Automated Sample Preparation for GC-MS and LC-MS

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This handbook about the automated sample preparation for GC-MS and LC-MS applications addresses the current and future potential of automated workflows for standard and customized sample preparation. Applications range from food, environmental, pharma, to forensic analyses and beyond. Automated solutions are presented for gas and liquid chromatography with mass spectrometry detection and provide an important not-to-miss contribution to green analytical chemistry. The Handbook provides the essential guidance for the transfer of manual sample preparation procedures into automated instrumental workflows for routine applications with many benefits.

Abstracts of the Chapters:

Chapter 1 Introduction

The first steps in any analytical workflow have the highest impact on the quality of the analytical data. Errors in analytical measurements are the random variability, expressed as the precision of the method, systematic bias affecting the trueness of results, and gross mistakes, the handling errors. The introduction to the subject of automated sample preparation deals with the perspective on human performance in the so important sample preparation process. Survey results confirm the major sources of error in chemical analysis from the manual sample preparation steps.

The established standard methods are still focused on manual sample preparation which is often the rate-determining step for sample throughput and too often the error-prone part of the analytical method. In this context, standardization calls for automation, not vice versa. The solutions provided in this textbook address this gap with the integration of robotic workflows for many routine tasks.

Chapter 2 The Analytical Process

The benefits and limitations of automated sample preparation are discussed. Routine chemical analyses benefit from many undeniable advantages of instrumental sample preparation workflows. In particular, the standardized sample preparation methods provide high potential for workflow automation on integrated sample preparation platforms. The strong growth of high-resolution accurate-mass instrumentation (HR/AM) using orbitrap or quadrupole/time-of-flight MS systems supports automated concepts with the inherent strength in non-targeted analysis for unknown contaminations or metabolites.

Instrumental workflows establish the analytical sample preparation methods with a turnkey programmed operating sequence for the laboratory. Automated workflows deliver consistent data quality, independent of the human factor. Sample turnaround times and sample reprocessings are significantly reduced. A downscaling of sample and solvent volumes is a significant achievement not only for green analytical chemistry (GAC) but also for the logistics and economy of the analytical laboratory.

Robotic x,y,z-systems execute workflows and hence become an integral part of an SOP-guided sample processing and analysis process. Automation is the welcome side-effect of

the programmed workflow control and CDS integration. The sample capacity of robotic systems supports high sample throughput for 24/7 operation.

Chapter 3 Workflow Concepts

The sample preparation workflow design follows the established methods maintaining the proven principles and sample prep chemistry with the transfer of standard methods to automated workflows. Method translation to automated workflows is presented considering online/offline and common robot tool and module configurations. Typical sample processing strategies for automation with batch, parallel and sequential processing, prep-ahead, and overlapped sample handling are discussed.

Additional features, specific and required for automated processing and traceability as the sample identification, transport, the decapping of vials, and mechanism for the tool change are illustrated.

Chapter 4 Analytical Aspects

The analytical performance of automated workflows is of paramount importance. This central Chapter 4 illustrates the many details of automated processing procedures and devices with their impact on data quality. Important are the tools and critical operational parameters of liquid handling with different syringe types and pipette tools. Also, the automated handling of solid materials and weighing is reviewed.

Further, the solutions for extraction and extract clean-up as the key steps in sample preparation are discussed for the automated approach. This covers the classical liquid extractions with the standard clean-up procedures of extracts integrated online for seamless workflow integration followed with filtration, centrifugation, micro SPE and online SPE, as well as GPC. Current developments for miniaturization on robotic sampling systems with DLLME, SPME, SBSE, and TFME are presented in detail. Important aspects are also the automated solutions for the main derivatization methods for GC-MS and LC-MS applications.

In addition, typical workflow requirements for many different applications cover temperature control with devices for heating and cooling, mixing, and vortexing.

Chapter 5 Integration into Analysis Techniques

Established automated workflows on x,y,z-robots are available for many standard methods already. Typical solutions are the analysis of volatile compounds with the classical static and dynamic headspace analysis with purge and trap, needle trap, in-tube extraction, and thermal desorption techniques. The instrumental solutions for overcoming matrix effects and increased analyte sensitivity are discussed.

The hyphenation of sample preparation with online analysis, the online injection of robotic sampling systems to GC-MS and LC-MS are reviewed. It covers the hot needle and liquid band injection as well as the sandwich injection methods for GC, also solutions for the automated inlet liner exchange. The LC-GC hyphenation using the normal phase LC as a powerful clean-up and group separation is exploited in many automated workflows and

featured in detail. LC valve injections are reviewed for operation with syringes and pipette tools, and the important dynamic wash solutions for high sample throughput in life science applications.

Chapter 6 Solutions for Automated Analyses

This chapter goes into real-life applications. A selection of twenty proven applications from food safety, environmental, pharma and forensic applications are featured with detailed automated workflows. The selected examples provide automated solutions for general liquid handling tasks like dilution of standards, preparation of working standards, and calibration curves. Standard derivatization procedures are covered, in particular also for SPME, FAMEs, and the 2-step process for metabolomics profiling analysis.

The workflows for automation of sample preparation are presented with the background and operation principle of the application, required chemicals, solvents, and consumables, followed by a description of the suggested configuration of a generic x,y,z-robotic rail system together with a note on the function of the required modules.

The discussed applications comprise the analysis of volatile compounds from different sample matrices like water, beverages, fruits and foods covering taste and odor, sulfur compounds, and solvent residues.

Automated sample preparation and clean-up workflows for trace analysis are presented for semivolatiles compounds, the multi-class pesticides analysis with uSPE extract clean-up and online SPE. PAHs, personal care products and chemical warfare agents are analyzed in a variety of matrices like food, water, and soil. Food safety analysis for contaminations from mineral oil hydrocarbons (MOSH/MOAH) and MCPD introduce current method developments for complex routine tasks.

Appendix

A comprehensive appendix provides additional information for the setup, operation, and optimization of automated sample preparation workflows in the laboratory. In short, typical and current standard robotic system control software is reviewed.

A particular focus is set on necessary maintenance procedures of robotic systems for sample preparation process safety, in particular covering syringes and pipette tools.

The appendix also covers a collection of tables and information indispensable for workflow developments on syringe needle gauges, pressure units conversion, solvent miscibility, viscosity, and stability. Important for the use of organic solvents, acids and bases are the resistance information of used materials with contact to solvents or reagents for polymers, glass, and stainless steel.

Glossary

The comprehensive glossary explains acronyms and short forms of analytical relevant expressions, methods, and principles used and referenced in this textbook.