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Review

Chromatographic Applications Supporting ISO 22002-100:2025 Requirements on Allergen Management, Food Fraud, and Control of Chemical and Packaging-Related Contaminants

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Abstract

ISO 22002-100:2025 introduces stringent and more technically explicit prerequisite programme (PRP) requirements for allergen management, food fraud mitigation, and the control of chemical and packaging-related contaminants across the food, feed, and packaging supply chain. This review examines how advanced chromatographic methods provide the analytical basis required to meet these requirements and to support alignment with GFSI-recognized certification schemes. Recent applications of liquid and gas chromatography coupled with mass spectrometry for allergen quantification, authenticity assessment, and the determination of packaging migrants, auxiliary chemical residues, lubricants, and indoor pest-control pesticides are presented to demonstrate their relevance as verification tools. Across these PRP-related controls, chromatographic methods enable trace-level detection, structural specificity, and reproducible measurement performance, thereby shifting PRP compliance from a documentation-based activity to a process verified through measurable analytical evidence. The review highlights significant progress in method development and simultaneous multi-target analytical approaches while also identifying remaining challenges related to matrix-appropriate validation, harmonization, and analytical coverage for chemical contamination, which is now formally defined as a measurable PRP requirement under ISO 22002-100:2025. Overall, the findings demonstrate that chromatographic analysis has become essential to demonstrating PRP effectiveness under ISO 22002-100:2025, supporting the broader shift toward evidence-based, scientifically robust food safety assurance.

Keywords: ISO 22002-100:2025; GFSI; prerequisite programmes; chromatographic verification; allergens; food fraud; chemical contaminants; packaging migrants; LC-MS/MS; GC-MS/MS

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1. Introduction

The prevention of contamination, adulteration, and allergen cross-contact has long been a fundamental objective of food safety management systems. Beyond microbiological hazards, chemical and physical hazards, ranging from undeclared allergens and fraudulent substitutions to packaging-derived contaminants and process-related chemical residues, including cleaning agents, disinfectants, lubricants, and pest control substances, require

continuous analytical verification to ensure product integrity and consumer protection. To address these multifactorial risks within the global food supply chain, the ISO 22000 family provides a harmonized management system integrating risk assessment with principles of continual improvement, while the ISO 22002 series specifies prerequisite programmes that translate these management principles into operational and environmental controls. These PRPs define the operational and environmental prerequisites needed to control hazards that extend beyond the scope of Critical Control Points [1].

ISO 22002-100:2025 (Food, Feed, and Packaging Supply Chain) represents a major step toward an integrated approach to risk management across all organizations in the agri-food chain [1]. It defines common prerequisite requirements applicable to the food, feed, and packaging supply chain and forms the basis for all sector-specific standards in the series. Alongside ISO 22002-100:2025, the recently issued ISO 22002-7:2025 for retail and wholesale activities further strengthens the consistency of requirements throughout the agri-food supply chain [2]. Together, these standards consolidate and update previous sector-specific requirements related to food manufacturing hygiene, packaging processes, and retail or distribution operations, creating a unified reference for prerequisite programmes across the food and packaging sectors. The transition from the previous Technical Specifications (ISO/TS 22002 series) to a full International Standard strengthens the authority and global recognition of these prerequisite programme requirements.

Focusing on ISO 22002-100:2025, the standard expands the preventive scope to three technically demanding areas:

(a) allergen management, requiring validated analytical verification of cleaning and segregation procedures,
(b) food fraud mitigation and authenticity verification, demanding vulnerability assessments supported by appropriate analytical verification methods; and
(c) chemical contamination and packaging migration control, including non-intentionally added substances (NIASs), residues of cleaning and disinfection agents, technical fluids, lubricants, and pest control chemicals [1].

In addition to allergen management, food fraud prevention, chemical contamination, and packaging safety, the revised standard strengthens requirements on food defence, waste management, and equipment suitability, reflecting a more integrated approach to food safety risks. ISO 22002-100:2025 adopts the Annex SL structure for management systems, ensuring consistency in terminology, clause alignment, and risk-based methodology with ISO 22000:2018 and ISO 22003-1:2022 [3,4]. The standard emphasizes a life-cycle approach to hazard prevention, integrating supplier assurance, analytical verification, and traceability into a unified compliance model. It also introduces explicit requirements for analytical verification, recommending the use of standardized and sensitive instrumental techniques for detecting allergens, authenticity markers, and chemical hazards, including chromatographic determination of residues arising from sanitation chemicals, technical substances, and packaging-related migrants [1]. Such provisions establish analytical chemistry, particularly chromatographic science, from a supporting activity to a key element in verifying PRP effectiveness.

Recent research linking regulatory compliance with ISO-based food safety management systems [5] shows that the development of the ISO 22000 and 22002 series reflects a broader shift toward measurable, evidence-based control. Analytical data derived from chromatographic and spectrometric methods are increasingly used as objective indicators connecting operational performance with product safety outcomes. This integration of analytical verification into risk management reinforces the expectation that control measures must be scientifically justified and reproducible.

Therefore, chromatographic methods such as HPLC and GC, along with complementary detection techniques including spectroscopic and hybrid analytical systems, provide a fundamental analytical basis for implementing and verifying the new ISO requirements. They enable trace-level detection of allergens, identification of authenticity markers, and monitoring of chemical contaminants as well as packaging migrants, transforming qualitative management principles into measurable compliance evidence. The following sections examine recent chromatographic advances and their role in supporting the implementation of ISO 22002-100:2025, from analytical verification of allergens and authenticity to the chromatographic control of packaging-derived migrants, process chemicals, and pest control residues, as well as broader aspects of system integration and compliance alignment with Global Food Safety Initiative (GFSI) requirements.

Accordingly, the scope of this review is to present recent compliance-relevant requirements introduced or reinforced by ISO 22002-100:2025 in relation to PRP implementation and verification within food safety management, and to discuss how these expectations align with GFSI-recognized schemes. Within this context, emphasis is placed on where chromatographic evidence can provide the accuracy, specificity, and reproducibility required in industrial practice to verify PRP effectiveness (e.g., allergen management, authenticity-related verification, and control of chemical and packaging-related contaminants), rather than providing an overview of chromatographic technologies used for these determinations. More specifically, contemporary chromatographic methods applied in these verification activities are discussed in this review. Finally, areas where ISO 22002-100:2025 verification expectations may not yet be fully supported by matrix-appropriate chromatographic methods are identified, thereby highlighting analytical gaps and priorities for further method development.

2. Chromatographic Methods Supporting Allergen Management

2.1. Regulatory and Traceability Context for Allergen Control

Allergen control in food production is fundamentally guided by Regulation (EU) No 1169/2011 on the provision of food information to consumers, which mandates the clear labelling of substances known to cause allergies or intolerances. Annex II of the Regulation lists fourteen major allergens, namely, cereals containing gluten, eggs, milk, soybeans, peanuts, tree nuts, fish, shellfish, celery, mustard, sesame, lupin, sulphur dioxide/sulphites, and molluscs, that must always be declared regardless of their concentration in the product [6]. These legislative requirements establish the basis for preventive allergen management throughout the food supply chain. In alignment, ISO 22002-100:2025 requires organizations to implement validated cleaning, segregation, and traceability measures within their PRPs to prevent cross-contact and ensure compliance with applicable food information legislation [1]. The integration of regulatory and analytical verification measures under this standard reinforces evidence-based allergen control as a core component of modern food safety management systems. The fourteen allergens listed in Annex II of Regulation (EU) No 1169/2011 are summarized in Table 1, together with their main food sources and categories.

Table 1. Overview of the fourteen major allergens listed in Annex II of Regulation (EU) No 1169/2011.

Category	Allergen	Typical Food Sources
Cereals containing gluten	Wheat, rye, barley, oats	Bread, pasta, beer, breakfast cereals, malt-based products, sauces
Crustaceans	Shrimp, crab, lobster	Seafood dishes, sauces, soups
Eggs	Egg proteins (ovalbumin, ovomucoid)	Bakery products, mayonnaise, sauces, desserts
Fish	Cod, salmon, tuna	Processed fish products, ready meals, sauces
Peanuts	Groundnuts (peanuts)	Peanut butter, snacks, confectionery
Soybeans	Soy proteins (including lecithins)	Tofu, soy-based sauces, meat substitutes, processed foods
Milk	Casein, β-lactoglobulin	Milk, cheese, butter, yogurt, dairy-based desserts
Nuts	Tree nuts (almond, hazelnut, walnut, cashew, pistachio, Brazil nut, macadamia)	Confectionery, desserts, bakery products
Celery	Celery (root and stalk parts)	Soups, sauces, spice mixes, ready meals
Mustard	Mustard seeds	Condiments, dressings, processed foods
Sesame seeds	Sesame seeds and derived products (e.g., tahini, oil)	Bakery products, spreads, sauces
Sulphur dioxide/sulphites	Sulphur dioxide and sulphite preservatives (>10 mg/kg or L)	Dried fruits, wine, beer, processed vegetables
Lupin	Lupin flour and derived ingredients	Gluten-free products, bakery goods, pasta substitutes
Molluscs	Wheat, rye, barley, oats	Seafood dishes, mixed seafood products

Effective allergen management relies not only on regulatory compliance but also on the traceability and quantification of allergenic ingredients throughout the food chain. Traceability provides the means to document ingredient origins, processing steps, and potential cross-contact points, thereby ensuring transparency and accountability within allergen control systems [7]. Modern approaches integrate traceability data with risk-based management tools to establish measurable thresholds that define acceptable exposure levels for allergic consumers. The establishment of population-based reference doses, such as the ED05 or VITAL thresholds, enables a scientifically sound basis for allergen risk assessment, linking analytical results with consumer protection goals [8]. This principle has been internationally endorsed by the FAO/WHO Expert Consultation on Risk Assessment of Food Allergens, which recommended health-based reference doses (RfDs) derived from probabilistic dose-distribution modelling as a foundation for evidence-based allergen management [9]. These developments align closely with the preventive intent of ISO 22002-100:2025, which emphasizes documented verification and validated analytical methods within PRPs.

2.2. Immunochemical and Molecular Techniques for Allergen Detection

A range of non-chromatographic analytical techniques complement chromatographic approaches in allergen detection, providing rapid and sensitive tools for both qualitative screening and quantitative determination. Enzyme-linked immunosorbent assays (ELISAs) remain the most established methods for routine monitoring, combining high specificity and sensitivity through antigen–antibody interactions and adaptable assay formats such as

sandwich and competitive ELISA [10]. Lateral flow devices (LFDs) extend this principle into portable, on-site applications, offering semiquantitative results within minutes and facilitating allergen verification along production lines [11]. More advanced optical thin-film biochips employ multiplexed detection techniques capable of simultaneously identifying several allergens through refractive index-based signal transduction, enabling miniaturized and high-throughput analyses [12]. DNA-based polymerase chain reaction (PCR) methods serve as confirmatory tools when protein integrity is compromised by processing, as they target stable genetic markers specific to allergenic species [13]. Finally, IgE antibody-based assays, including singleplex and multiplex immunoassays, link analytical outcomes with clinical relevance by detecting allergenic epitopes recognized by human sera [14]. Together, these immunochemical and molecular tools provide the foundation for effective allergen surveillance, supporting compliance verification and consumer protection across the food supply chain.

2.3. Chromatographic and Spectrometric Techniques in Allergen Management

Chromatographic and spectrometric techniques are fundamental to allergen management, supporting both traceability and quantitative verification across the food supply chain. Their use provides a robust scientific basis for evidence-based risk assessment and compliance validation under current food safety standards. Liquid chromatography–tandem mass spectrometry (LC-MS/MS) has become the reference analytical method for allergen determination, allowing simultaneous identification and quantification of allergenic peptides in complex matrices. Targeted proteomics using multiple reaction monitoring (MRM) and high-resolution mass spectrometry (HRMS) offers confirmatory protein identification, reinforcing validated traceability frameworks within food safety systems [15].

Advances in proteomics have enhanced allergen characterization using untargeted LC-MS analysis, data-independent acquisition (DIA), and label-free quantification, enabling precise peptide-level quantitation and detection of processing-induced modifications that affect allergenicity [16]. LC-MS-based proteomics now surpasses immunoassays in selectivity and robustness, combining reversed-phase and ion-exchange chromatography with electrospray ionization (ESI) or matrix-assisted laser desorption/ionization (MALDI) for sensitive peptide fingerprinting. The use of stable isotope-labelled standards further ensures quantitative reproducibility and method validation [17]. Despite advances in biosensor technologies, LC-MS remains the reference technique for confirmatory allergen analysis, providing high specificity, multiplexing capability, and robust structural characterization. Integration with miniaturized and automated microsystems represents a step toward real-time allergen surveillance [18].

Recent advances in LC-MS methods highlight the importance of standardization and matrix-aware quantification. Targeted selected and multiple reaction monitoring (SRM/MRM) methods can quantify allergenic proteins in complex food with sensitivity comparable to or exceeding ELISA, while offering multiplexing and structure-based confirmation [19]. The adoption of proteotypic peptides as reference analytes, supported by curated databases such as the Allergen Peptide Browser, improves method reproducibility and inter-laboratory consistency [20]. Extraction efficiency and peptide preparation, through defatting, enzymatic digestion, and desalting, remain critical to reliable quantification, particularly in lipid-rich or thermally processed products. Matrix components such as polyphenols and metal ions may alter protein solubility and epitope exposure, but advances in ultrasound- and microwave-assisted extraction mitigate these effects and enhance recovery [21].

Integration of systems biology and bioinformatics into MS-based allergenomics has broadened understanding of allergen structure-function relationships, enabling identification of clinically relevant peptide biomarkers and post-translational modifications [22]. Overall, LC-MS/MS has become the reference method for confirmatory allergen analysis and a key tool for validated analytical verification under ISO 22002-100:2025 requirements. Table 2 provides a comparative overview of analytical methods for allergen detection, summarizing their principles, main advantages, and limitations. It illustrates the complementary role of chromatographic methods alongside immunochemical and molecular assays in achieving reliable allergen quantification.

Table 2. Comparison of analytical techniques used for food allergen detection and quantification.

Method	Principle	Performance Characteristics	Limitations	Typical Use
ELISA	Specific antigen–antibody binding	High sensitivity, good reproducibility, easy to perform	Possible cross-reactivity, limited multiplexing	Official controls, HACCP verification, compliance testing
LFIA	Immunochromatographic reaction on membrane strips	Rapid results, portable format, suitable for on-site use	Mainly qualitative or semi-quantitative	On-site regulatory screening, hygiene and labelling verification
PCR	Amplification of species-specific DNA sequences	High specificity for source identification	Does not directly measure allergenic proteins	Traceability and compliance with EU allergen labelling requirements
LC-MS/MS	Detection of allergen-derived peptides after digestion	High specificity and sensitivity, multi-target capability	High instrumentation cost, complex sample preparation	Confirmatory analysis in official control and reference laboratories
MALDI-MS	Direct ionization and mass analysis of peptides	Fast analysis, low solvent consumption	Limited quantitative performance	Pre-screening in regulatory monitoring programmes
Biochip-based biosensors (optical or electrochemical)	Biological recognition combined with signal transduction	Miniaturization potential, integration into portable systems	Requires frequent calibration and validation	Supportive tools for regulatory surveillance and risk assessment

2.4. Validation, Standardization, and Harmonization of Chromatographic Methods for Allergen Analysis

Reliable allergen quantification depends on validated chromatographic methods that consistently perform across different food matrices and processing conditions. Validation confirms that the method is fit for purpose by assessing parameters such as specificity, recovery, precision (repeatability and reproducibility), accuracy, linearity, and limits of detection and quantification. Matrix-related factors (including extraction efficiency, peptide stability, and signal interference) must also be evaluated to ensure reliable measurement and traceable results. Standardization initiatives focus on defining common performance criteria, consistent reporting units, and calibration. Collaborative inter-laboratory studies coordinated by organizations such as AOAC, CEN/TC 275/WG12, and iFAAM aim to harmonize chromatographic and immunochemical methods, improving data comparability and ensuring that analytical performance aligns with clinically relevant allergen thresholds [23].

A major challenge in allergen analysis is the limited availability of well-characterized incurred certified reference materials (CRMs) for key allergens and food matrices. Existing CRMs, such as those developed for milk, egg, almond, hazelnut, and walnut, provide reli-

able materials for method validation, matrix-matched calibration, and recovery correction. They enable laboratories to assess inter-laboratory comparability and verify quantitative performance across analytical methods. However, the current CRMs cover only a small number of allergens and mainly simple or lightly processed matrices, which limits their applicability to more complex or heat-treated food [24]. Expanding CRM availability and implementing harmonized validation protocols are essential to ensure that chromatographic allergen measurements are scientifically reliable and suitable for evidence-based verification under ISO 22002-100:2025.

2.5. Targeted Chromatographic Applications for Individual Allergens

Targeted chromatographic applications have become essential tools for the accurate detection and quantification of specific food allergens. By focusing on characteristic peptides or proteins within defined food matrices, they achieve high selectivity and quantitative accuracy, even in thermally processed or complex food. Their application across individual allergen groups, including milk, egg, cereals, nuts, sesame, soy, fish, and mustard, demonstrates their importance for confirmatory allergen verification and compliance with food safety requirements.

For **milk allergens** (β-lactoglobulin, caseins), targeted LC-MS/MS enables reliable determination of heat-stable proteins and modified peptides in a wide range of processed matrices [25–30]. For **cereal allergens and gluten**, chromatographic and mass-spectrometric determination supports species-level discrimination and quantification of gliadins and glutenins below regulatory thresholds, including in thermally processed foods [31–33]. Analysis of **sesame allergens** benefits from targeting stable 2S, 7S, and 11S globulins, enabling accurate determination despite strong resistance to heat and digestion [34–36]. For **soy allergens**, peptide-based MS methods allow sensitive quantification of Gly m proteins in matrices where immunochemical assays often face limitations [37–40]. Chromatographic determination of **egg allergens** supports simultaneous identification of Gal d proteins and reliable assessment of processing effects on allergenicity [41–44]. Detection of **fish allergens**, especially parvalbumin, is strengthened by MRM-based LC-MS/MS, which minimizes interference arising from protein homology across fish species [45,46]. For **mustard allergens**, LC-MS/MS provides confirmatory detection of Sin a 1 in processed or acidic foods where ELISA and PCR methods may be affected by matrix interference [47,48]. Finally, targeted chromatographic applications for **peanut and tree nut allergens** allow sensitive determination of heat-stable proteins such as Ara h 3 and Jug r isoforms, supporting product validation and labelling accuracy [49,50].

A consolidated overview of all targeted chromatographic applications, including analytical techniques, matrices, and validation characteristics, is presented in Table 3. These studies highlight the selectivity and quantitative capability of peptide-based chromatographic determination and support confirmatory allergen verification in alignment with ISO 22002-100:2025 requirements. As individual allergen methods continue to advance in sensitivity and selectivity, analytical practice has increasingly shifted toward approaches capable of detecting multiple allergens simultaneously.

Table 3. Summary of targeted chromatographic methods for individual food allergens, including characteristic analytes, analytical techniques, food matrices, and key validation elements.

Ref.	Allergen(s)/Target Proteins	Matrix	Analytical Technique(s)	Sample Preparation Conditions	Key Findings/ Validation Notes
[25]	Milk proteins: Caseins, β-lactoglobulin, α-lactalbumin	Processed foods	LC-MS/MS	General extraction and peptide-level detection	Demonstrates need for highly sensitive and validated methods due to persistence of milk proteins after processing.
[26]	Milk peptides	Heat-treated and baked products	LC-MS/MS with immobilized trypsin	Rapid immobilized trypsin digestion enabling high peptide yield	High-throughput quantification, suitable for thermally processed matrices with efficient peptide release.
[27]	Hidden milk allergens	Multi-ingredient foods (e.g., meat products)	LC-ESI-HRMS	Standard protein extraction and HRMS profiling	Higher sensitivity and specificity than immunoassays, robust identification of hidden allergens in complex matrices.
[28]	β-lactoglobulin, αS1-casein, κ-casein, α-lactalbumin	Various food matrices	Multiplex LC-MS	Single-run multi-peptide quantification	Enables simultaneous determination of major milk allergens, high accuracy with reduced analysis time.
[29]	Milk proteins	Fruit-based or composite snacks	LC-MS/MS	Optimized digestion and solid-phase extraction and matrix-matched calibration	Highlights matrix interferences (polyphenols, pectins) reducing protein recovery, importance of tailored extraction strategies.
[30]	Milk proteins	Processed foods	LC-MS/MS	Optimized digestion and SPE and matrix-matched calibration	Detects milk < 5 mg/kg with high repeatability and recovery, suitable for confirmatory analysis and PRPs verification.
[31]	Gluten proteins (gliadins, glutenins)	Various cereal-based foods	General LC-MS/MS approach	Standard extraction and peptide profiling	Underscores need for sensitive/validated methods for gluten-free compliance due to very low reaction thresholds.
[32]	Gluten peptides, species differentiation (wheat, barley, rye)	Complex and processed cereal foods	LC-MS/MS	Robust extraction and peptide marker profiling	Capable of distinguishing cereal species, accurate quantification below the 20 mg/kg Codex gluten-free threshold.
[33]	Gluten peptides	Thermally processed foods	LC-MS/MS	Emphasis on peptide marker selection and matrix-matched calibration	Ensures reliable quantification in heat-treated matrices, highlights importance of optimized preparation and calibration to match matrix effects.

Table 3. *Cont.*

Ref.	Allergen(s)/Target Proteins	Matrix	Analytical Technique(s)	Sample Preparation Conditions	Key Findings/ Validation Notes
[34]	Gluten (qualitative/ semiquantitative)	On-site hygiene verification	TLC and colorimetric readout	Simple extraction and TLC migration	Portable screening tool with low detection limits (approximately 0.12 mg), suitable for rapid cross-contamination monitoring in industrial settings.
[35]	Sesame allergens (2S albumins, 7S vicilin-like, 11S globulins)	Various sesame-containing foods	General chromato-graphic/MS context	Standard extraction and protein/peptide detection	Highlights high heat and digestion resistance of major sesame allergens, underscores analytical challenges in processed foods.
[36]	Sesame storage proteins	Thermally processed foods	LC-MS/MS	Peptide biomarker selection for heat-stable proteins	Demonstrates difficulty of detecting sesame in processed matrices due to allergen stability, supports need for MS-based confirmatory methods.
[37]	Sesame peptide biomarkers (e.g., Ses i 1)	Diverse food matrices (including baked/fried/ boiled)	LC-MS/MS (targeted)	Extraction and optimized signature peptide selection	High specificity/sensitivity even after extensive processing, improved inter-laboratory consistency using robust peptide markers.
[38]	Soy allergens (general)	—	—	—	Background reference describing the regulatory importance of soy allergens and the need for accurate detection due to widespread use of soy-derived ingredients.
[39]	Soy proteins: Gly m 4, Gly m 5 (β-conglycinin), Gly m 6 (glycinin)	Various soy-containing foods	LC-MS/MS (targeted)	Protein extraction and peptide-level detection	Demonstrates high processing stability of key soy allergens, supports the need for MS-based confirmation where immunoassays show reduced performance.
[40]	Total soy protein (via stable isotope-labelled peptides)	Bakery and meat model matrices	Targeted LC-MS/MS	Enzymatic digestion and isotope-labelled internal standards	Enables precise quantification at very low levels (approximately 10 ppm), robust across isolates, concentrates, roasted and hydrolysed soy flours.
[41]	Soy residues in plant-based beverages and dairy alternatives	Complex matrices with denatured proteins	LC-MS/MS	Optimized enzymatic digestion and external calibration	Reliable detection in matrices where ELISA underestimates due to denaturation, supports PRP verification and regulatory compliance.

Table 3. *Cont.*

Ref.	Allergen(s)/Target Proteins	Matrix	Analytical Technique(s)	Sample Preparation Conditions	Key Findings/ Validation Notes
[42]	Egg allergens (Gal d 1–4: ovomucoid, ovalbumin, ovotransferrin, lysozyme)	Various food products	—	—	Background reference describing major egg allergens, their heat/enzymatic stability, and analytical challenges in processed/composite foods.
[43]	Gal d 1–4 (major egg white proteins)	Egg-containing foods, purified fractions	Ion-exchange chromatography	Protein extraction and IEC purification	Enables purification of four major allergens in a single step with high recovery and preserved immunological activity, supports allergenicity evaluation and method validation.
[44]	Gal d 1–4 (major egg white proteins)	Raw and baked egg matrices	LC-MS/MS	Extraction and optimized digestion and calibration	Demonstrates influence of thermal processing on allergen detectability, quantification requires matrix-specific calibration for accuracy.
[45]	Gal d 1–4 (major egg white proteins)	Fresh and heat-treated egg-containing foods	LC-MS/MS	Targeted peptide quantification	Provides trace-level detection (<10 mg/kg) with high reproducibility, suitable for analytical verification under ISO 22002-100:2025.
[46]	Fish allergens (parvalbumin)	Various fish-containing foods	—	—	Background reference describing parvalbumin as the main fish allergen, its high stability, and the analytical difficulty posed by species homology and processing.
[47]	Fish allergens (β-parvalbumin)	Fish-based products (including processed foods)	LC-MS/MS (MRM)	Tryptic digestion and mild denaturants to improve peptide recovery	Achieves sensitive quantification (<0.1 μg/g) with high precision and linearity, robust method suitable for routine allergen monitoring and verification of labelling accuracy.
[48]	Mustard allergens (Sin a 1)	Complex processed foods (sauces, bakery products)	LC-MS/MS (SRM)	Extraction and targeted peptide quantification	Highly specific and sensitive for Sin a 1 in processed matrices, detects sub-ppm levels (approximately 0.25 ppm), suitable for allergen traceability and ISO 22002-100:2025 verification.

Table 3. *Cont.*

Ref.	Allergen(s)/Target Proteins	Matrix	Analytical Technique(s)	Sample Preparation Conditions	Key Findings/ Validation Notes
[49]	Mustard allergens	Thermally treated or acidic foods	ELISA and PCR-based assays	Standard immunochemical/ molecular techniques	Describes limitations of ELISA/PCR due to matrix interference and thermal effects, supporting the need for confirmatory LC-MS/MS methods in mustard detection.
[50]	Peanut allergens (Ara h 3)	Processed nut-containing foods	DAS-ELISA and LFIA	Standard immunochemical extraction	Achieves highly sensitive detection (39 ng/mL), maintains precision and specificity after heat–moisture treatment, useful for monitoring processing-induced changes in antigenicity.

2.6. Simultaneous Chromatographic Determination of Multiple Food Allergens

Multiplex chromatographic approaches have become increasingly important for comprehensive allergen determination, as they allow several allergenic proteins to be measured within a single analytical sequence. By integrating common extraction and digestion steps with chromatographic separation and peptide-based quantification, these methods reduce analytical effort and improve consistency relative to single-target assays. When supported by appropriate validation, multi-allergen procedures offer robust trace-level detection across a wide range of food matrices and provide the analytical assurance required for verification of allergen control measures under ISO 22002-100:2025.

Applications of simultaneous LC-MS/MS determination demonstrated accurate quantification of combinations such as milk and egg allergens and later extended to nut proteins through peptide-based approaches targeting characteristic globulins [51,52]. Subsequent studies expanded multiplex analysis to priority allergens including milk, egg, and peanut, where optimized extraction, enzymatic digestion, and internal standardisation improved reproducibility and agreement with immunochemical methods [53–55]. Additional studies incorporating micro-HPLC, LC-MS, ELISA cross-comparison, and complementary optical techniques enabled concurrent detection of allergens such as casein, soy, and gluten in baked or composite foods [56–58]. More recent LC-MS/MS and LC-HRMS developments have achieved simultaneous determination of four or more allergenic proteins, including milk, egg, soy, peanut, crustaceans, and tree nuts, across processed and multi-ingredient matrices, with strong linearity, consistent recoveries, and inter-laboratory reproducibility [59–62]. Foundational studies further established the feasibility of detecting up to seven allergens within a single chromatographic run, while newer HRMS applications extend coverage to gluten and seafood allergens with high analytical accuracy [63–66]. A consolidated overview of simultaneous chromatographic applications (including analyte coverage, analytical techniques, matrices, and validation characteristics) is presented in Table 4.

Table 4. Summary of simultaneous (multiplex) chromatographic methods for the determination of multiple food allergens, including analyte coverage, analytical approaches, representative matrices, and validation characteristics.

Ref.	Allergen(s)/ Target Proteins	Matrix	Analytical Technique(s)	Sample Preparation Conditions	Key Findings/ Validation Notes
[51]	Milk, egg allergens	Composite and processed foods	LC-MS/MS with isotope-labelled internal standards	Extraction and unified digestion protocol	Accurate quantification of both allergen classes, detection < 1 mg/kg, excellent repeatability, demonstrates robust dual-allergen determination.
[52]	Nut allergens (walnut, almond proteins)	Diverse food matrices	LC-MS/MS (targeted)	Extraction and selection of 2S, 7S, 11S globulin peptides	High linearity (>0.999) and recoveries > 90%, demonstrates reliable simultaneous nut allergen identification and quantification.
[53]	Milk, egg, peanut allergens	Thermally processed foods	LC-MS/MS	Optimized extraction, enzymatic digestion, targeted peptide monitoring	LOQ ~5 mg/kg, enables simultaneous quantification of three priority allergens in complex heat-treated matrices.
[54]	Multiple allergens (milk, egg, peanut)	Processed foods	LC-MS/MS with matrix-matched calibration	Extraction, digestion, calibrants using allergen ingredients, isotope-labelled internal standards	Matrix-matched calibration significantly improves accuracy, strong reproducibility across matrices.
[55]	Multiple allergens (milk, egg, peanut)	Processed foods	LC-MS/MS and ELISA comparison	Standard extraction, peptide-based quantification	Good correlation with ELISA, suitable for verifying thermal processing effects and supporting allergen-labelling compliance.
[56]	Milk, egg, soy allergens	Baked food matrices	Micro-HPLC and dual-cell linear ion trap MS	Sonication-assisted extraction, tryptic digestion	Highly sensitive detection in baked matrices, LOD 0.1–2 µg/g, enhanced signal intensity due to optimized sonication, suitable for multi-allergen determination in thermally processed foods.
[57]	Casein, soy, gluten	Incurred cookies (baked products)	LC-MS and ELISA and multiplex flow cytometry	Extraction, digestion, multimethod comparison	Consistent detection across techniques, recoveries > 80% after baking, supports robustness of combined LC-MS with immunochemical tools for processed matrices.
[58]	Various food allergens including seafood proteins	Mixed food matrices	LC-MS/MS (reviewed approaches)	—	Review highlighting the expansion of multi-allergen LC-MS/MS strategies to additional allergenic protein groups (e.g., seafood), emphasizes potential for comprehensive allergen profiling.

Table 4. *Cont.*

Ref.	Allergen(s)/ Target Proteins	Matrix	Analytical Technique(s)	Sample Preparation Conditions	Key Findings/ Validation Notes
[59]	Milk, egg, soy, peanut allergens	Processed foods (cookies, sauces, chocolate)	UHPLC-MS/MS	Extraction, enzymatic digestion, targeted peptide quantification	LOQ < 5 mg/kg, high linearity in incurred samples, reliable multi-allergen detection across different processed foods.
[60]	Milk, egg, soy, crustacean peptides	Fish and meat products	LC-HRMS	Extraction, digestion, high-resolution identification	Recoveries 80–95%, strong agreement with ELISA screening, robust simultaneous detection across mixed matrices.
[61]	Milk, egg, peanut, hazelnut allergens	Mixed food matrices	Quantitative LC-MS	Standard extraction, enzymatic digestion, use of internal standards	Demonstrated strong inter-laboratory reproducibility, suitable for harmonized multi-allergen verification protocols.
[62]	Milk, egg, peanut, hazelnut allergens	Various processed foods	LC-MS/MS	Generic extraction, digestion	High consistency across runs, confirms applicability of LC-MS/MS for harmonized multi-allergen determination.
[63]	Milk, egg, soy, hazelnut, peanut, walnut, almond	Incurred bread samples	LC-MS/MS	Unified extraction, digestion, single-run targeted MS	First demonstration of simultaneous detection of 7 allergens in one run, quantification range 10–1000 µg/g, feasibility of multiplex allergen LC-MS/MS established.
[64]	Multiple allergens	Processed foods	LC-MS/MS (enhanced conditions)	Optimized extraction, digestion, refined chromatographic setup	Improved peptide recovery vs. Ref. [60] and throughput, better performance in processed matrices, advances in multiplex method robustness.
[65]	Multiple allergens including gluten and crustacean proteins	Diverse processed food matrices	LC-HRMS	High-resolution identification, targeted quantification	Accurate simultaneous quantification, recoveries 70–110%, extended scope beyond classical priority allergens.
[66]	Multiple allergens including gluten and crustaceans	Mixed food products	LC-HRMS	Standardized extraction, HRMS acquisition	High accuracy across complex matrices, confirms HRMS suitability for expanded multi-allergen suites, aligns with needs for broad-spectrum verification.

These analytical methods provide the sensitivity, selectivity, and traceability required for the verification activities specified in ISO 22002-100:2025, ensuring that allergen control measures can be confirmed with validated methods. Their capacity to deliver harmonized quantitative data for several allergenic proteins within a single chromatographic analysis strengthens risk-based allergen assessment, supporting objective evaluation of cross-contact and enabling scientifically justified decisions regarding precautionary and mandatory allergen labelling.

3. Chromatographic Approaches for Food Fraud and Authenticity Control

3.1. Regulatory and Management System Standards Requirements for Food Fraud Control

Food fraud and authenticity assurance are shaped by a defined set of regulatory and management system requirements in the European Union (EU) and internationally. Regulation (EC) No 178/2002 establishes consumer protection, non-misleading practices, and traceability as core legislative obligations, forming the basis for authenticity across the supply chain [67]. Regulation (EU) 2017/625 further requires competent authorities to implement risk-based controls addressing integrity, fair trade practices, and potential consumer deception related to origin, composition, or production method [68]. Together, these regulations position authenticity as a legal requirement and support the use of analytical testing, including chromatographic approaches, for compliance verification.

ISO 22000:2018 incorporates risk-based thinking at both organizational and operational levels, requiring food businesses to identify and manage risks that may affect the performance of the food safety management system [3]. Although the standard primarily targets food safety hazards, its structure permits the integration of food fraud–related vulnerabilities through requirements on traceability, control of externally provided materials, and verification activities. The newly published ISO 22002-100:2025 further strengthens this approach by introducing a dedicated Food Fraud Mitigation Plan, systematic vulnerability assessment, reinforced supplier approval and traceability expectations, and explicit requirements for analytical verification of authenticity [1]. As the first cross-sectoral PRP to address food fraud in a structured manner, it provides a harmonized basis for linking supply-chain controls with laboratory-based authenticity verification.

Core definitions, typologies, and mechanisms of food fraud are well established in the scientific literature, providing the basis for identifying fraud types and their implications for food chain integrity [69]. Foundational analyses further document recurring commodity-specific vulnerabilities, characteristic adulteration patterns, and the enduring public health and economic impacts that shape current prevention requirements [70]. Recent evidence reinforces the need for structured, vulnerability-based approaches to food fraud mitigation. Consumer-oriented analyses show that effective countermeasures depend on proactive, data-driven identification of fraud risks and on transparency and alignment between regulatory and industry controls [71]. Holistic vulnerability-assessment approaches incorporate multiple indicators, including product characteristics, historical fraud patterns, and supply-chain complexity, supporting the systematic assessment processes now embedded in ISO 22002-100:2025 [72]. EU agri-food fraud surveillance data demonstrate the practical implementation of Regulation (EU) 2017/625, with recurring fraud patterns detected through market controls, border inspections, and operators' own checks [73]. Long-term global fraud records likewise show persistent vulnerabilities in high-value commodities such as milk, olive oil, honey, meat products, and spices, underscoring the need for structured mitigation plans within management system standards [74].

These regulatory and standards-based requirements create the prerequisite conditions for effective food fraud mitigation, which increasingly relies on structured prevention models and analytical verification tools.

3.2. Mitigation Strategies for Food Fraud Prevention

Food fraud mitigation has shifted from reactive incident response to structured, prevention-oriented strategies based on risk and vulnerability assessment. Core principles emphasize an iterative cycle of vulnerability identification, risk prioritization, targeted control implementation, and continuous verification [75]. Effective prevention requires coordinated governance, integration of relevant intelligence, and focused controls across procurement, specifications, and supply-chain management. Reviews of major mitigation

guides consistently highlight four prevention elements, reducing motivation, limiting opportunities, improving detection, and reinforcing deterrence, operationalized through supplier controls, specification requirements, risk-based analytical testing, and market-surveillance activities [76].

Recent analyses of food fraud mitigation guidance documents underscore the importance of consistent terminology and clearly structured guidance, as definitional clarity strengthens both vulnerability assessment and the selection of proportionate preventive measures [77]. Complementary research on anticounterfeiting solutions highlights the expanding role of technological tools, such as overt and covert security features, tamper-evident packaging, digital identifiers, and enhanced traceability systems, in supporting product authentication and reinforcing supply-chain integrity, especially for high-value commodities [78]. Together, these developments complement management system controls and contribute to more robust, prevention-focused fraud-mitigation schemes.

Additional evidence highlights contextual and operational factors that shape the effectiveness of food fraud mitigation. Studies note that structural constraints, such as limited analytical capacity, insufficient data, and resource shortages, can hinder implementation, particularly in low-income settings, underscoring the need for scalable controls and improved information sharing across the supply chain [79]. At the same time, comparative evaluations of Vulnerability Assessment and Critical Control Points (VACCP) approaches show that the lack of a harmonized methodology produces variability in how vulnerabilities are assessed and controls prioritized [80]. Greater alignment on assessment steps, data inputs, and prioritization criteria is increasingly recognized as essential for ensuring risk-based, consistent, and globally comparable mitigation practices.

GFSI has played a critical role in formalizing industry-wide expectations for food fraud prevention, progressively moving from general authenticity requirements to clearly defined obligations for vulnerability assessment, documented mitigation plans, and evidence-based verification. Over the past decade, GFSI-recognized certification schemes have increasingly required structured, risk-driven controls that integrate supplier assurance, specification management, incident monitoring, and targeted analytical verification [81]. These developments have influenced the structure of ISO 22000-based systems and have contributed to the explicit food fraud requirements included in ISO 22002-100:2025.

In parallel, food defence has become a key complementary component of current management system standards. Through structured Threat Assessment and Critical Control Points (TACCP), organizations are expected to assess and manage intentional, malicious contamination threats, whether arising from disgruntled employees, extortion attempts, ideological actors, cyber-attacks, or broader terrorism-related risks. Effective food defence plans rely on clear threat characterization, identification of vulnerable points, proportionate security measures, and regular verification to ensure ongoing effectiveness [82]. Although not directly connected to analytical chromatography, food defence reflects the broader move toward integrated integrity systems in which food safety, fraud prevention, and threat management function as interconnected elements of supply-chain resilience.

Recent evaluations highlight that analytical testing serves as a targeted verification tool within food fraud mitigation rather than a primary detection mechanism. Most fraud events are not identified through routine laboratory testing due to sampling limitations, cost constraints, and the variable nature of adulteration practices. Analytical methods are therefore applied selectively and require fit-for-purpose sampling plans, validated methods, and appropriate detection capabilities [83]. In this context, chromatographic and mass-spectrometric methods operate as risk-based confirmatory tools that strengthen preventive controls and support the validation of authenticity claims.

3.3. Chromatographic Strategies for Food Fraud Detection and Authenticity Assessment

Chromatography is widely used in food fraud detection because it enables selective separation and reliable identification of compounds linked to authenticity. It supports targeted analysis of known markers or adulterants and non-targeted profiling, where LC-MS or GC-MS data are compared with authenticated reference samples. Targeted approaches confirm specific fraud scenarios, whereas non-targeted methods detect unexpected compositional deviations. The integration of high-resolution mass spectrometry and chemometric data analysis further improves sample classification and strengthens authenticity assessment [84].

Over the past five years, chromatographic techniques have been increasingly complemented by modern data-analysis tools that enhance the interpretation of complex analytical results. Strategies combining LC-MS and GC-MS with advanced computational models allow more efficient detection of compositional deviations and subtle indications of adulteration. Improvements in automated processing, faster separation procedures, and simplified evaluation steps have reduced analysis time without compromising analytical reliability, supporting broader application across diverse food categories [85].

Recent reviews highlight the rapid expansion of chromatographic and MS-based techniques used for authenticity assessment. Systematic evaluations report growing integration of LC-MS, GC-MS, isotope ratio methods, and other hyphenated approaches with chemometric modelling to improve the detection of adulteration, origin misrepresentation, and species substitution [86]. Beyond conventional approaches, multi-modal analytical methods that combine chromatographic separation with complementary spectral or molecular techniques yield more robust compositional information for authenticity verification [87].

Further studies show that combining chromatographic data with spectroscopic or DNA-based techniques enhances detection capability, particularly in complex matrices. Multimodal chemometric fusion models increase classification accuracy and reduce false detections, while advanced statistical classification of chromatographic data enables rapid screening and supports the early recognition of abnormal compositional patterns indicative of adulteration [88,89].

Despite these advances, a notable gap remains between academic developments and methods routinely implemented in industry. Comparative assessments reveal that although high-resolution untargeted LC-MS and GC-MS approaches can detect subtle deviations, industrial laboratories often rely on simpler targeted methods due to cost, throughput, and expertise constraints [90]. At the same time, there is increasing emphasis on rapid screening solutions, such as LC-MS-based triacylglycerol profiling, streamlined MS evaluations, and multivariate classification, to support faster decision-making and reduce reliance on labour-intensive confirmatory testing. Collectively, these trends point to the need for analytically robust yet operationally feasible chromatographic tools for routine authenticity verification [91].

Volatile-metabolome analysis represents an important complementary area, with GC-MS enabling sensitive characterization of volatile organic compounds (VOC) reflective of raw materials, processing conditions, and storage history. Distinct VOC compositions can act as authenticity markers or indicators of tampering, while solvent-free sampling approaches such as solid-phase micro-extraction facilitate detection of trace-level components relevant to adulteration. Untargeted VOC profiling has demonstrated strong discriminatory ability across multiple food categories, underscoring its value in authenticity assessment [92].

High-performance thin-layer chromatography coupled with mass spectrometry (HPTLC-MS) has also emerged as a practical supplementary technique. It enables rapid separation of key constituents and direct MS verification of selected zones, offering an

efficient screening option for detecting adulteration in products with characteristic chemical markers [93].

Chromatographic and MS-based methodologies now constitute a technically mature tool for authenticity assessment. Their practical relevance becomes most evident when these methods are applied to real food matrices, where fraud patterns and compositional variability determine analytical performance.

3.4. Applications in Food Analysis

3.4.1. Overview Across Major Food Categories

Chromatographic techniques demonstrate their full analytical value when adapted to specific food matrices, where compositional complexity, characteristic adulteration pathways, and matrix-dependent interferences shape method selection and detection capability. Across major food categories, targeted and non-targeted LC-MS and GC-MS approaches supported by chemometrics are widely applied to verify identity, detect substitution, and strengthen traceability within risk-based control systems.

Organic foods. LC-MS/GC-MS compositional profiles combined with multivariate modelling reliably distinguish organic from conventional products and support verification of cultivation and origin claims, while advanced chemometric tools enhance detection of irregularities in the organic supply chain [94,95].

Beverages. Chromatographic methods and isotope ratio analysis enable authentication of wine, spirits, and beer by verifying botanical origin, geographic provenance, processing practices, and detecting dilution or exogenous components. Integrated approaches combining chromatography, spectroscopy, and stable isotopes address complex fraud mechanisms. Non-alcoholic beverages remain vulnerable to counterfeiting and adulteration, requiring targeted analytical testing within robust control systems [96–99].

Plant-origin foods. LC-MS, GC-MS, and isotope ratio methods support discrimination of origin, cultivar, and botanical identity in coffee, spices, herbs, grains, and cereals. Chromatographic, spectroscopic, and DNA-based methods improve detection of common adulterants, fillers, and misrepresentation of quality or geographic origin [100–103].

Oils and fats. Extra virgin olive oil (EVOO) authenticity assessment relies on chromatographic compositional characteristics, targeted markers, and MS-based profiling to detect blending and verify origin. Similar chromatographic and spectroscopic approaches are applied to other specialty oils, reflecting diverse adulteration risks across the category [104,105].

Honey. Due to compositional variability and globalized sourcing, honey authentication relies on combined chromatographic, isotopic, and spectroscopic techniques, including LC-IRMS, HRMS profiling, NMR fingerprinting, and targeted/non-targeted LC-MS and GC-MS approaches, to detect sugar-syrup dilution, verify botanical and geographical origin, and evaluate potential losses of functional bioactive constituents [106–108].

Meat, poultry, and seafood. High fraud susceptibility, through species substitution, dilution, or mislabelling, necessitates DNA-based identification supported by chromatographic, spectroscopic, and elemental analysis to verify species integrity and production claims [109,110].

Milk and dairy products. LC-MS and related chromatographic methods are central for detecting water dilution, foreign proteins or fats, species mislabelling, and cheese adulteration. Combined chromatographic, spectroscopic, and MS-based approaches, often chemometric-assisted, strengthen authentication of milk, dairy ingredients, and PDO/PGI cheeses [111–115].

For **PDO/PGI products**, stable isotope profiles can support verification of geographical and, in some cases, production-origin claims. From a chromatographic perspective,

compound-specific isotope analysis (CSIA) using GC-C-IRMS and/or LC-IRMS resolves isotopic information at the compound level, which is useful in complex matrices when markers such as sugars, amino acids, fatty acids, or selected aroma constituents are targeted. Bulk IRMS may still be used for screening purposes, while chromatographic CSIA is better suited to situations where confirmatory, compound-resolved evidence is required [116–118].

Recent reviews on synthetic **food colourant** analysis highlight LC-MS/MS (typically triple-quadrupole methods operated in selected reaction monitoring mode) as a confirmatory approach for targeted determination of both permitted dyes (compliance control) and undeclared/unauthorized synthetic colourants, supported by matrix-adapted sample preparation (e.g., solid-phase extraction, QuEChERS, microextraction) to mitigate interferences in complex foods [119].

To provide a structured synthesis aligned with the verification requirements introduced in ISO 22002-100:2025, Table 5 summarizes key fraud mechanisms across major food categories together with the chromatographic and complementary analytical techniques most frequently applied for authenticity assessment.

Table 5. Overview of chromatographic and related analytical techniques applied to food authenticity verification across key food categories.

Refs.	Food Category	Typical Fraud Mechanisms	Chromatographic/Analytical Approaches
[94,95]	Organic foods	Mislabelling of cultivation practices, substitution with conventional products	LC-MS/GC-MS fingerprints, multivariate chemometrics, compositional pattern recognition
[96–98]	Beverages (alcoholic)	Dilution, addition of exogenous alcohol, misrepresentation of origin/vintage	LC-MS/GC-MS profiling, trace-level markers, multi-isotope IRMS, combined chromatography–spectroscopy techniques
[99]	Beverages (non-alcoholic)	Counterfeiting, juice adulteration, undeclared additives	Targeted chromatographic assays, quality system-based verification, complementary spectroscopic screening
[100]	Plant-origin foods (general)	Substitution, dilution, misrepresentation of botanical/geographical origin	LC-MS/GC-MS, isotope ratio analysis, chemometric classification
[101,102]	Coffee, spices, herbs	Addition of fillers, undeclared plant materials, colorants, flavour enhancers	Chromatographic profiling, MS-based detection, spectroscopic markers, DNA assays
[103]	Grains and cereals	Varietal substitution, protein inflation, false origin/production claims	LC-MS/GC-MS, spectroscopic profiling, DNA assays, IRMS
[104,105]	Oils and fats	Blending with cheaper oils, misrepresentation of botanical/geographic origin	Fatty acid profiling, sterol analysis, MS-based fingerprints, chemometrics
[106–108]	Honey	Sugar-syrup dilution, false botanical or geographical origin, post-processing manipulation, loss of functional bioactive constituents	LC-IRMS, HRMS profiling, NMR fingerprinting, targeted and non-targeted LC-MS/GC-MS analysis

Table 5. *Cont.*

Refs.	Food Category	Typical Fraud Mechanisms	Chromatographic/Analytical Approaches
[109]	Meat and poultry	Species substitution, dilution, misrepresentation of production attributes	Chromatographic profiling, spectroscopic screening, DNA-based species authentication
[110]	Seafood	Species substitution, false origin/method of production, short-weighting	Molecular identification, LC-MS/GC-MS, elemental and isotope ratio markers
[111,114]	Milk	Water dilution, addition of foreign proteins/fats, false species origin	LC-MS/HPLC profiling of proteins and peptides, detection of nitrogen-rich adulterants
[112,115]	Cheese	Substitution with cheaper milk types, dilution, false PDO/PGI claims	LC-MS profiling, MIR/NIR/Raman spectroscopy, MS-assisted screening with chemometrics

3.4.2. Analytical Evidence from Individual Studies Across Food Matrices

Chromatographic, isotopic, and mass-spectrometric methods have been applied across major food matrices to quantify compositional markers, identify adulterants, and support origin and species verification. The following examples summarize representative applications based on targeted analytical parameters and experimentally validated chemical characteristics.

Beverages. LC-MS metabolomics has been used to distinguish grape varieties and production regions through quantitative metabolic profiles, while combined isotopic and chromatographic analyses detect dilution, C4-sugar enrichment, and synthetic additives in wine. In fruit juices, HPLC carbohydrate profiling and GC-MS volatile analysis enable detection of syrup addition and non-declared juice substitution, with multivariate analysis providing clear group separation [120–124].

Fruits and Vegetables. HS-SPME-GC-MS volatile profiling differentiates citrus cultivars and storage conditions, GC-C-IRMS separates natural from synthetic aroma constituents in apples, and chiral GC-MS identifies maturity and logistic pathways in pineapple. GC-MS/LC-MS analysis of grape volatiles and glycosides supports cultivar authentication, while chromatographic and colloidal profiles enable classification of processed tomato products and combined compositional parameters support discrimination of cherry and carrot geographical origin, with HPLC-DAD also supporting verification of natural colourant claims in relevant processed foods [125–132].

Cereal and Grain Products. Targeted lipidomics quantifies adulterated rice admixtures, untargeted LC-MS metabolomics separates organic from conventional rice, and proteomic peptide markers distinguish wheat, rye, and spelt in cereal products. LC-based phenolic profiling further differentiates organic and conventional wheat across successive harvests [133–136].

Vegetable and Seed Oils. NIR spectroscopy, TGA-GC-MS, and GC-MS volatile profiling detect undeclared seed-oil addition in camellia, olive, and sesame oils, while combined GC-MS analysis and hyperspectral data enable discrimination of adulterants in safflower oil. LC-MS glyceride analysis characterizes TAG/DAG distributions in fruit seed oils to verify botanical identity and identify compositional deviations indicative of adulteration [137–143].

Honey. HPLC-UV carbohydrate profiling quantifies C3/C4 syrup additions, HPLC organic-acid analysis differentiates monofloral honeys, and multi-wavelength HPLC-UV fingerprinting separates honeys of different geographical origin. These chromatographic approaches detect adulteration and origin mislabelling with higher resolution than routine physicochemical testing [107,108,144–146].

Meat and Fish. LC-MS/MS proteomics identifies species through proteotypic peptides in raw and processed meats, while targeted peptide markers discriminate bovine from non-bovine matrices. In fish, LC-HRMS profiling of muscle proteins provides species-specific peptide markers enabling discrimination of closely related species [147–149].

Milk and Dairy Products. SFC-Q-TOF-MS and RP-HPLC TAG profiling differentiate milk from multiple animal species and detect non-milk fat addition, while LC-MS/MS peptide profiling and MALDI-TOF-MS protein spectral characteristics authenticate cheeses and reveal undeclared milk sources. UHPLC-ELSD/UV chromatographic analysis enables detection of fat adulteration in butter, while RP-HPLC peptide profiling combined with MIR spectroscopy supports classification of cheeses according to geographical origin [150–155].

Table 6 summarizes representative studies across major food categories in alignment with the verification requirements introduced in ISO 22002-100:2025, with emphasis on documented analytical evidence, multi-marker validation, and matrix-specific vulnerability assessment. The table consolidates key fraud mechanisms, chromatographic and MS-based analytical approaches, diagnostic markers, and reported performance characteristics, providing a comparative overview of method performance under practical testing conditions.

Table 6. Representative chromatographic and MS-based applications for food fraud and authenticity assessment across major food matrices.

Ref.	Food Category	Food Matrix	Fraud/Authenticity Objective	Analytical Technique(s)	Target Analytes/ Diagnostic Markers	Reported Analytical Performance
[120]	Beverages	White wine	Varietal and geographical origin	LC-MS metabolomics	metabolic profiles	Clear discrimination of Greek varieties, robust PCA/PLS-DA separation
[121]	Beverages	Red wine	Dilution, C4 sugar chaptalization, synthetic additives	Stable isotope ratio analysis ($\delta^{13}C/\delta^{18}O$) and LC/GC compositional profiling	isotopic ratios, HMF, sweeteners, anthocyanins	Differentiation of authentic/ suspicious/adulterated samples
[122]	Beverages	Wine and fruit juices	Dilution, syrup addition, substitution, mislabelling	LC/GC profiling and IRMS and chemometrics	multi-marker chemical signatures	Review evidence supporting multi-approach strategies
[123]	Beverages	Apple juice concentrate	Syrup adulteration	HPLC-RID and chemometrics	glucose/fructose ratio, maltose, sorbitol shifts	Detection $\geq$ 10% adulteration
[124]	Beverages	Citrus juices	Mandarin overblending	HS-SPME-GC-MS VOC profiling	monoterpenes, esters, aldehydes	Clear PCA separation, detection $\geq$ 10%
[125]	Fruits	Oranges	Organic vs. conventional, cultivar/storage effects	HS-SPME-GC-MS	terpenes and esters	Distinct volatile profiles for organic fruit
[126]	Fruits	Apples	Synthetic vs. natural aroma	GC-C-IRMS	$\delta^{13}C$ of 16 marker volatiles	Accurate detection of synthetic additions
[127]	Fruits	Pineapple	Maturity stage and logistic history	Chiral HS-SPME-GC-MS	γ-/δ-lactone enantiomers	Differentiation of air- vs. sea-freighted fruit
[128]	Fruits	Grapes	Cultivar identification	GC-MS and LC-MS	monoterpenes, norisoprenoids, glycosides	Stable cultivar-associated chemical profiles

Table 6. *Cont.*

Ref.	Food Category	Food Matrix	Fraud/Authenticity Objective	Analytical Technique(s)	Target Analytes/ Diagnostic Markers	Reported Analytical Performance
[129]	Vegetables/ processed food	Tomato sauce	Origin/brand traceability	GC-FID/GC-IMS and asymmetric flow field-flow fractionation	volatile and colloidal profiles	High discrimination with chemometrics
[130]	Vegetables/ processed food	Blue confectionery	Verification of natural colourant claim	HPLC-DAD	C-phycocyanin (from *Arthrospira platensis*)	Validated method applied to commercial samples suitable for targeted verification of product claims
[131]	Vegetables	Sweet cherry	Botanical and geographical origin	HPLC sugars and GC-MS volatiles and ICP-OES minerals	multi-parameter profiles	>95% correct classification
[132]	Vegetables	Carrots	Geographical origin	Untargeted UHPLC-HRMS	region-specific metabolites	Strong origin discrimination, weaker for production mode
[133]	Cereals	Rice	Detection of adulterated admixtures	Targeted lipidomics (LC-MS based) and chemometric classification	lipid classes, molecular species	High accuracy classification of blends
[134]	Cereals	Rice	Organic vs. conventional	Untargeted LC-MS metabolomics	discriminant metabolites	Clear metabolic separation via PCA/PLS-DA
[135]	Cereals	Bread	Wheat/rye/spelt species ID	Targeted proteomics	species-unique peptides	Reliable detection of grain-type substitution
[136]	Cereals	Wheat	Organic vs. conventional	LC-based phenolic profiling	protocatechuic acid	Consistent differentiation across harvest years
[137]	Oils	Camellia oil	Seed oil adulteration	NIR spectroscopy and PLS-DA/PCA	lipid overtone absorption	>95% accuracy across mixtures
[138]	Oils	Olive oil	Soybean oil adulteration	TGA-GC-MS	thermal/volatile markers	Strong discrimination via temperature-resolved MS
[107]	Honey	Mixed botanical honeys	Syrup adulteration, floral origin	HPLC sugars, GC-MS/LC-MS volatiles, IRMS	$\delta^{13}C/\delta^{2}H$, sugar profiles	Strong adulteration detection, origin classification
[108]	Honey	Honey (general)	Functional degradation	LC-MS/LC quantification of phenolics, flavonoids, MGO	bioactive markers	Identifies heat/processing-induced loss of activity
[139]	Oils	Milk (oil adulteration)	Detection of added vegetable oils	Flash-GC and chemometrics	VOC profiles	High accuracy quantification despite matrix complexity
[140]	Essential oils	Mentha/ Ocimum	Adulteration with vegetable oils	FT-NIR and GC-MS/GC-FID and chemometrics	volatile chemical profiles	Detection at 3–30% adulteration
[141]	Seed oils	Safflower oil	Adulteration with multiple oils	GC-MS and hyperspectral imaging	sterols, fatty acids, and spectra	Fusion model improves discrimination

Table 6. *Cont.*

Ref.	Food Category	Food Matrix	Fraud/Authenticity Objective	Analytical Technique(s)	Target Analytes/ Diagnostic Markers	Reported Analytical Performance
[142]	Seed oils	Sesame oil	Undeclared seed oil blends	GC-MS and chemometric classification	volatile and lignan markers	Sensitive detection of low-level adulteration
[143]	Seed oils	Fruit seed oils	Botanical identity and adulteration	LC-MS glyceride profiling	TAG/DAG compositional profiles	Clear separation of oil types, non-authentic profiles detectable
[144]	Honey	Honey (adulteration trials)	C3/C4 syrup addition	HPLC-UV carbohydrates and PCA/PLS-DA	mono/di/ oligosaccharides	Clear grading across dilution levels
[145]	Honey	Robinia honey	Authentic vs. blended	HPLC organic acids	malic, citric, gluconic acids	Robust classification of authentic samples
[146]	Honey	Various origins	Geographical origin	Multiwavelength HPLC-UV	Spectral retention features	High accuracy regional discrimination
[147]	Meat	Multi-species meats	Species identification	LC-MS/MS proteomics (untargeted)	proteotypic peptides	Simultaneous detection in mixed/processed products
[148]	Meat	Beef	Species verification	Targeted LC-MS/MS peptides and chemometrics	bovine-specific peptides	Strong separation from non-beef matrices
[149]	Fish	Pollock vs. hake	Species identification	LC–HRMS protein-based analysis	species-specific peptide features	Accurate differentiation of closely related species
[150]	Dairy	Milk (multi-species)	Species authentication	SFC-Q-TOF-MS TAG profiling	TAG species	Clear species clustering (PCA/OPLS-DA)
[151]	Dairy	Milk/butter /cheese	Non-milk fat adulteration	RP-HPLC-RID TAG analysis	3 diagnostic TAG peaks	Detects 1–2% palm oil, strong chemometric separation
[152]	Dairy	Farmer's cheese	Authentic vs. industrial	LC-MS/MS peptides	endogenous peptide profiles	Distinguishes authentic/non-authentic samples
[153]	Dairy	PDO feta	Milk source adulteration	MALDI-TOF-MS	species-specific spectra	Rapid detection of non-sheep/goat milk
[154]	Dairy	Butter	Fat adulteration	UHPLC-ELSD and UHPLC-UV	pointwise chromatographic response profiles	Sensitive detection without markers
[155]	Dairy	Coalho cheese	Geographical origin	RP-HPLC peptides and MIR	peptide profiles and MIR spectral features	Accurate classification across 4 regions

The reported applications indicate that recently developed high-sensitivity chromatographic and MS-based techniques provide the analytical specificity required to address fraud vulnerabilities in high-risk food matrices. These approaches support industry implementation of evidence-based verification, as expected under ISO 22002-100:2025, while also strengthening regulatory-aligned controls for detecting economically motivated adulteration and managing authenticity risks.

4. Chromatographic Monitoring of Packaging Contaminants and Chemical Contamination in Food (Cleaning Agents, Disinfectants, Lubricants, and Pesticides Used for Pest Control)

Monitoring chemical contamination from packaging materials and auxiliary services in the food supply chain has become a critical requirement of prerequisite programmes (PRPs) under ISO 22002 100:2025. In line with ISO 22003-1:2022, the term auxiliary services encompasses activities such as cleaning and disinfection, pest control, maintenance, and other supporting operations that may represent indirect sources of chemical contamination in food processing environments. At the regulatory level in the European Union, general food hygiene obligations such as those set out in Regulation (EC) No 852/2004 require food business operators to establish, implement, and maintain effective cleaning, disinfection, pest control, and monitoring systems [156]. Meanwhile, specific regulations for materials intended to come into contact with food, such as Regulation (EC) No 1935/2004 and Regulation (EU) No 10/2011 on plastic materials, stipulate that migrations of constituents must not endanger human health nor lead to unacceptable changes in the food [157,158].

From an implementation perspective, guidance from industry and certification schemes further emphasizes the necessity of validated analytical verification of auxiliary chemical residues. For example, the GFSI guidance "Chemicals in Food Hygiene-Volume 2: Cleaning Agents, Sanitizers and Disinfectants in Food Businesses" provides a decision-tree approach for risk assessment of residues from cleaning and disinfection processes and highlights the need for precise detection methods, targeted sampling, systematic monitoring, and analytical verification [159].

Packaging materials, cleaning agents, disinfectants, lubricants, and pest control compounds are recognized sources of chemical contamination in food processing environments. Typical contaminants include migrant monomers and oligomers, NIASs, mineral oil hydrocarbons, quaternary ammonium compounds, peracetic acid by-products, glycols, and rodenticides. Chromatographic and spectrometric methods are therefore essential for verifying control effectiveness, ensuring regulatory compliance, and providing traceable analytical evidence in accordance with ISO 22002-100:2025.

4.1. Packaging-Related Chemical Contaminants

Food packaging materials represent a significant source of chemical contamination due to the migration of both intentionally and non-intentionally added substances into food. Migration involves monomers, oligomers, plasticizers, stabilizers, printing inks, adhesives, and varnish-derived components and is influenced by polymer structure, multilayer design, and storage conditions [160]. Contemporary food contact materials, including laminated plastics and biodegradable polymers, contain diverse classes of migrants such as impurities, degradation products, and reaction by-products, collectively classified as NIASs, which require selective chromatographic techniques for reliable detection [161]. The growing use of bio-based and biodegradable polymers adds further analytical complexity, as these materials may generate low-molecular-weight oligomers and transformation products that are not always covered by conventional toxicological datasets, increasing the need for non-target screening and high-resolution detection [162,163].

A wide range of chromatographic applications has been developed for the identification and quantification of packaging-derived contaminants, including volatile and semi-volatile migrants, oligomeric NIASs, photoinitiators, plasticizers, antioxidants, biocides, and mineral oil hydrocarbons across plastic, multilayer, paper-based, and recycled materials. These studies employ targeted and untargeted GC-MS/MS, LC-MS/MS, GC-GC/TOF-MS, HRMS, DI-SPME-GC-MS, LC-GC, and Orbitrap-based approaches, providing enhanced sensitivity for the characterization of complex migrant profiles, polymer-degradation products,

and MOSH/MOAH fractions. A consolidated overview of these analytical applications is provided in Table 7, which summarizes key compound classes, target analytes, matrices, and chromatographic methods reported across references [164–177].

These chromatographic advances directly support the verification requirements of ISO 22002-100:2025 by enabling sensitive, matrix-appropriate detection of packaging migrants, thereby strengthening evidence-based control of packaging-related chemical hazards.

4.2. Cleaning and Disinfectant Agent Residues

Cleaning agent residues represent a critical source of indirect chemical contamination in food processing environments, particularly in operations based on cleaning-in-place (CIP) systems, where alkaline, acidic, and oxidizing cleaning formulations are routinely applied to ensure hygienic conditions and the removal of organic and inorganic residues [178]. Conventional CIP performance indicators (e.g., visual inspection and conductivity-based rinse control) are insufficient to confirm the complete elimination of low-level chemical residues, supporting the need for validated, analytically based residue-monitoring strategies [178,179]. Risk-based cleaning procedures increasingly rely on systematic verification that combines optimized formulations with trace-level analytical control of post-cleaning surfaces to prevent hazardous carry-over into food matrices [179].

Dairy-processing environments represent a high-risk setting due to the intensive use of alkaline and chlorine-free cleaning agents in closed-loop CIP systems. Incomplete rinsing can result in the persistence of residues on product-contact surfaces, and chlorine-free cleaning practices have been shown to modify chemical residue profiles on equipment and pipelines, requiring dedicated analytical monitoring to ensure compliance with hygiene and safety requirements [180].

Various chromatographic applications have been developed for the determination of cleaning- and disinfectant-related residues, encompassing QACs, non-quaternary biocides, preservatives, disinfectants, cationic compounds, and surface-derived residues across dairy matrices, CIP-validation samples, rinse water, and stainless-steel surfaces. These methods employ UHPLC-MS/MS, LC-MS/MS, LC-HRMS, electromembrane extraction, magnetic SPE, modified stationary phases, and trace-level HPLC methods, enabling sensitive, selective, and regulatory-compliant residue determination. A consolidated overview of these analytical applications is provided in Table 7, which summarizes key compound classes, targeted analytes, matrices, and chromatographic approaches reported in references [181–190].

Overall, effective control of cleaning- and disinfectant-agent residues requires integration of risk-based cleaning design, validated sampling strategies, and sensitive chromatographic methods to ensure robust chemical-hazard control and compliance with PRP requirements.

4.3. Lubricant and Technical Fluid Contamination

Lubricants and technical fluids are widely used in food processing industries for the operation of equipment such as conveyors, bearings, compressors, hydraulic systems, and filling units and are recognized as potential indirect sources of chemical contamination when leaks, overapplication, or seal failures occur [191]. They consist of base oils blended with additives that provide thermal stability, load-bearing performance, and corrosion protection, characteristics that also make them relevant candidates for incidental food contact under industrial operating conditions [192].

Food-grade lubricants are formulated for applications where incidental food contact may occur and are subject to defined quality and safety criteria. Regulatory-oriented studies describe their classification, intended uses, and the importance of proper selection and handling to minimize contamination risks, particularly in high-throughput processing

environments [193]. Increasing equipment complexity and extended production cycles further reinforce the need for analytical verification of lubricant-related residues as part of PRP implementation.

A comprehensive set of chromatographic applications has been developed for the determination of lubricant- and technical-fluid-derived residues across food, edible oils, packaging simulants, and food contact materials. These include normal-phase LC, GC-FID, HPLC-GC, SPE-GC, LC-MS/MS, GC-MS/MS, and LC-ESI-MS approaches for the characterization of MOSH/MOAH fractions, lipid-like migrants, degradation products, ester-oil transformation products, antioxidants, and additive profiles. Recent studies have also addressed the analytical challenges posed by biodegradable and natural oil-based lubricants, which generate polar hydrolysis- and oxidation-derived by-products requiring dedicated chromatographic detection beyond conventional residue methods. A consolidated overview of these applications is presented in Table 7, summarizing compound classes, target analytes, matrices, and analytical methodologies reported in references [194–202].

Overall, lubricant and technical fluid contamination remains an analytically demanding but important aspect of chemical risk management in food processing industries. The combined use of targeted chromatographic methods, advanced sample preparation techniques, and risk-based monitoring is essential for effective control of these indirect contaminants within PRP-based food safety systems.

4.4. Insecticide Residues from Indoor Pest Control

Indoor pest control treatments can leave insecticidal residues on equipment, structural surfaces, or stored foods within processing facilities. The compounds most commonly detected indoors belong to the synthetic pyrethroid and neonicotinoid classes, with diamides contributing to a smaller extent due to their targeted use in structural pest management programmes. This subsection focuses on insecticidal active substances used in structural pest management (pest control treatments) and the resulting residues in facility environments. In this context, pest control insecticides (e.g., insecticides for arthropod control) should be distinguished from disinfectants, which represent a separate category of sanitation-related substances.

Synthetic pyrethroids, characterized by low volatility, strong surface affinity, and high stability, persist on metal and polymeric materials and accumulate in dust and packaging, enabling secondary transfer to food products. Their determination relies mainly on GC-MS/MS and LC-MS/MS supported by microextraction techniques that reduce solvent use and enhance trace-level sensitivity [203]. Neonicotinoids such as imidacloprid, acetamiprid, and thiamethoxam may also be encountered in facility environments and show persistence together with stable metabolites (e.g., olefin, urea, hydroxy derivatives) detected on treated surfaces. SPE or QuEChERS extraction followed by LC-MS/MS or HRMS allows selective identification of parent compounds and metabolite residues at low μg/kg levels [204].

Diamide insecticides, including chlorantraniliprole and flubendiamide, represent a smaller but relevant group of indoor residues. Their polarity and structural diversity require clean-up prior to LC-MS/MS to minimize matrix interference [205]. For flubendiamide, both parent compound and the des-iodo metabolite can be reliably determined using LC-UV or LC-MS/MS [206].

Green microextraction approaches enhance the analytical capacity for pyrethroid determination. Hydrophobic deep-eutectic-solvent ferrofluids enable rapid magnetic preconcentration with GC-MS/MS detection at ng/mL levels [207]. Rhamnolipid-based bio-supramolecular solvents support low-solvent HPLC-UV analysis of pyrethroids in complex matrices [208]. Broader evaluations confirm the suitability of deep eutectic sol-

vents across multiple microextraction formats, enhancing selectivity and reducing solvent consumption in pesticide residue monitoring [209].

A broad spectrum of chromatographic applications has been developed to verify indoor pesticide residues across rodenticides, neonicotinoids, organophosphates, pyrethroids, and other insecticide classes relevant to food processing facilities. These include multi-residue determinations, chiral separations, micellar- and magnetic-based microextraction approaches, modified QuEChERS pretreatment techniques, dual-detector GC analyses, and high-resolution LC-MS methods applied to a wide range of commodities and contamination scenarios. Collectively, the studies reported in references [210–220] demonstrate the analytical diversity required to detect parent compounds, metabolites, stereoisomers, and degradation products at low concentrations, while also highlighting the influence of washing and processing steps on residue behaviour. A consolidated overview of these applications is presented in Table 7, which summarizes target analytes, matrices, and chromatographic methodologies relevant to indoor pest control verification.

These applications demonstrate the analytical capacity required to effectively monitor indoor-relevant pesticide residues. They further indicate that sensitive, low-solvent chromatographic approaches are essential for meeting the verification and control requirements established under ISO 22002-100:2025.

Table 7. Consolidated overview of chromatographic applications for the determination of packaging-related migrants, cleaning and disinfectant residues, lubricant- and technical-fluid-derived contaminants, and indoor pesticide residues relevant to PRPs under ISO 22002-100:2025.

Ref.	Class/Chemical Group	Target Analytes	Matrix	Analytical Technique(s)	Key Analytical Notes
Packaging-related chemical contaminants					
[164]	Multiple packaging-related migrants	Various emerging contaminants	Food contact materials	LC-MS/MS, GC-MS/MS, HRMS	Integration of targeted and untargeted approaches
[165]	Volatile and semi-volatile migrants	VOCs, SVOCs	Polypropylene containers → food simulants	GC×GC-TOF-MS	Reveals complex migration profiles
[166]	Bisphenols, phthalates, oligomeric NIAS	BPA/BPS, phthalates, oligomers	Infant food packaging	Targeted LC-MS/MS and suspect screening	Migration influenced by multilayer design and storage
[167]	Plasticizers	Phthalates, adipates, citrates	Various packaging plastics	GC	Migration dependent on fat content, temperature, contact time
[168]	Plasticizers	Phthalates	Packaging materials and foods	Microextraction and GC/LC	Improved preconcentration with reduced solvent use
[169]	Recycled plastic NIAS	Degradation and oxidation products	Recycled plastics	Non-target HRMS	Complex NIAS mixtures identified
[170]	Recycled material NIAS	Oxidation products, unknown migrants	Recycled plastics	HRMS, suspect screening	Supports identification of potentially hazardous NIAS
[171]	Isothiazolinone preservatives	Methylisothiazolinone, others	Food contact adhesives	LC-MS/MS	Selective quantification for routine compliance
[172]	Paper/cardboard migrants	Semi-volatile migrants	Fibre-based packaging	DI-SPME–GC-MS	Efficient screening under realistic contact conditions
[173]	Photoinitiators, bisphenols, plasticizers	Photoinitiators, bisphenols, phthalates, antioxidants, biocides	Paper and cardboard	GC-Orbitrap	Expanded non-targeted detection capabilities

Table 7. *Cont.*

Ref.	Class/Chemical Group	Target Analytes	Matrix	Analytical Technique(s)	Key Analytical Notes
Packaging-related chemical contaminants					
[174]	NIAS (paper-based)	Multiple NIAS classes	Recycled paper/cardboard	LC-HRMS/MS	High-resolution profiling of packaging migrants
[175]	Mineral oil hydrocarbons (MOH)	MOSH/MOAH	Cocoa products contaminated via jute bags/recycled packaging	LC-GC	Supply-chain contribution demonstrated
[176]	Mineral oil hydrocarbons	MOSH/MOAH	Packaging migrants	On-line LC-GC	Improved fractionation and reduced interferences
[177]	Mineral oil hydrocarbons	MOSH/MOAH	Routine surveillance	LC-GC-FI (with N_2 carrier)	Nitrogen as alternative carrier gas with acceptable performance
Cleaning and disinfectant agent residues					
[181]	Residual milk proteins and cleaning compound residues	Stainless-steel surfaces after CIP	SPE-RP-HPLC	Sensitive, reproducible quantification for CIP validation	Residual milk proteins and cleaning compound residues
[182]	Multiple QACs (e.g., BACs, DDACs)	Milk, yogurt, powdered dairy products	UHPLC-MS/MS	Low µg/kg detection, supports routine regulatory monitoring	Multiple QACs (e.g., BACs, DDACs)
[183]	N-(3-aminopropyl)-N-dodecylpropane-1,3-diamine	Dairy matrices	LC-MS/MS	High selectivity and sensitivity for non-quaternary biocides	N-(3-aminopropyl)-N-dodecylpropane-1,3-diamine
[184]	BACs and DDACs	Milk and dairy products	Multiresidue LC-MS/MS	EU-criteria-compliant performance for official control	BACs and DDACs
[185]	>100 biocidal actives: QACs, isothiazolinones, phenolics, etc.	Dairy products and slurry feed	LC-HRMS after acetate-buffered QuEChERS	LOQs 10 ng/g, full SANTE compliance, wide screening capability	>100 biocidal actives: QACs, isothiazolinones, phenolics, etc.
[186]	Representative disinfectant actives	Wash-water, wastewater, sanitation fluids (in-field)	Portable miniaturized LC	Low-solvent, rapid on-site screening, lower sensitivity than benchtop LC	Representative disinfectant actives
[187]	Quaternary and non-quaternary cationic substances	Cleaning-validation samples	Electromembrane extraction and RP-HPLC	Enhanced selectivity and sensitivity for trace residues	Quaternary and non-quaternary cationic substances
[188]	Multiple polar/semi-polar analytes	Surface and rinse-water samples	LC with modified stationary phases	Improved selectivity using poly(dimethyldiphenylsiloxane)-nitrile phases	Multiple polar/semi-polar analytes
[189]	Multiple disinfectant/antiseptic residues	Cleaning and sanitation samples	Magnetic SPE (functionalized nanoparticles) and HPLC–MS/MS	Efficient enrichment, reduced preparation time, high recovery	Multiple disinfectant/antiseptic residues

Table 7. *Cont.*

Ref.	Class/Chemical Group	Target Analytes	Matrix	Analytical Technique(s)	Key Analytical Notes
Cleaning and disinfectant agent residues					
[190]	Residues recovered from stainless-steel sampling	Stainless-steel surfaces (CIP validation)	Recovery testing and HPLC-based determination	Multi-step reconditioning improves reproducibility (>90% recovery)	Residues recovered from stainless-steel sampling
Lubricant and technical fluid contamination					
[194]	Lubricant-derived migrants from food contact materials	Lipid-like lubricant fractions	Food contact simulants, packaging systems	Normal-phase LC	Demonstrates migration of lubricant components into simulants, supports inclusion in routine monitoring.
[195]	Biodegradable lubricants	Polar degradation by-products	Ester-based biodegradable lubricants, residues in processing environments	LC-based chromatographic monitoring	Detects hydrolysis-derived low-MW compounds not captured by conventional residue methods.
[196]	Natural oil–based lubricants	Oxidation and transformation products	Lubricant residues in industrial settings	LC/MS	Monitors oxidative degradation pathways, highlights need for dedicated methods.
[197]	Mineral oil hydrocarbons (MOSH)	MOSH fractions	Foods exposed during harvesting, transport, storage	On-line HPLC-GC-FID	Shows transfer of MOSH to food, high relevance for fat-rich matrices.
[198]	Mineral oil hydrocarbons (MOSH)	MOSH fractions in edible oils	Edible oils, industrial fluids	Large-scale SPE and GC-FID	Enhances sensitivity and robustness for MOSH profiling in oils.
[199]	Lubricant additives and residues	Multi-class lubricant-related compounds	Incidental contamination events, equipment surfaces	Combined GC-MS/MS and LC-MS/MS	Supports source differentiation and profiling of additive signatures.
[200]	Ester-based lubricants	Oxidation, hydrolysis and tribological degradation products	Lubricants aged under thermal/ tribological stress	LC-ESI-MS	Distinguishes primary lubricant constituents from degradation by-products at trace levels.
[201]	Mineral oil hydrocarbons (MOSH/MOAH)	MOSH and MOAH fractions	Complex food and food contact matrices	Standardized LC–GC approaches	Enables selective determination, aligned with harmonized monitoring criteria.
[202]	Antioxidants and degradation markers	Lubricant-derived antioxidants, oxidative markers	Food contact materials and technical fluids	TLC and spray-MS	Provides rapid screening capability for lubricant-related contaminants.
Pesticide Residues from Indoor Pest Control					
[210]	Anticoagulant rodenticides	First- and second-generation anticoagulants	Contaminated food materials	HPLC-DAD, HPLC-fluorescence	Qualitative multi-residue screening in complex matrices
[211]	Anticoagulant rodenticides	Multiple rodenticides	Accidentally contaminated foods	Rapid LC-MS/MS	Short run times, reduced sample handling for routine control
[212]	Anticoagulant rodenticides (chiral)	Bromadiolone, difenacoum, brodifacoum stereoisomers	Biological matrices, contamination verification	Chiral LC-MS/MS	Enantiomer-specific quantification, differential persistence analysis

Table 7. *Cont.*

Ref.	Class/Chemical Group	Target Analytes	Matrix	Analytical Technique(s)	Key Analytical Notes
Pesticide Residues from Indoor Pest Control					
[213]	Neonicotinoids	Imidacloprid, acetamiprid, clothianidin and metabolites	Wine matrices	Direct-injection LC-MS/MS	Simultaneous multi-residue determination with minimal prep
[214]	Neonicotinoids	Imidacloprid	Leafy vegetables	Micellar-based microextraction and HPLC	Low-solvent extraction, selective imidacloprid determination
[215]	Organophosphates	Dimethoate, quinalphos, chlorpyrifos	Bean vegetables	Modified QuEChERS and GC-FTD	Quantitative screening for high-consumption crops
[216]	Organophosphates	Chlorpyrifos, trichlorfon	Aquaculture products (shrimp)	LC-MS/MS, GC-MS/MS	Low μg/kg detection, monitoring of illegal/ off-label use
[217]	Organophosphates and pyrethroids	Malathion, λ-cyhalothrin	Zucchini	GC-NPD, GC-ECD	Optimized solvent extraction, dual-detector confirmation
[218]	Multiple insecticide classes	Fipronil, indoxacarb, avermectin, pyridaben	Beverages (tea, juices)	d-SPME and DLLME and HPLC-MS/MS	Trace-level (ng/L) preconcentration with minimal solvent
[219]	Neonicotinoids (multi-residue)	Multiple neonicotinoids	Fruit samples	Magnetic SPE (Ti-modified silica-PSA) and UPLC-HRMS	High clean-up efficiency, improved selectivity vs. conventional SPE
[220]	Various insecticides	Emamectin benzoate, spirotetramat, tolfenpyrad, fipronil	Green and red chilli	LC-MS/MS	Evaluation of washing/ processing effects, dehydration concentrates residues

5. From Standards to Science: Integrating ISO 22002-100:2025 with GFSI Requirements Through Chromatographic Evidence

The publication of ISO 22002-100:2025 has strengthened the alignment between prerequisite programme requirements across the food, feed, and packaging sectors and the core expectations embedded in internationally recognized food safety standards, including those defined in major GFSI-recognized certification schemes. When compared with the detailed provisions of FSSC 22000, BRCGS Food Safety, and IFS Food, a consistent convergence emerges across key aspects that directly affect product integrity and certification performance, including allergen control, food fraud mitigation, traceability, and the management of chemical and packaging-related contaminants. ISO 22002-100:2025 establishes explicit operational prerequisites for validated allergen-cleaning procedures, structured vulnerability assessments for authenticity, traceable control of externally provided materials, and analytical verification of chemical hazard controls, including residues arising from cleaning chemicals, disinfectants, lubricants, and packaging migrants [1]. These requirements are closely aligned with those found in FSSC, BRCGS, and IFS, all of which emphasize demonstrable control measures, documented verification of PRPs, and the use of scientifically justified methods for assessing the effectiveness of allergen, authenticity, and chemical contamination controls [221–223].

The increasing consistency among these standards reflects the broader effort of ISO to ensure that its technical requirements remain compatible with globally accepted expectations for food safety assurance, thereby supporting uniformity in compliance interpretation, auditing depth, and certification reliability. In this context, chromatographic analysis emerges as a natural technical interface between ISO- and GFSI-recognized requirements,

providing objective and reproducible measurements that allow organizations to demonstrate conformity.

Building on this alignment, ISO 22002-100:2025 introduces a decisive conceptual advance by redefining prerequisite programmes as measurable performance obligations rather than procedural requirements. The standard explicitly expects organizations to demonstrate the actual effectiveness of their preventive controls through quantifiable outcomes, moving PRP verification from documentation-based confirmation to scientifically supported assessment [1]. This expectation is consistent with the requirements of FSSC, BRCGS, and IFS, all of which mandate that verification activities be based on technically adequate, scientifically justified methods for determining whether allergen control, authenticity protection, traceability measures, and chemical contamination controls are functioning as intended [221–223].

In a risk-based compliance model, ISO 22002-100:2025 integrates PRP specifications with chemical food safety requirements by establishing the need for verification evidence that controls operate as intended. In Europe, chemical hazards relevant to PRPs, such as residues from cleaning chemicals and lubricants, pest control chemicals, and packaging migrants, are managed through documented controls supported by measurable verification. In this context, chromatographic results become compliance-relevant evidence by substantiating control effectiveness against predefined acceptance criteria and enabling audit-ready demonstration that PRPs deliver measurable performance rather than procedural conformity alone. Here, "chemical food safety" within the FSMS is understood as risk-based control of chemical hazards arising from incoming materials and packaging interactions, processing, hygiene operations, and equipment maintenance [1,3]. Operationally, this approach aligns hazard identification and PRP control measures with defined verification activities, documented acceptance criteria/decision limits, and corrective actions when criteria are not met [3,4].

From a PRP verification perspective, method selection is fit-for-purpose and risk-based: high-throughput screening may support routine monitoring, whereas confirmatory chromatographic evidence is required for specificity, traceability, or results suitable for regulatory use. Practical implementation is determined by matrix effects, sampling design, sample preparation requirements, instrument availability, and the feasibility of validated performance characteristics (e.g., selectivity, sensitivity, and reproducibility) across sites. Accordingly, verification is often structured as a two-step scheme in which predefined performance criteria (acceptance criteria/decision limits) guide the transition from routine checks to targeted LC-MS/MS or GC-MS confirmatory analysis, generating traceable, measurable evidence suitable for risk-based verification and auditing [3,4].

Within this context, the "From Standards to Science" transition becomes evident: analytical data are no longer supplementary but constitute the primary means of demonstrating control effectiveness. Chromatographic measurements in particular provide the level of precision, reproducibility, and traceability needed to substantiate PRP performance, thereby meeting both ISO's strengthened technical expectations and the evidence-based verification approach required by GFSI-recognized certification schemes [1,221–223].

As demonstrated across the allergen, authenticity, and chemical contaminant applications reviewed in Sections 2–4, chromatographic techniques constitute the primary analytical basis through which organizations can generate the objective evidence required to demonstrate effective PRP performance. Advanced analytical methods such as LC-MS/MS, GC-MS/MS, and LC-HRMS provide the quantitative precision, structural specificity, and traceability needed to verify whether allergen removal, packaging migration controls, auxiliary chemical residues, and lubricant or pesticide contamination are maintained within acceptable limits. These data-driven outputs support the risk-based auditing approach

defined in ISO 22003-1:2022 [4], enabling auditors and operators to assess control effectiveness using measurable and reproducible criteria rather than procedural descriptions alone. Accordingly, chromatographic evidence has evolved from a supplementary analytical tool to a compliance-critical component of modern food safety verification.

Despite this progress, notable analytical gaps remain: many chromatographic methods for cleaning agents and disinfectants have been validated primarily in water, surface swabs, or CIP-rinse matrices rather than in complex food matrices, and methods for lubricant-derived contaminants remain largely focused on MOSH/MOAH fractions in oils and packaging materials rather than diverse food products. These limitations underline the need for broader method development, matrix-appropriate extraction protocols, and expanded reference materials to fully support the evidence-based verification expectations embedded in ISO- and GFSI-recognized requirements.

In conclusion, the increasing alignment between ISO- and GFSI-recognized requirements creates a compliance environment in which PRPs must be supported by measurable verification rather than procedural descriptions. As auditing becomes more evidence-driven, analytical data, particularly chromatographic measurements, provide the technical basis for demonstrating control of allergens, authenticity risks, chemical residues, and packaging-related contaminants. In this context, analytical verification is no longer optional but an essential component of credible and reproducible certification outcomes. The integration of chromatographic evidence into PRP verification therefore represents a necessary condition for consistent, technically defensible food safety assurance across the global food chain.

6. Conclusions

The continuous updating of international food safety standards, particularly the transition toward more technically explicit PRP requirements, underscores the increasing need for verification approaches that are both analytically robust and adaptable to emerging compliance requirements.

Chromatographic analysis has become an essential component of current food safety verification, supporting the transition to measurable, evidence-based control across allergens, authenticity risks, chemical contaminants, and packaging-related migrants within ISO 22002-100:2025-aligned PRP verification. As regulatory requirements and ISO-GFSI alignment increasingly emphasize demonstrable PRP performance, analytical data provide the most reliable basis for validating preventive measures and assessing system robustness.

From a compliance perspective, chromatographic verification adds the greatest value when it delivers targeted, matrix-appropriate evidence for PRP effectiveness (e.g., allergen-cleaning verification, authenticity-related markers, and control of chemical/packaging-related contaminants). This is strengthened when results are interpreted against clearly defined acceptance criteria and decision limits and documented in a traceable form suitable for risk-based verification and auditing. Practical implementation therefore benefits from verification designs that distinguish routine monitoring from confirmatory testing when higher specificity is required. Several analytical approaches reported in the literature do not yet consistently meet the level of matrix-specific validation, decision limit establishment, and performance harmonization needed to support PRP verification under ISO 22002-100:2025, meaning that analytical capability does not always translate into audit-ready compliance documentation. In addition, many published chromatographic methods were developed primarily for research studies or regulatory monitoring rather than for PRP verification within management system standards, which can limit their direct transferability to audit-driven, risk-based compliance.

Ongoing developments in liquid and gas chromatographic techniques, together with expanded availability of reference materials and more consistent validation practices, will enhance the scientific robustness of food safety compliance. These advancements also indicate the need for closer coordination among laboratories, operators, and certification bodies to ensure the consistent and reliable application of analytical verification in food safety decision-making.

In this context, routine analytical verification is essential for demonstrating that PRPs operate as intended, particularly when chromatographic results are interpreted against pre-defined acceptance criteria/decision limits and documented in a traceable, audit-ready form suitable for risk-based verification and auditing. By focusing on PRP verification and auditability, this review distinguishes analytical innovation from compliance readiness and highlights method development priorities relevant to ISO- and GFSI-aligned systems. Future research should prioritize developing matrix-appropriate methods and expanding analytical coverage for emerging contaminants, ensuring that verification practices keep pace with evolving PRP requirements.

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Abbreviations

The following abbreviations are used in this manuscript:

PRPs	Prerequisite programmes
NIAS	Non-intentionally added substances
HPLC	High-performance liquid chromatography
GC	Gas chromatography
GFSI	Global Food Safety Initiative
ED05	Eliciting Dose for 5% of the allergic population
VITAL	Voluntary Incidental Trace Allergen Labelling
FAO	Food and Agriculture Organization
WHO	World Health Organization
RfDs	Health-based reference doses
ELISAs	Enzyme-linked immunosorbent assay
LFDs	Lateral flow devices
PCR	Polymerase chain reaction
LC-MS/MS	Liquid chromatography–tandem mass spectrometry
MRM	Multiple reaction monitoring
HRMS	High-resolution mass spectrometry
DIA	Data-independent acquisition
ESI	Ion-exchange chromatography with electrospray ionization
MALDI	Matrix-assisted laser desorption/ionization

SRM	Selected reaction monitoring
QC	Quality Control
AOAC	Association of Official Analytical Collaboration
CEN/TC 275/WG12	European Committee for Standardization, Technical Committee 275—Food Analysis—Working Group 12: Food Allergens
iFAAM	Integrated Approaches to Food Allergen and Allergy Risk Management
CRMs	Certified reference materials
SPE	Solid-phase extraction
TLC	Thin-layer chromatography
IEC	Ion-exchange chromatography
DAS	Double Antibody Sandwich
LFIA	Lateral flow immunoassay
LOQ	Limit of Quantitation
LOD	Limit of Detection
UHPLC	Ultra-high-performance liquid chromatography
VACCP	Vulnerability Assessment and Critical Control Points
TACCP	Threat Assessment and Critical Control Points
VOC	Volatile organic compounds
HPTLC-MS	High-performance thin-layer chromatography coupled with mass spectrometry
EVOO	Extra virgin olive oil
LC-IRMS	Liquid chromatography–isotope ratio mass spectrometry
NMR	Nuclear Magnetic Resonance
PDO	Protected Designation of Origin
PGI	Protected Geographical Indication
CSIA	Compound-specific isotope analysis
IRMS	Isotope ratio mass spectrometry
MIR	Mid-Infrared
NIR	Near-Infrared
HS-SPME-GC-MS	Headspace solid-phase microextraction–gas chromatography–mass spectrometry
GC-C-IRMS	Gas chromatography–combustion–isotope ratio mass spectrometry
TGA	Thermogravimetric Analysis
TAG	Triacylglycerols
DAG	Diacylglycerols
UV	Ultraviolet
SFC-Q-TOF-MS	Supercritical fluid chromatography–quadrupole time-of-flight-mass spectrometry
RP-HPLC	Reversed-phase high-performance liquid chromatography
MALDI-TOF-MS	Matrix-assisted laser desorption/ionization–time-of-flight–mass spectrometry
UHPLC-ELSD/UV	Ultra-high-performance liquid chromatography–evaporative light scattering detection/ultraviolet detection
PCA	Principal Component Analysis
PLS-DA	Partial Least-Squares Discriminant Analysis
HMF	Hydroxymethylfurfural
HPLC-RID	High-performance liquid chromatography-refractive index detection
GC-FID	Gas chromatography-flame ionization detection
GC-IMS	GC-ion mobility spectrometry
HPLC-DAD	High-performance liquid chromatography with diode array detection
ICP-OES	Inductively Coupled Plasma—Optical Emission Spectrometry
ID	Identity document
FT-NIR	Fourier-transform NIR spectroscopy
MGO	Methylglyoxal

OPLS-DA	Orthogonal Partial Least Squares-Discriminant Analysis
GC-GC/TOF-MS	Comprehensive two-dimensional gas chromatography coupled with time-of-flight mass spectrometry
DI-SPME-GC-MS	Direct immersion–solid-phase microextraction–gas chromatography–mass spectrometry
MOSHs	Mineral oil saturated hydrocarbons
MOAHs	Mineral oil aromatic hydrocarbons
SVOCs	Semi-volatile organic compounds
BPA	Bisphenol A
BPS	Bisphenol S
MOHs	Mineral oil hydrocarbons
CIP	Cleaning-in-place
SPE-RP-HPLC	Solid-phase extraction combined with reversed-phase high-performance liquid chromatography
QACs	Quaternary ammonium compounds
BACs	Benzalkonium chlorides
DDACs	Dialkyl-dimethylammonium chlorides
QuEChERS	Quick Easy Cheap Effective Rugged Safe
SANTE	Directorate-General for Health and Food Safety of the European Commission
GC-FTD	Gas chromatography–flame thermionic detector
GC-NPD	Gas chromatography with nitrogen-phosphorus detection
GC-ECD	Gas chromatography–electron capture detector
DLLME	Dispersive liquid–liquid microextraction
PSA	Porous Silica
FSSC	Food Safety System Certification 22000
BRCGS	British Retail Consortium Global Standards
IFS	International Featured Standards

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