it enables analysts to manage remote control runs, ensuring faster regulatory decisions. Automation can alleviate the workload of the technicians while allowing a laboratory to continue to provide quick results to customers. However, recruiting employees with relevant expertise in the field is of utmost importance. As the amount of chemical waste is decreased, analytical methodologies become more eco-friendly. Furthermore, automated extraction minimizes the exposure of humans to toxic chemicals, improving public health. Closing methodological gaps and validating workflows are some of the areas that require further research. Another area of concern is recruiting an informed staff. Human error is unavoidable, and this risk can be heightened when combined with a lack of trained personnel and high employee turnover. While a fully automated food testing laboratory is not yet a reality, certain procedures can be easily automated to improve efficiency. Despite its high cost, automation has the potential to dramatically improve the performance of analytical laboratories and advance green chemistry.

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Notes

The authors declare the following competing financial interest(s): H.-J.H. is involved as a consultant for analytical sample preparation methods for CTC Analytics AG in Zwingen, Switzerland, and Axel Semrau GmbH, part of Trajan Scientific and Medical, in Germany.

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