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FOREWORD

This document provides guidance on the validation requirements for analytical methods used for pre-registration and monitoring in the context of pesticide authorisation. Analytical methods are used to generate data to estimate dietary exposure, establish Maximum Residue Levels (MRLs), determine processing factors, and assess storage stability. Analytical methods are also used in support of environmental fate studies, efficacy studies, ecotoxicological and toxicological studies (including dose verification), as well as operator, worker, resident, and bystander exposure studies. Analytical methods are also used to enable compliance with any statutory MRLs.

In 2003, the Organisation for Economic Co-operation and Development (OECD) initiated work to develop harmonised Test Guidelines and Guidance Documents on pesticide residue chemistry. Harmonised guidelines are essential to further work sharing goals of the Working Party on Pesticides (WPP) for pesticide registration and re-registration. The first edition of the Guidance Document on Pesticide Residue Analytical Methods was published in March 2007.

Revision of this Guidance Document was added to the work plan of the WPP in 2021 with an aim to further improve and update the Guidance Document on Pesticide Residue Analytical Methods to allow for improved harmonisation across OECD countries. Beginning in February 2023, an OECD Expert Group, co-chaired by Germany and Business and Industry Advisory Committee (BIAC) and composed of experts from Canada, Costa Rica, France, Germany, the United Kingdom, the United States, the European Food Safety Authority and BIAC, initiated discussions on the revision process and, in particular, the scope of the revised guidance. As a result, the Expert Group decided to expand the scope of the guidance to include additional matrices and method types (e.g. environmental matrices, dose verification methods, etc.). The work was divided into subgroups (matrix grouping, dose verification methods and extraction efficiency). Where necessary, joint sub-group discussions were initiated to facilitate harmonisation within this guidance and alignment with other ongoing OECD test guideline revisions (i.e., matrix grouping alignment with the revised Test Guideline 506 Stability of Pesticide Residues in Stored Commodities).

The revision to the Guidance Document incorporated several significant updates, including newly added sections on extraction efficiency, matrix and commodity groupings, and designations with respect to analytical methods. A new section and accompanying table on dose verification methodologies were also introduced. This revision includes a reformatting and reorganisation of existing text and sections, as well as an integration of feedback received during the initial request phase. These updates were made to enhance harmonisation with contemporary global guidance documents and to ensure the validation criteria reflect the most recent standards.

The updated Guidance Document was circulated to the Working Group of the National Coordinators of the Test Guidelines Programme (WNT) and WPP for review and comments on two occasions, September 2025 and February 2026. The feedback received was incorporated into the document, and the Guidance Document was approved in July 2026.

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ABBREVIATIONS

AAS	Atomic Absorption Spectroscopy
AOEL	Acceptable Operator Exposure Level
BIAC	Business and Industry Advisory Committee
CAS	Chemical Abstracts Service
CBC	Chemicals and Biotechnology Committee
DAD	Diode Array Detector
ECD	Electron Capture Detector
EFSA	European Food Safety Authority
ESI	Electrospray Ionisation
FID	Flame Ionisation Detector
FLD	Fluorescence Detector
GC	Gas Chromatography
GHS	Globally Harmonized System of Classification and Labelling of Chemicals
HRMS	High Resolution Mass Spectrometry
ICP	Inductively Coupled Plasma
I-IS	Injection/instrument Internal Standard
IL-IS	Isotopically Labelled Internal Standard
ILV	Independent Laboratory Validation
IS	Internal Standard
LC	Liquid-Chromatography
LC ₅₀	Concentration showing 50% lethal effect
LOD	Limit of Detection
LOQ	Limit of Quantification (here: lowest successfully validated level)
MRL	Maximum Residue Level
MS	Mass Spectrometry
m/z	Mass-to-charge ratio

NOEC	No Observed Effect Concentration
NPD	Nitrogen Phosphorus Detector
OECD	Organisation of Economic Cooperation and Development
OES	Optical Emission Spectroscopy
OPEX	Operator Exposure (also covering workers, residents and bystanders)
PAM	Pesticide Analytical Manual
P-IS	Procedural Internal Standard
RAC	Raw Agricultural Commodity
RAC _{Ecotox}	Regulatory Acceptable Concentration
RSD	Relative Standard Deviation (coefficient of variation)
SFC	Supercritical Fluid Chromatography
TRR	Total Radioactive Residue
UV-Vis	Ultraviolet-visible (detector)
WNT	Working Party of the National Coordinators of the Test Guidelines Programme
WPP	Working Party on Pesticides

1 INTRODUCTION

1. The purpose of this document is to provide guidance to applicants and risk assessors on the validation requirements for analytical methods used for pre-registration and monitoring in the context of pesticide authorisation. It is not intended to give guidance to enforcement laboratories; such guidance is available in other documents **(1)**.
2. The objective of analytical method validation is to demonstrate that the procedure, when correctly applied, produces results that are reliable.
3. Method validation is not required for methods analysing radiolabelled analytes.
4. Pre-registration methods (sometimes also referred to as data generation methods or risk assessment methods) may be categorised as either residue methods or dose verification methods. Residue methods are used to determine residues in plants and animal commodities for estimating dietary exposure, to establish MRLs, to derive processing factors, and to assess storage stability. In addition, residue methods are used to investigate the dissipation of pesticides in soil and/or water systems in environmental fate studies, or dislodgeable residues in exposure studies. Dose verification methods are mainly used in toxicological and ecotoxicological studies to confirm administered dose concentrations and/or homogeneity of the test substance in dosing vehicles.
5. Monitoring methods or enforcement methods or post-registration methods are used to determine compliance with established MRLs in or on food of plant and animal origin, but also for monitoring purposes in soil, water (drinking-, ground- and surface water), air and body fluids and tissues, if required. For the sake of simplicity, these terms are used interchangeably in this guidance and shall henceforth be referred to collectively as 'monitoring methods'.
6. For countries with their own national method validation requirements (e.g. **(2)(3)(4)**), it is important to note that this guidance is neither intended to override specific additional regional requirements, nor to establish new requirements.

2 GENERAL ASPECTS

Hazardous Substances

7. Pre-registration and monitoring methods should be developed without using hazardous substances classified according to the Globally Harmonized System of Classification and Labelling of Chemicals (GHS) as carcinogenic (category 1A or 1B), mutagenic (category 1A or 1B) or toxic for reproduction (category 1A or 1B) (e.g. diazomethane, benzene). In addition, chlorinated solvents, such as chloroform and dichloromethane, should be avoided. However, it could be unavoidable and justifiable to use chlorinated solvents in some cases, e.g. for cross-validations of historical methods to assess extractability.

8. Pre-registration methods developed and applied in studies completed prior to this revised guidance, and that do not meet the above requirement are exempt. However, using such a method in new studies is not acceptable without clear justification.

Analytes Under Consideration

9. Selection of analytes to include should take account of anticipated components in both the definition of residues for dietary risk assessment and the definition of residues for MRL enforcement and monitoring purposes. The method(s) should be capable of determining all analytes of interest (active ingredient and/or relevant metabolites/transformation products) depending on the purpose of the method. If the sample contains multiple isomers, analogues or other forms of the active substance or its relevant metabolite(s)/transformation product(s), the method(s) should be able to distinguish between the individual isomers or analogues, if required.

Extraction Efficiency / Radio-Validation

10. Extraction efficiency is defined as the ratio of the amount of analyte extracted by a solvent compared to the total amount of analyte present in a sample. The suitability of the extraction procedures applied in residue analytical methods for pre-registration and monitoring for the determination of residues in food/feed of plant and animal origin should be verified and aligned with the appropriate residue definition.

11. Extraction efficiency should be evaluated for representative commodities from plant matrix groups and animal commodities for which residue analytical methods are required. Extraction efficiency investigations are not required for dose verification methods. In the case of methods used in storage stability studies (5), extraction efficiency only needs to be considered if the study involves samples containing incurred residues. Extraction efficiency does not need to be addressed for analytical methods for animal commodities where significant (≥ 0.01 mg/kg) residues are not expected.

12. Extraction efficiency investigations are not routinely needed for water and air, since incorporation of residues into the matrix is unlikely. For soil matrices, it is generally advisable to use comparable

extraction procedures to those used in the lab and field studies (especially from radiolabelled studies). There is currently no guidance on how to address extraction efficiency for soil. The general principles outlined below may be useful for evaluating extraction efficiency for this matrix.

13. Extraction efficiency is regarded as key for the development of methods, and data should be provided for the solvents and conditions (e.g. temperature, pH, time, agitation) typically used. Poor extraction efficiency can be a major source of bias in a method and therefore may significantly influence the accuracy of the analytical results. The demonstration of extraction efficiency cannot be verified by routine method validation with fortified samples. The effectiveness of the extraction of all residues included in the residue definition may only be adequately evaluated using samples with incurred residues.

14. Primarily the suitability of the extraction procedures should be evaluated based on information from metabolism studies performed with radiolabelled pesticides. Information about the extracted fraction of the total radioactive residue (TRR) from these studies should be considered. Extraction efficiency is sufficient if (i) a large fraction of the TRR (e.g. 70%) is extractable with the extraction solvent being considered in the analytical method, and (ii) the compounds included in the residue definition account for a large portion of this extract (e.g. 50% TRR), as in SANTE/2017/10632 rev. 5 (6). Compounds included in the residue definition should not be found in significant amounts following other additional exhaustive extraction procedures. If these criteria are met, extraction efficiency is demonstrated for the tested solvent system and extraction conditions.

15. Generally, the evaluation of extraction efficiency is only necessary for residues found at or above the limit of quantification of the analytical method (residues ≥ 0.01 mg/kg in metabolism studies performed at maximum seasonal rate). For common moiety methods without previous extraction, the evaluation of extraction efficiency is not necessary.

16. Where a different solvent is used in the metabolism study compared to the analytical method being considered, cross validation may be used to address extraction efficiency. There are two types of cross validation to consider:

- (i) cross validation using radiolabelled substances: Where samples from metabolism studies are still available, the samples are extracted using the solvent and conditions from the analytical method. The amount of residue in the extractable portion of the TRR is compared to the amount of residue extracted using the original solvent of the metabolism study.
- (ii) cross validation: Where samples from supervised residue field trials with quantifiable amounts of incurred residues are available, the samples are extracted using (a) the solvent system and extraction conditions from the analytical method and (b) the solvent system and extraction conditions from the metabolism study, and the results are then compared. For both types of cross validation, the analyte of interest should not differ by more than 30%, as in SANTE/2017/10632 rev. 5.

17. For additional matrix group(s), not tested in the metabolism studies, extraction efficiency can be approximated by indirect cross validation. Results from samples of incurred residues in the additional matrix group(s), extracted with the solvents used in the metabolism studies, and solvents used in the residues analysis, could be compared. A consideration of the most appropriate metabolism data available could also be made (based on matrix extrapolation).

18. Compared to plants, honey is a relatively simple matrix, with incorporation and matrix bound residues being unlikely. Additionally, often honey samples are only diluted prior to analysis, therefore no separate studies to address the extraction efficiency in honey are routinely needed. Metabolism studies for honey are not required in the OECD guidance (7)(5) as the nature of residues in honey is determined by reference to appropriate plant metabolism and processing studies. Therefore, extraction methods used for plants could be considered sufficient to extract residues from honey also.

19. It is not expected that new animal metabolism or feeding studies are set up only to evaluate aspects of analytical method validation and extraction efficiency. For any new animal studies being planned, the study of extraction efficiency should be fully considered.

20. A justification based on 'similar' physical or chemical properties of solvents (e.g. polarity) is not sufficient to address extraction efficiency.

21. For further information regarding extraction efficiency, some useful information is included in SANTE/2017/10632 rev. 5.

Hydrolysis

22. A hydrolysis step may be required if esters, amides and/or conjugates are included in the residue definition. In order to demonstrate the efficiency of the hydrolysis, the following approaches can be applied:

- If standards of esters, amides and conjugates are available, they can be determined directly, or the efficiency of the hydrolysis can be verified with these standards for each relevant matrix group.
- If no standards are available, but hydrolytic conditions are identical to those applied in the metabolism studies, total hydrolysis is assumed if in the metabolism studies the efficiency of the hydrolysis step has been demonstrated and sufficient characterisation/identification has been carried out.
- If no standards are available and hydrolytic conditions differ from those in the metabolism studies, these conditions should be verified by performing a cross-validation study. This could be done using incurred residues from field trials or metabolism studies and comparing the hydrolytic conditions of the method to those used in the metabolism study or varying conditions (e.g. use of strong acid or base, refluxing overnight, use of enzymes). The hydrolytic conditions of the method can be considered sufficient, if no additional conjugates/esters are cleaved by the extreme conditions ($\pm 20\%$ are considered acceptable).

Confirmatory Techniques

23. Confirmatory analysis is not required for pre-registration methods for data generation purposes. However, confirmatory methods are required for monitoring methods to demonstrate the selectivity of the primary method. Confirmation can be performed concurrently with primary detection or by an alternative analytical technique.

24. Concurrently performed confirmation is possible when the primary method is based on mass spectrometry or another highly specific method. For example:

- For single quad mass spectrometry (MS), validation for a total of 3 characteristic mass ions, including the molecular ion and/or fragment ions
- For triple quad mass spectrometry (MS/MS), validation for 2 mass transitions
- High resolution MS: by monitoring 2 ions with a mass accuracy of ≤ 5 ppm (< 1 mDa for $m/z < 200$), preferably including the molecular ion, (de)protonated molecule or adduct ion and at least one fragment ion.

25. If confirmation by an alternative analytical technique is required, the following techniques are most commonly applied and considered acceptable:

- Different ionisation technique (e.g. ESI vs. APCI)
- Change in ionisation polarity (ESI negative vs. ESI positive)

- Chromatographic stationary phase of significantly different selectivity (e.g. reverse phase ⇔ normal phase ⇔ hydrophobic interaction ⇔ hydrophilic interaction ⇔ pi-pi bonding ⇔ ion-exchange)
- An alternative chromatographic principle deviating from the primary method (e.g. LC ⇔ GC ⇔ SFC)
- An alternative detection technique (e.g. MS ⇔ ECD ⇔ DAD) or high resolution/accurate mass MS
- Additionally, the following techniques can be considered if previously mentioned techniques were not successful.
- Change in mobile phase such as methanol vs. acetonitrile (may not be applicable to stereoisomeric analyses) and/or change in pH for acid/base compounds (ideally ≥ 3 pH units)
- Derivatisation (if it was not the first choice method)

26. No confirmatory method is required for the determination of elements by AAS, ICP-OES or ICP-MS since the analysis is specific.

Derivatisation

27. For analysis of some compounds, such as those with high polarity or with poor chromatographic properties, derivatisation may be necessary. Derivatives may be prepared prior to chromatographic analysis or as part of the chromatographic procedure (pre- or post-column). The use of derivatisation methods should be fully reported and justified. The derivative should be stable and its formation reproducible.

28. When quantification is based on the determination of a derivative, the calibration is preferably conducted using standard solutions of that derivative, unless the derivatisation step is an integral part of the detection system. If the derivative is not available as a reference standard, it should be generated within the analytical set by using the same derivatisation procedure as that applied for the samples. Under these circumstances, a full justification should be given.

29. The efficiency (mean yield) and precision of the derivatisation step should be demonstrated with analyte in sample matrix against pure derivative. If no pure derivative is available, a justification of the suitability of the derivatisation reaction should be provided (e.g. by suitable literature data). The method is considered to be specific to the analyte of interest if the derivatized species is specific to that analyte. However, when the derivative formed is a common derivative of two or more active substances or metabolites or is classed as another active substance, the method should be considered non-specific.

Common Moiety Methods and Non-Specific Methods

30. For some analytes, specific residue analytical methods might be unavailable or difficult to perform. In these cases, conversion to a common moiety is valid when all components containing that moiety are considered toxicologically important and when no single component is an adequate marker of residue concentration.

31. However, disadvantages of using non-specific or common moiety methods are:

- When a non-specific method has been used, the identity of the source of the analyte is likely to be questionable. For example, the method may also detect breakdown products either containing a moiety common to the intended analyte, or which have been derivatized to a common species, or which cannot be resolved from the target analyte. Such methods may also be subject to interferences from other similarly structured compounds.

- When analysing active substance content in a product that has undergone storage as part of a storage stability study, stability/degradation may be impossible to determine with a method that is not specific to the active substance.
- When the method determines a moiety common to two or more distinct active substances with differential toxicities, it may not be possible to perform separate risk assessments on the toxicologically relevant residue components.

32. In practice, data may have to be generated in such a way to give flexibility to establish two separate residue definitions where appropriate, one for risk assessment and a second for MRL compliance and/or monitoring. In such cases, where possible, either separately analyse for the individual components of the residue definition, rather than carrying out a common moiety method; or, carry out first analyses according to a common moiety approach, and a second series of analyses for a suitable indicator molecule in parallel, if the common moiety methodology is unsuitable for practical routine monitoring at a reasonable cost. The availability of appropriate methods for monitoring purposes should be considered.

33. Non-specific and common moiety methods will be acceptable only in exceptional circumstances where there is no other practical means of determining the target analyte. In these cases, full justification should be provided. This should include an explanation as to why the compound cannot be determined by a specific analytical technique. When common moiety methods are proposed, validation data should be presented separately for all relevant components of the residue definition.

Direct Injection Methods

34. The use of direct injection methods for samples where there is no or minimal sample manipulation before analysis (e.g. test medium, dose solutions, surface/drinking water) is acceptable. Basic method validation parameters need to be addressed: calibration data, matrix effects and precision. While recovery data is not required because no analyte losses can occur in the absence of any sample manipulation, precision should be demonstrated by fortifying blank matrix samples (≥ 5 replicates) at a defined LOQ plus a second level.

35. If dilution is performed, and the diluted sample is directly injected, it is sufficient to address the basic method validation parameters as described in paragraph 34, if a justification that the dilution step has no significant impact on the validity of the method is provided (e.g. samples are diluted with the same solvent or comparable solvent present in the original sample and demonstrate no significant losses in concentration). If such justification cannot be provided, all aspects of method validation as defined for non-direct injection methods should be addressed. In the case of adding a stabilizing agent, such as acid, and directly injecting without dilution, basic method validation parameters are sufficient.

Use of Internal Standards

36. The purpose of an internal standard is to account for potential various sources of errors in the analytical method. The use of an internal standard will not compensate for incomplete extraction of incurred residues. It should be noted that this approach is not replacing the need to evaluate extraction efficiency of incurred residues. Internal standards can be used in risk assessment and monitoring methods.

37. The internal standard should be chemically stable and typically show the same behaviour as the target analyte. A stable, isotopically labelled analyte is the ideal internal standard.

38. An isotopically labelled internal standard (IL-IS) differs from the analyte only in terms of the isotopes in the molecule (e.g. ^{15}N , ^{13}C , ^{18}O , deuterium). A prerequisite for the use of IL-ISs is the use of mass spectrometry as the detection system which allows for simultaneous detection of the analyte and

corresponding IL-IS, and that the stable-isotope labelled standard is largely free of the native analyte so that the quantification is not interfered with. Determination of matrix effects is generally not required if using a stable IL-IS. However, it is important to note that deuterium-hydrogen exchange may alter physiochemical properties and negatively impact quantitative results. Use of IL-IS can compensate for matrix effects and loss during storage in extracts.

39. A non-isotopically labelled internal standard (non-IL-IS) can be used. When selecting an internal standard, there should be confidence that the substance will not interfere with the analysis of the target analyte, and it is unlikely that they are present in the samples being analysed from any other source. Where a non-IL-IS is used, determination of matrix effects is usually required.

40. An internal standard can be added at different stages of the analytical method to compensate for analyte loss, volumetric effects, instrument injection, response drift, method variability, and matrix effects, noting that use of an internal standard will only compensate for these potential losses to downstream methods steps, following the step in which it is added. For example:

- Addition to the analytical sample, just prior to the initial extraction step. IS addition at this stage will account for various sources of errors throughout all stages in the method. This can be known as a procedural internal standard (P-IS).
- Addition to final sample extracts, just prior to the determination/injection step. This can be known as an injection/instrument internal standard (I-IS).

41. When the internal standard is added to the calibration standard solutions, the detector response ratio of analyte and internal standard obtained from the injected calibration standard solutions are plotted against their respective concentrations or the ratio of their respective concentration. The concentration of analyte is then obtained by comparing the detector response ratio of analyte and internal standard of the sample extract, against the calibration curve.

Analysis of Stereoisomers

42. Enantioselective methods are only required if specific enantiomers require quantification (e.g. stereospecific residue definitions). For the chromatographic separation of enantiomers, chiral LC or GC columns or a chiral modifier of the LC eluent may be required. Other suitable techniques, such as supercritical fluid chromatography (SFC) can also be applied for the determination of enantiomers. Quantification should be performed for the individual enantiomers, provided that sufficient chromatographic resolution can be achieved and reference compounds are available; the latter includes individual enantiomers, racemates or isomeric mixtures of known composition.

43. For more information on active substances that have stereoisomers, please refer to the EFSA guidance on stereoisomers (8).

Stability Investigations

Stability of Working (Stock/Fortification/Calibration) Solutions

44. If stability under controlled storage conditions has been demonstrated, the stock, fortification and calibration solutions can be used over the proven time period of stability. Otherwise, the solutions have to be prepared freshly on a daily basis.

45. The duration of the stability test should reflect typical usage. In general, solutions are used over a period of several days or weeks. The test conditions (e.g. appropriate solvent systems, ambient

temperature or refrigerator, light/dark) should be selected to reflect usual storage conditions applied within the conduct of analyses.

46. For stability testing, the stored solutions should be compared with freshly prepared stock, fortification and/or calibration solutions. The concentrations should be chosen so that potential degradation can be observed. If no concentration dependency is observed, it is not necessary to investigate all concentrations applied. In order to obtain reliable data, the mean concentrations of 5 replicate measurements for each of the two solutions should differ by no more than 10%. If the difference is more than 10%, a conclusion on stability will be based on a case-by-case decision.

Stability of the Analytes in Raw and Final Extracts

47. Ideally, the sample extracts are analysed within 24 hours after initial extraction. Under some circumstances they may be stored longer under ambient conditions, e.g. in the autosampler, or in a refrigerator, e.g. if the analyses cannot be completed within one working day. Information on the storage stability of the analytes in raw extracts (initial extracts before clean-up) and/or in the final extract should be provided, if stored longer than 24 hours. If the extracts contain an IL-IS for quantification, testing of final extract stability is not required since the IL-IS will compensate for losses during extract storage.

48. The relevant information on the stability in the final extract can be derived from the fortification experiments performed during method validation. If the recoveries in the fortified samples are within the acceptable range according to Table 1 for food/feed of plant and animal origin or 70-120% for all other matrices (measured with freshly prepared calibration solutions or solutions otherwise within previously demonstrated stability duration), stability is sufficiently proven for the storage duration and conditions of the method validation. It is sufficient to test extracts from a representative matrix from the respective matrix groups (in Annex A). The storage conditions tested should be reported and should reflect typical storage conditions applied during analysis.

Stability of Analytes in Stored Matrices

49. It is not a requirement of the method validation to address the stability of analytes in stored matrices. Guidance on this is available for the different specialist areas (e.g. OECD Test Guideline 506) or respective principles can be followed if no such guidance is in place. Stability testing of analytes in stored matrices is out of scope of this guidance.

3 VALIDATION PARAMETERS

50. To be acceptable for the intended purpose, the method should meet standards for certain validation parameters. Typical validation parameters for analytical methods that should be considered are: recovery, precision, selectivity (specificity), calibration, limit of detection (LOD), limit of quantitation (LOQ) and matrix effects. These parameters are defined below, including their associated minimum performance characteristics. They are of importance for both, pre-registration and monitoring methods.

Recovery

51. Recovery is the amount measured as a percentage of the amount of analyte(s) (active substance and relevant metabolites) originally added to a sample of an appropriate matrix, which contains either no detectable level of the analyte or a known detectable level. Recovery experiments provide information on both precision and trueness (bias), and thereby the accuracy of the method. While recovery is determined during initial method validation, concurrent or procedural recoveries are determined by analysis of samples as outlined here, at the same time as analysis of test samples. Their purpose is to confirm that the method was working at the time of analysis of the test samples within any given study. The results should comply with the recovery levels outlined in Table 1. For pre-registration methods, concurrent recoveries can also be used for validation/verification purposes.

52. The mean recovery for food/feed of plant and animal origin, at each fortification level and for each commodity should be in the range given in Table 1. For all other matrices (e.g. bee matrices, environmental matrices, animal test diets, body fluids), the range of mean recoveries at the relevant concentration level should be 70-120%. In certain justified cases, recoveries outside this range will be accepted for matrices which are difficult to analyse, e.g. tobacco, hops, coffee, tea and spices, providing that precision data are acceptable, or in cases of very low concentration levels (e.g. < 0.005 mg/kg in soil or < 0.05 µg/L in water).

Precision

53. According to ISO 5725-1 (9), precision is defined as the “closeness of agreement between independent test results obtained under stipulated conditions. The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results”. The relative standard deviation (RSD) should be reported at each fortification level and for each matrix. The correlation between the concentration level and the precision according to the Horwitz equation (10) is given in Table 1 for food/feed of plant and animal origin. For all other matrices (e.g. environmental matrices, animal test diets, body fluids), the precision should be ≤20% RSD. In certain justified cases, referenced in paragraph 52, (e.g. in cases of difficult matrices or very low concentration levels), a higher variability may be accepted.

Table 1: Precision and recovery criteria for analysis of pesticide residues for food/feed of plant and animal origin (11)

Concentration level	Precision (% relative standard deviation)	Range of mean % recovery
≤ 0.01 mg/kg	30	60-120
> 0.01 mg/kg - ≤ 0.1 mg/kg	20	70-120
> 0.1 mg/kg - ≤ 1.0 mg/kg	15	70-110
> 1.0 mg/kg	10	70-110

54. Outliers should be identified using appropriate statistical methods (e.g. Grubbs or Dixons test) and should not exceed 1/5 of the results at each fortification level. Where more than 1/5 of validation samples at one fortification level have been identified as outliers, additional validation samples might be included, and an explanation should be provided.

Selectivity/Specificity

55. Selectivity refers to the extent to which the method can be used to determine particular analytes in mixtures or matrices without interferences from other components of similar behaviour. To demonstrate sufficient selectivity, uncorrected recoveries and matrix blank values should be reported. Blank values in the area of analytical interest from untreated/unfortified samples must be determined from the matrices used in fortification experiments and should not be higher than 30% of the LOQ. If unavoidable due to natural background levels or environmental contamination, corrected recoveries may be reported along with the uncorrected recoveries, and a detailed justification should be provided.

56. When mass spectrometry is used for detection, a mass spectrum (in case of MS/MS; product ion spectrum) should be provided during validation to justify the selection of ions used for determination and thereby demonstrating specificity of the applied method.

Calibration

57. Calibration refers to the ability of a detection system to produce a well-defined correlation between the instrumental response and the concentration of the analyte in the sample. The analyte concentration to be measured should be within the defined dynamic range of the instrument.

58. The analytical calibration should ideally extend over a range at least from 30% of the LOQ to 20% above the highest validation level (after considering possible dilution) of the analyte in final volume solutions. Either duplicate determinations at three or more concentrations or single determinations at five or more concentrations should be used, with concentrations generally evenly distributed over the calibration range. Raw data should be presented exemplarily at least for each analyte and matrix group together with the equation of the calibration curve, a regression parameter (e.g. the correlation coefficient (r)), and a typical calibration plot. Linear weighted calibrations are preferred but non-linear calibrations can be justified (e.g. based on analyte behaviour or where suitable detection system accuracy is demonstrated).

59. In order to compensate for matrix effects, calibration can be generated using standards prepared in blank matrix extracts (matrix-matched standards). Other calibration procedures to compensate for matrix

effects are using IL-IS or standard addition (addition of standards of the analyte of interest to test samples at varying concentrations). Only if experiments clearly demonstrate that matrix effects are not significant ($\leq \pm 20\%$), calibration with standards in solvent may be used.

Limit of Detection (LOD)

60. The limit of detection of an analytical procedure is the lowest amount of an analyte in a sample that can be detected but not necessarily quantitated as an exact value. At the limit of detection, a positive identification can be achieved with reasonable and/or previously determined confidence in a defined matrix using a specific analytical method. It should be expressed as the lowest calibration standard, ideally in matrix, which should be selected based on adequate signal-to-noise ratios (e.g. $\geq 3:1$). The LOD can provide valuable information for pre-registration methods (e.g. for refinement of risk assessment).

Limit of Quantitation (LOQ)

61. Limit of quantitation is defined from a regulatory perspective as the lowest concentration tested at which an unambiguous identification of the analyte can be proven and at which an acceptable mean recovery with an acceptable relative standard deviation (RSD) is obtained. The LOQ should be low enough to achieve the intended purpose of the method. For specific LOQ requirements, refer to the respective sections under methods for pre-registration and monitoring methods and further consult specific country or regional requirements.

62. In the case where analytical limitations (e.g. detection, sensitivity, etc.) exist, such that a targeted LOQ may be unachievable (e.g. due to high matrix load or blank values), an explanation and justification should be provided.

63. In case of complex residue definition(s) containing more than one analyte, the regulatorily required LOQ may be reported (calculated as stated in the residue definition), taking into account the LOQs for the individually validated components.

Matrix Effects

64. Matrix effects are defined as an influence of one or more components from the sample matrix on the measurement of the analyte concentration or mass. It can result in an increased or decreased detector response compared to that produced by solvent solution of the analyte with the same concentration.

65. Evaluation of matrix effects is required for ionisation methods (e.g. LC-MS/MS, GC-MS(/MS)) and GC-NPD where solvent standards are used and internal standards are not used according to paragraphs 36-41. Evaluation of matrix effects is not required for other non-ionisation methods (e.g. LC-UV).

66. Assessment of matrix effects should be performed by comparing the analyte response of at least one individual standard prepared in solvent to at least one prepared in blank matrix, for all sample materials included in the corresponding validation study. Alternatively, the slope of the calibration function prepared with standards in pure solvent can be compared with that for the calibration with standards in matrix. Matrix effects, expressed as the % enhancement or suppression can be evaluated according to the following equation:

$$\text{Matrix effects [\%]} = 100 * \text{peak area or slope (matrix)} / \text{peak area or slope (solvent)} - 100$$

Matrix effects are considered significant if they exceed $\pm 20\%$.

67. For compensation of matrix effects using internal standards see paragraphs 36-41. If isotopically labelled standards and/or matrix-matched standards are used, an assessment of matrix effects is not necessary.

4 VALIDATION OF PRE-REGISTRATION METHODS

68. In general, pre-registration methods should be validated for all matrices for which the method is used. Such methods can be used for dose verification purposes (e.g. in mammalian toxicology or ecotoxicology studies) or the determination of residues (e.g. in environmental fate studies, field trials, or feeding studies). The data can be presented in multiple ways:

- Stand-alone validation: The method validation is performed separately from the actual study and presented in a separate report.
- In-study validation: The method validation is performed and reported within the study requiring an analytical method (use of concurrent recoveries is possible to address method precision and accuracy).
- In-study method verification: In case a method validation has been previously done as stand-alone or as in-study, reference should be made to this report, and a verification of the method should be presented (use of concurrent recoveries is possible to address method precision and accuracy).

69. Validation data (stand alone or in-study) should be provided for new methods or when existing methods are significantly changed (e.g. change of extraction solvents, quantitation techniques, additional or alternative clean-up steps) and/or applied to new matrix groups. Verification data is sufficient when a fully validated method is adapted to other commodities of the same matrix group or is applied by a laboratory for the first time. The extent of the required validation/verification data depends on the type of method (dose verification or residue method). Refer to Tables 3 and 5 which show the verification and validation requirements.

70. Confirmatory methods are not required for pre-registration methods (dose verification and residue methods).

Dose Verification Methods

Matrices for Validation

71. Dose verification methods are methods that are used to quantify a known analyte of a known concentration, developed in support of (eco)toxicological studies. Typical areas of application are methods to test for homogeneity of animal test diets or to verify the concentration of a test substance in test media. A comprehensive list of matrix types is presented in Table 2. Methods used to determine a suitable dosing range in (eco)toxicological studies (range finding methods) are exempt from the following requirements, as the results of these studies are used for planning of the main pre-registration studies and are not used for setting of regulatory endpoints.

Table 2: Areas of application and matrices requiring dose verification

Function	Matrix/ Formulation Types	Matrix/ Formulation Commodity Examples	Notes/Comments
Toxicology support	Gas	Air	Inhalation study types (e.g. analysis on filter paper via LC-UV, gaseous analysis by GC-MS).
	Solid	Diets for laboratory animals: (e.g. rodent [rat, mouse], dog, rabbit)	No extrapolation between matrices to be considered, reduced requirements (see below) should be fulfilled. Historic studies: See paragraph 78
	Solutions/ Suspension	Carboxy methyl cellulose Tween80 Corn oil Olive oil Mineral oil Dimethyl sulfoxide (DMSO) Ethanol Water	No extrapolation between matrices to be considered, reduced requirements (see below) should be fulfilled. Historic studies: See paragraph 78
Eco toxicology support	Solutions	Water (e.g. salt water, fresh water, drinking water) Test media (e.g. M4-Medium, AAP-Medium) Sugar solution(s) (e.g. nectar surrogate), larvae diet (e.g. chronic bee, larvae, bumble bee) Spray solutions (non-target plant tests)	No extrapolation between matrices to be considered, reduced requirements (see below) should be fulfilled. Historic studies: See paragraph 78
	Solid	Sediment Bird diet Fish diet Soil	No extrapolation between matrices to be considered, reduced requirements (see below) should be fulfilled. Historic studies: See paragraph 78

Validation Requirements for Dose Verification Methods

72. Due to their inherent purpose and the known (and often high) test concentration(s), a reduced validation data set is considered sufficient for dose verification methods (see Table 3).

Table 3: Reduced validation requirements for dose verification methods

Validation Parameter	Detailed Requirements
Levels and number of fortifications	Stand-alone or in-study <i>validation</i> studies: 2 levels to cover the intended doses; 3 fortified samples per level + 1 matrix blank. If only 1 conc. is required, a single conc. is considered acceptable + 1 matrix blank.

	In-study method <i>verification</i> : Verification of the dosing levels being tested within respective studies using concurrent recoveries (minimum of 3 fortified samples at lower dose and 1 at higher dose + 1 matrix blank).
Recovery/precision	See paragraphs 51-54.
Selectivity/specificity	Chromatograms: Minimum set 1 x matrix blank, 1 x recovery at LOQ or lowest dose level, 1 x lowest calibration standard, for each matrix type. No confirmatory method is required for dose verification studies.
Calibration	See paragraph 57-59.
LOQ	Should be at or below the lowest dose.
Matrix effects	See paragraphs 64-67.
Solution stability	See paragraphs 44-48.

Residue Methods

Matrices for Validation

73. Residue methods are methods used to quantify an unknown concentration of an analyte, developed in support of risk assessment/data generation (i.e. methods for environmental fate, toxicology, non-dietary (occupational and residential) exposure, dietary exposure or eco-toxicological studies). Typical areas of application, study types and matrix types are presented in Table 4.

Table 4: Examples of areas of application and matrices requiring residue methods

Function	Matrix/Formulation Commodity examples	Example Study types	Notes/Comments
Environmental fate support	Soil	Terrestrial field degradation (with e.g. incorporation, coverage etc.) Terrestrial field dissipation (without any treatment) Accumulation Field leaching Photodegradation in soil* Adsorption/Desorption* Degradation in soil under aerobic and anaerobic conditions* Storage Stability * For examples where no radiolabelled material is available	Where appropriate, method validation data should be submitted using standard soils or any other appropriate test soils used in environmental fate and/or ecotoxicological studies.
	Water	Aerobic and anaerobic transformation in aquatic sediment systems * Aqueous dissipation Aqueous photolysis Groundwater monitoring	Where appropriate, method validation data should be submitted using water samples (drinking or groundwater and surface water), or any other test water or media used in environmental fate and/or ecotoxicological studies. Extrapolation between different water/media types can be accepted if they only differ in their composition of salts.

		Field leaching Adsorption/Desorption* Hydrolysis* Storage stability * For examples where no radiolabelled material is available	
	Sediment	Aerobic and anaerobic transformation in aquatic sediment systems	
	Air	Photo transformation Wind tunnel (spray drift and deposition after volatilisation)	If the chemical is deemed volatile and has a potential for long-distance transport.
Toxicology support	Mammal tissues and body fluids (plasma, whole blood, urine, breast milk, tissues)	Proof of exposure studies Repeated dose studies Dermal absorption studies (<i>in vitro</i>)	
Non-dietary Exposure support (OPEX)	Clothing/fabric materials (inner/outer garment, wipes/swabs, gloves) Air filters, Aqueous solutions	Occupational exposure studies (dosimetry) Residential exposure studies Bystander exposure studies	
	Leaf wash (e.g. aqueous surfactant solution (e.g.	Dislodgeable foliar residues Turf transferable residues	

	0.01% Aerosol® OT solution or similar))		
	Cloth		
Dietary exposure support	Plant matrices (raw & processed)	Field trials - Magnitude of residue studies Field rotational crop studies Storage stability Processing studies	<u>High acid content, high water content, high oil content, dry, dry high sugar content (including their processed commodities)</u> The validation (stand alone or in-study) should be performed on at least one matrix from each of the representative matrix groups as listed in Annex A and relevant for <u>the intended uses</u>. If samples with high water content are extracted at a controlled pH (e.g., extraction with an acidified/basicified solvent), bridging between high water and high acid is possible and a particular method or validation for commodities with high acid content is not required. The matrix group scheme is not intended to imply that if the method is successfully validated using the representative commodity, the method will work for all commodities in the same matrix group. A case for matrix comparability and method verification data may be considered where two or more very similar commodities are to be analysed (see Annex A). Method verification data for commodities belonging to the same matrix group are acceptable and are still needed for commodities which are analysed with pre-registration methods. <u>Difficult matrices (including their processed commodities)</u> Commodities containing high percentages of soluble natural products that might interfere with the analytes under consideration are referred to in Annex A as “difficult”. Interference can vary depending on the methodology selected and the properties of the compounds. In case of difficult matrices, validation data are required for each raw agricultural commodity (RAC). An exception are the groups of spices and teas, where a successful method validation, using the same method for at least half of the RACs in the respective groups, allows extrapolation to additional RACs within these groups. Method verification data (use of concurrent recoveries) should be provided for the additional RACs.

			Where validation data are available for the RAC, method verification data (use of concurrent recoveries) should be provided for its processed commodities.
	Animal matrices	Livestock feeding studies Storage stability	If animals are likely to consume treated commodities and if feeding studies are required/submitted, methods for determination of residues in products of animal origin should be validated in the following matrices: milk, eggs, and tissues. The tissues normally include muscle, fat and liver or kidney. Additional matrices such as fish tissues should be validated where fish feeding studies are required. Generally, recovery data for a specific matrix can be extrapolated to the same matrix of a different species (except fish).
	Honey Pollen Nectar Royal jelly	Residues in bee product studies	The compositions of royal jelly (composed of water, lipids, proteins and products with an acidic pH) and pollen (composed of proteins, sugars and pigments) are different from the composition of honey, the analytical methods should be validated separately for each matrix. For nectar, a validation in diluted honey is acceptable.
Eco toxicology support	Water Sediment/Soil Flowers Nectar Pollen Bees/Pollinators Hyalomma tissue Fish tissue Insects (arthropods)	Bee studies, (semi)field, tunnel studies Residue studies on non-target/young plants Myriophyllum studies (water/sediment) Chron. Chironomus studies (water/sediment), spiked water or spiked sediment Earthworm studies (if degradation is to be monitored) Arthropod studies Bioconcentration factor (BCF) studies (Fish/Hyalomma)	

	Plants (young)		
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Validation Requirements for Residue Methods

74. The validation requirements are outlined in Table 5. It is important to note that these validation requirements differ from what is required for a monitoring method, which may consider the same analytes and matrices.

Table 5: Validation requirements for residue methods

Suggested Validation Requirements	Explanation
Levels and number of fortified samples:	Method validation (stand alone or in-study): LOQ (at least 5 fortified samples), 10 x LOQ or other relevant level (at least 5 fortified samples), and matrix blank (2 samples). Lower numbers of fortified samples should be justified (e.g. if available sample amount is insufficient). For a method verification, at least 3 fortified samples at each validation level plus one matrix blank (for proving sample is free of detectable residue) should be used.
Recovery/precision	See paragraphs 51-54.
Selectivity/specificity	According to paragraph 55-56. Should be demonstrated by providing at least the following chromatograms: blank, lowest standard and LOQ fortification, for each analyte and matrix No confirmatory method is required for residue methods.
Calibration	See paragraphs 57-59.
LOQ	Will depend upon the sensitivity necessary for the risk assessment. In general, it should be as low as required to meet the study needs.
Matrix effects	See paragraphs 64-67.
Stability	Raw and final extracts: See paragraphs 47-48. Working solutions (stock/fortification/calibration): according to paragraph 44-46

75. For residue methods for food and feed of plant and animal origin (excluding frozen storage stability studies), extraction efficiency should be assessed and considered when selecting the extraction solvent(s). Refer to extraction efficiency paragraphs 10-21 for further details.

Minimum Validation Requirements for Historical Data

76. Pre-registration methods developed prior to this guidance often do not meet the validation requirements. Nevertheless, in order to reduce the need to repeat studies, minimum validation requirements were defined for such methods to be deemed fit for the intended purpose. These minimum validation requirements do not replace expert judgement on the acceptability of a method.

77. For each matrix, the presented validation data should comprise at least the following parameters:

- Demonstration of linearity (calibration plot or raw data). For dose verification methods single point calibration is acceptable, provided samples were diluted to a similar concentration.

- Demonstration of selectivity and specificity (chromatogram of a sample at LOQ, chromatogram of blank sample)
- Demonstration of acceptable recovery (minimum of three recovery samples, across all levels, with at least one of them at the LOQ level). For dose verification methods, also study data (e.g. homogeneity data from animal test diets and suspensions used in toxicity studies, or measured concentrations in test medium from aqueous toxicity studies) can be used to demonstrate sufficient recovery. Combining multiple data sets is possible.

Other validation parameters such as evaluation of matrix effects or stability investigations are not required.

78. Extrapolation from an acceptable dose verification method using a similar matrix may be possible, if the validation data supports the dosing levels of the study in question. For example, extrapolation may be possible between different animal test diet types, different dosing solution concentrations (e.g., 0.5% and 1% carboxy methyl cellulose) or different test media. Other extrapolations might be possible if a justification is given.

79. Deviations from the requirements above can be justified for vertebrate animal studies. This is the case if a study contains no or insufficient validation data for dose verification.

5 METHODS FOR MONITORING

80. Generally, monitoring methods are used for those matrices of plant and animal origin where an MRL is set. If no MRL is set, there is no need to provide any further information on monitoring methods. However, some regulatory authorities will establish MRLs for trade purposes, although no residues are expected in those commodities. Monitoring methods are therefore required to demonstrate adherence to appropriate limits of quantitation and to establish MRLs at LOQ. Additionally, monitoring methods may be required for soil, drinking/ground- and surface water, air and body fluids and tissues in some countries/regions.

81. If applicable, the methods should cover the analytes included in the definition of the residue relevant for monitoring. The methods should be fast, easy to perform and use commonly available techniques/equipment. However, it might not be possible in each case to fulfil these requirements due to analyte specific characteristics, extraction efficiency constraints etc. and, hence, alternative or new analytical techniques could be considered for monitoring purposes provided respective justification is given.

82. For monitoring methods, a multi-residue method approach is clearly preferred over a single residue method as the monitoring laboratories do not have sufficient capacity to apply individual methods to all compounds that may possibly be present (1)(2)(12). Therefore, the suitability of any existing multi-residue method approaches should be assessed/considered. A complete set of validation data including calibration information, and representative chromatograms should be provided to demonstrate that the multi-residue method is able to determine the analyte of interest. If data show that multi-residue methods cannot be used, single residue methods are acceptable for monitoring.

83. Confirmatory methods are required for monitoring methods. These have the same validation requirements as the primary method (see paragraphs 23-26).

Matrices for Validation

84. For matrices of plant origin, validation should be performed for at least one representative raw agricultural commodity (RAC), from each of the following matrix groups.

- High acid content
- High water content
- High oil content
- Dry

85. RACs which are defined in Annex A as “difficult” often contain high loads of matrix in their extracts, potentially interfering with the detection of the analyte(s) of interest. Hence, a validation (primary method including confirmation and ILV) for difficult commodities should be provided. However, validation is only required for RACs of an intended use. Extrapolation within the groups of spices (see Table 7, Codex commodity group 028) and teas (Table 7, Codex commodity group 066) is possible if the same method has been successfully validated for at least half of the RACs in their respective commodity group. Within the group of spices, there are significant variations in the composition of the individual commodities. For

example, some have a high essential oil content, while others are more related to dry commodities. If there are justified grounds for deviating from "difficult" to any other matrix group, this should be supported with relevant data and/or information.

86. Commodities, which are defined in Annex A as "dry high sugar content" do not have to be validated for monitoring purposes, since they exclusively occur as processed commodities in residue studies. At the same time, they cannot be chosen as a representative commodity in validations to cover the dry matrix group.

87. For matrices of animal origin validation should be performed for milk, eggs, meat/muscle, fat and kidney or liver. In addition, validation may be required for honey.

88. If in some countries/regions, validations for additional matrices (e.g. environmental matrices, body fluids) are required, the following requirements should be considered:

- Validation for soil should be performed with a representative soil for crop growing areas.
- Validation for water should be performed with drinking water (tap or groundwater) and surface water (freshwater, e.g. from rivers or ponds).
- Validation for air should be performed, if possible, under enhanced conditions (at 35°C and at least 80% relative humidity). In justified cases (e.g. heat sensitive analyte) and if it is shown that a method does not work at 35°C and 80% RH, other conditions are applicable (e.g. ambient temperature and normal humidity).
- Validation in body fluids should be performed in one of the following matrices: blood, serum, plasma or urine.
- Validation in body tissues should be performed in one of the following matrices: meat/muscle, liver or kidney.

Limit of Quantitation

89. For plant and animal commodities, the validated LOQ of monitoring methods should generally meet 0.01 mg/kg. Exceptions are higher default MRLs for difficult matrices such as spices and hops or lower MRLs, e.g. for health protection reasons.

90. If monitoring of other matrices is required, the following LOQs are recommended if no specific country or regional requirements are in place:

- For soil, a general LOQ of 0.05 mg/kg (lower for e.g. phytotoxic herbicides, if required (2))
- For drinking water, a general LOQ of 0.1 µg/L
- For surface water, the LOQ should be at or below the lowest limit value derived from ecotoxicological endpoints (e.g. LC₅₀, NOEC, RAC_{Ecotox})
- For air, the LOQ may be derived from the AOEL (if no AOEL_{inhalative} is available, the AOEL_{systemic} or the ADI value can be used for calculation) as follows:

$$c = AOEL_{inhalative \text{ or } systemic} \times \frac{\text{safety factor} \times \text{body weight}}{\text{air intake}}$$

With safety factor: 0.1; body weight: 60 [kg]; air intake: 20 [m³/day]

$$c = AOEL_{inhalative \text{ or } systemic} \times 300 \frac{\mu g}{m^3}$$

- For body fluids and tissues at 0.01 mg/L and 0.01 mg/kg, respectively.

Validation Levels and Number of Fortification Experiments

91. Recovery data should be generated for the following fortification levels: LOQ (5 fortified samples); 10x LOQ or MRL (set or proposed) or other relevant level (5 fortified samples); and matrix blank (2 samples).

Independent Laboratory Validation (ILV) Studies

92. ILVs should be performed for multi-residue and single-residue methods. However, it should be noted that in some regions (e.g. United States) independent laboratory validation studies may only be required for single-residue methods.

93. The laboratory chosen to conduct the ILV should not have been involved in the method development and in its subsequent use. Provided this criterion is met, the laboratory chosen to conduct the ILV may belong to the same organisation where the method was developed and first validated but must not be at the same location. If the chosen laboratory requires communication with the developers of the method to carry out the analysis, this should be reported. Any subsequent additions or modifications to the primary method should be reported as well. Table 6 gives an overview of some modifications to the primary method and their consequences with regard to the acceptability of the ILV.

Table 6: Examples for acceptable and non-acceptable deviations of the ILV from the primary method

Deviation of ILV from primary method	ILV acceptable	ILV not acceptable
LOQ higher than that of the primary method and > MRL ¹		X
LOQ higher than that of the primary method and < MRL ¹	X (higher LOQ is then considered for the overall method)	
Validation with different crops from same matrix group (e.g. cereal grain ↔ dry legumes)	X	
Validation for high water content matrix is missing but ILV for 2 matrices available and primary method identical for all matrices		X
Number of replicates or levels not sufficient		X
Different extraction solvents		X
Additional clean-up steps are performed		X
Use of different laboratory equipment (shaker, vessels, pipets...)	X	
Use of different GC or LC column	X (if justified) ²	
Use of different mass or transition	X (if justified) ²	
Use of different calibration models • matrix matched-solvent • linear-quadratic	X (if justified) ²	
Use of different ionisation mode (ESI+ ↔ ESI-)	X (if justified) ²	

Use of different detection technique (e.g. LC-MS<-> LC-MS/MS or LC-MS/MS <-> HRMS)	X (if justified) ²	
Modular multi-methods (DFG S19, modular QuEChERS): use of different modules		X

¹ Lowest MRL of commodity from the respective matrix group

² For example, if interferences were detected.

Number of Matrices for Independent Validation

94. For products of plant origin, an ILV should be conducted for representative commodities of all matrix groups for which a primary method is required. If the required method is identical for all matrix groups, it is sufficient to perform the ILV for commodities of two of these groups, one of them with high water content. If a validated primary method is required for commodities difficult to analyse, an ILV should be performed for the same matrix. However, it should be noted that in some regions other requirements may apply.

95. For ILV studies for the determination of residues in products of animal origin, the following animal commodities should be used, if an MRL is established or is likely to be proposed: milk, eggs, meat/muscle and/or fat, and kidney and/or liver. If the primary method is identical for all animal matrices, it is sufficient to perform the ILV for two of these matrices.

96. ILVs may have to be provided additionally for honey and/or drinking water, depending on regional requirements (2).

97. The number of fortification experiments for an ILV should be in agreement with paragraph 91. The ILV should confirm the LOQ of the primary method or at least cover the lowest MRL.

6 STUDY REPORT

98. This section describes general information that should be included in a study report when describing analytical methods and the associated validation data.

A. Introduction.

- (i) Scope or applicability. Describe suitable commodities/matrices and source of method, e.g., the PAM, published multi-residue method, company reports, etc.
- (ii) Principles of the analytical procedure, including identification of the chemical species determined and the limits of detection (if needed) and quantitation.

B. Materials and methods.

(i) Standard Compounds.

- (1) Description e.g. Chemical name, CAS number, Chemical structure, molecular formula and mass, purity, expiration date, storage conditions.
- (2) Preparation of stock solutions.
- (3) Preparation of calibration solutions.
- (4) Preparation of fortification solutions.

(ii) Procedure. Describe detailed analytical procedure in a stepwise fashion, with special emphasis on reagents or procedural steps requiring special precautions to avoid safety or health hazards.

- (1) Preparation of sample.
- (2) Fortification, if applicable - i.e. during method validation runs.
- (3) Extraction (solvent, technique). Extraction efficiency, if relevant (radio- or cross-validation) data may be provided in a separate report at the discretion of the petitioner.
- (4) Addition of internal standard (if any).
- (5) Clean-up.
- (6) Hydrolysis (if any).
- (7) Derivatisation (if any).
- (8) Flowchart of analytical procedure.
- (9) Chromatographic conditions/mobile phase composition if chromatographic separation is used.
- (10) Stability of standard solutions and extracts.

(iii) Instrumentation.

- (1) Description (e.g. make/model, type/selectivity of detectors, columns (packing materials, size), carrier gases, etc.).
- (2) Operating conditions (e.g., flow rates, temperatures, voltage, chromatography conditions, etc.).
- (3) Calibration procedures.
- (4) A mass spectrum (and product ion spectrum for MS/MS) (if applicable).

- (iv) Interferences. Describe any interferences in chromatograms, such as:
 - (1) Sample matrices (matrix effects).
 - (2) Other pesticides.
 - (3) Solvents.
 - (4) Laboratory ware.
 - (v) Confirmatory techniques (if required).
 - (vi) Describe modifications or potential problems, if any, in the analytical method (detail circumstances and corrective action to be taken).
 - (vii) Calculations. Describe in a stepwise fashion:
 - (1) Molecular conversion factors (e.g. parent – metabolite).
 - (2) Concentration in extract, based on calibration equation.
 - (3) Concentration in sample equivalents
 - (4) Calculation of recovery
 - (viii) Other. Any and all additional information considered appropriate and relevant to provide a complete and thorough description of residue analytical methodology and the means of calculating the residue results.
- C. Performance. Describe performance of method.
- (i) Recovery (expected mean and range of recoveries). Include the individual recovery values, average recoveries, and relative standard deviation thereof for each component of the residue of concern at each fortification level in each commodity tested during the method validation.
 - (ii) Precision.
 - (iii) Limits of detection (if needed) and quantitation (provide definition).
 - (iv) Calibration information (linearity plots, equation, r value, weighting, raw data, range (as standard concentration (e.g. ng/mL) and sample equivalents (e.g. mg/kg))).
 - (v) Limitations.
- D. Representative Chromatograms. At a minimum, the following representative chromatograms should be included in the study report:
- (i) Matrix blank.
 - (ii) Lowest calibration standard.
 - (iii) Lowest fortification level
 - (iv) Incurred residue samples (where applicable).
- E. Conclusions. Summarize applicability of analytical procedure for measuring specific test compounds in various test substrates, availability of equipment, interferences, stability, etc.

7 REFERENCES

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Annex A. List of food and feed commodities of plant origin for pre-registration and monitoring method validation and attribution to matrix groups

In the following table, a colour coding was used to designate the following matrix groups:

high acid content
high water content
high oil content
dry
dry high sugar content
difficult

Explanations for Table 7:

- Column “R/P” distinguishes between raw commodities (“R”) and processed commodities (“P”). In this context, “processing” includes also the process of separating peel from pulp.
- While Table 7 is organised according to Codex commodity groups for all raw commodities, it was decided not to follow the Codex classifications for processed commodities. Those were rather listed directly beneath the respective raw commodity instead of using the Codex groups 055-059, 065, 067-071. In general, the latest version of the Codex classification has been used (13).
- Directly harvested feed items can be found in the Codex Commodity Groups 050, 051 and 052, which are dedicated to feed-only products. The parts consumed by humans and harvested from the same crops are contained in other Codex Commodity Groups (for example cereal grains in group 020). Feed items obtained as processing by-products are listed together with the raw food commodities and the entry “feed item” is provided in the column “Comments”. For these entries reference is made to the latest version of the OECD Guidance document on residues in livestock (14).
- It should be noted that the table is not complete and further commodities (raw and processed) might be used that require attribution to one of the matrix groups.
- Rules for attribution of further commodities:
 - Juices, pulps, wet pomaces, cooked or canned commodities not mentioned in the table should be attributed to the same matrix group as the respective raw commodity.

- Commodities having undergone a drying process should be attributed to the dry (e.g. dried pulses or dried vegetables) or dry high sugar content (e.g. dried fruits) matrix group, even if they still contain a certain percentage of water. An exception is made for those with still high oil content after drying, which are attributed to the high oil matrix group.
- pH values vary between commodities, but even between different species of the same commodity. Commodities belonging to or produced from “citrus fruits” or “berries and small fruit” are likely to have a comparably high acid content as the other members of the group should be attributed accordingly. A pH value below 4 may serve as a justification to attribute a commodity to the high acid content matrix group.
- Commodities in the high oil content matrix group typically have a fat/oil content of 20-70 %. “Oil” also includes “essential oils”. Commodities not explicitly mentioned in Table 7, but belonging to “tree nuts”, “oilseeds” or “oilfruits” and commodities used for oil production are always attributed to the high oil content matrix group.
- Commodities not considered as high acid or high oil and which have not been dried are normally attributed to the high water content matrix group.
- Only very few composite foods are mentioned in the table such as beer. Though it is noted that jam, jelly and marmalade have been frequently produced in processing studies, they are composite foods prepared according to various recipes. Based on the particular composition, the appropriate assignment to a matrix group should be justified on a case-by-case basis.
- Dry processed commodities derived from high acid raw commodities require specific attention when samples are prepared for analysis: while being “dry” during storage, in solution the acidic character could become relevant again and impact the analytical method.

Table 7: List of food and feed commodities of plant origin (raw and processed) and attribution to commodity groups

Codex Commodity group	Commodity (examples from the group including typical processed commodities)	Matrix group	R/P	Comments
001 Citrus fruits	Lemon	high acid content	R	
	Lime	high acid content	R	
	Kumquat	high acid content	R	
	Mandarin	high acid content	R	
	Orange	high acid content	R	
	Grapefruit	high acid content	R	
	Pomelo	high acid content	R	
	Citrus fruit, canned	high acid content	P	
	Citrus juice	high acid content	P	
	Citrus oil	high oil content	P	
	Citrus peel (see also group 028)	high acid content	P	
	Citrus peel, dried	dry	P	
	Citrus pomace, dry	dry	P	feed item
	Citrus pomace, wet	high acid content	P	feed item
	Citrus pulp	high acid content	P	
002 Pome fruits	Apple	high water content	R	
	Pear	high water content	R	
	Quince	high water content	R	

Codex Commodity group	Commodity (examples from the group including typical processed commodities)	Matrix group	R/P	Comments
	Medlar	high water content	R	
	Loquat/Japanese medlar	high water content	R	
	Kaki/Japanese persimmon	high water content	R	
	Pome fruit, canned	high water content	P	
	Pome fruit, cooked	high water content	P	
	Pome fruit, dried	dry high sugar content	P	storage stability: dry
	Pome fruit juice	high water content	P	
	Pome fruit puree	high water content	P	
	Pome fruit, wet pomace	high water content	P	feed item
003 Stone fruits	Cherry	high water content	R	
	Plum	high water content	R	
	Peach	high water content	R	
	Apricot	high water content	R	
	Stone fruit, canned	high water content	P	
	Stone fruit, cooked	high water content	P	
	Stone fruit, dried	dry high sugar content	P	storage stability: dry
	Stone fruit juice	high water content	P	
	Stone fruit pulp	high water content	P	
	Stone fruit puree	high water content	P	
	Stone fruit, wet pomace	high water content	P	
004 Berries and other small fruits	Blackberry	high acid content	R	
	Dewberry	high acid content	R	
	Raspberry	high acid content	R	
	Blueberry	high acid content	R	
	Currant	high acid content	R	
	Gooseberry	high acid content	R	
	Elderberries	high acid content	R	
	Cranberry	high acid content	R	
	Strawberry	high acid content	R	
	Berries, canned	high acid content	P	
	Berries, cooked	high acid content	P	
	Berries, dried	dry high sugar content	P	storage stability: dry
	Berry juice	high acid content	P	
	Berry puree	high acid content	P	
004 Berries and other small fruits: Grapes	Table grape (berries)	high acid content	R	
	Wine grape (berries)	high acid content	R	
	Grape pomace, wet	high acid content	P	feed item
	Grape juice	high acid content	P	
	Must	high acid content	P	
	Raisin	dry high sugar content	P	storage stability: dry
	Grape spirit	high water content	P	
	Wine	high acid content	P	
	Jambolan	high water content	R	

Codex Commodity group	Commodity (examples from the group including typical processed commodities)	Matrix group	R/P	Comments
005 Assorted tropical and subtropical fruits - edible peel	Table olive	high oil content	R	
	Table olive, fermented	high oil content	P	
	Carambola	high acid content	R	
	Carob	difficult	R	high matrix load, storage stability: dry
	Fig	high water content	R	
	Fig, dried	dry high sugar content	P	storage stability: dry
	Guava	high water content	R	
	Date	high water content	R	
	Date, dried	dry high sugar content	P	storage stability: dry
006 Assorted tropical and subtropical fruits – inedible peel	Litchi	high water content	R	
	Logan	high water content	R	
	Avocado	high oil content	R	
	Banana	high water content	R	
	Mango	high water content	R	
	Papaya	high water content	R	
	American persimmon/Virginia kaki	high water content	R	
	Granate apple/pomegranate	high acid content	R	
	Star apple/cainito	high water content	R	
	Breadfruit	high water content	R	
	Cherimoya	high water content	R	
	Durian	high water content	R	
	Pineapple	high acid content	R	
	Pineapple, process waste	high acid content	P	feed item
	Soursop/guanabana	high water content	R	
	Prickly pear/cactus fruit	high water content	R	
	Kiwi fruit	high acid content	R	
	Passion fruit/maracuja	high acid content	R	
	Tropical and subtropical fruit with inedible peel, dried	dry high sugar content	P	storage stability: dry
009 Bulb vegetables	Garlic	high water content	R	fresh & dry garlic cloves
	Garlic, peeled	high water content	P	
	Onion	high water content	R	
	Onion, peeled	high water content	P	
	Shallot	high water content	R	
	Shallot, peeled	high water content	P	
	Spring onion/green onion and Welsh onion	high water content	R	
	Leek	high water content	R	
	Bulb vegetable, dried	dry	P	
	Broccoli	high water content	R	

Codex Commodity group	Commodity (examples from the group including typical processed commodities)	Matrix group	R/P	Comments
010 Brassica vegetables (except Brassica leafy vegetables)	Cauliflower	high water content	R	
	Brussels sprout	high water content	R	
	Head cabbage	high water content	R	
	Sauerkraut	high acid content	P	fermented white cabbage
	Sauerkraut juice	high acid content	P	
	Chinese cabbage/pe-tsai	high water content	R	
	Kohlrabi	high water content	R	
	Brassica vegetable, cooked	high water content	P	
011 Fruiting vegetables, Cucurbits	Cucumber	high water content	R	
	Gherkin	high water content	R	
	Gherkin, canned	high acid content	P	
	Gherkin, fermented	high acid content	P	
	Summer squash/zucchini	high water content	R	
	Summer squash/zucchini, cooked	high water content	P	
	Melon	high water content	R	
	Melon, pulp	high water content	P	
	Pumpkin	high water content	R	
	Pumpkin, cooked	high water content	P	
	Pumpkin, wet pomace	high water content	P	
	Pumpkin, pulp	high water content	P	
	Pumpkin, puree	high water content	P	
	Watermelon	high water content	R	
	Watermelon, pulp	high water content	P	
012 Fruiting vegetables, other than Cucurbits	Tomato	high water content	R	
	Tomato, canned	high water content	P	
	Tomato, dried	dry	P	
	Tomato juice	high water content	P	
	Tomato paste	high water content	P	
	Tomato puree	high water content	P	
	Tomato, wet pomace	high water content	P	feed item
	Tomato, dry pomace	dry	P	feed item
	Okra/lady's finger	high water content	R	
	Sweet pepper/bell pepper	high water content	R	
	Chili pepper	high water content	R	
	Aubergine/egg plant	high water content	R	
	Fruiting vegetables other than Cucurbits, canned	high water content	P	
	Fruiting vegetables other than Cucurbits, cooked	high water content	P	
	Fruiting vegetables other than Cucurbits, dried	dry	P	

Codex Commodity group	Commodity (examples from the group including typical processed commodities)	Matrix group	R/P	Comments
013 Leafy vegetables (including Brassica leafy vegetables)	Chard/beet leaves	high water content	R	
	Escarole/broad-leaved endive	high water content	R	
	Lamb's lettuce/corn salad	high water content	R	
	Lettuce	high water content	R	
	Purslane	high water content	R	
	Spinach	high water content	R	
	Cress	high water content	R	
	Kale	high water content	R	
	Mustard greens	high water content	R	
	Roman rocket/rucola	high water content	R	
	Grape leaves	high water content	R	
	Water cress	high water content	R	
	Witloof	high water content	R	
	Baby leaves	high water content	R	baby crops listed in the leafy vegetable group and harvested up to 8 true leaf stage
	Leafy vegetable, canned	high water content	P	
	Leafy vegetable, cooked	high water content	P	
014 Legume vegetables	Bean (green, with pods)	high water content	R	
	Bean (succulent, without pods)	high water content	R	
	Pea (green, with pods)	high water content	R	
	Pea (succulent, without pods)	high water content	R	
	Legume vegetable, canned	high water content	P	
	Legume vegetable, cooked	high water content	P	
015 Pulses	Bean (dry)	dry	R	
	Cowpea (dry)	dry	R	
	Lupin (dry)	dry	R	
	Lupin, extracted meal	dry	P	feed item
	Pea (dry)	dry	R	
	Lentil (dry)	dry	R	
	Pulses (dry seeds), canned	high water content	P	
	Pulses (dry seeds), cooked	high water content	P	
015 Pulses: Soya bean	Soya bean, dry	high oil content	R	
	Soya bean, extracted meal	dry	P	feed item
	Soya bean, flour	dry	P	
	Soya bean, hulls	dry	P	feed item
	Soya oil	high oil content	P	
	Soya drink	high water content	P	
	Soya sauce	high water content	P	
	Tofu	high water content	P	
016 Root and tuber vegetables	Beetroot	high water content	R	
	Carrot	high water content	R	

Codex Commodity group	Commodity (examples from the group including typical processed commodities)	Matrix group	R/P	Comments
	Celeriac/turnip rooted celery	high water content	R	
	Horseradish	high water content	R	
	Parsley, turnip- rooted	high water content	R	
	Parsnip	high water content	R	
	Radish	high water content	R	
	Salsify	high water content	R	
	Swede/rutabaga	high water content	R	
	Turnip	high water content	R	
	Arrowroot	high water content	R	
	Cassava root/manioc	high water content	R	
	Jerusalem artichoke	high water content	R	
	Yam	high water content	R	
	Root and tuber vegetable juice	high water content	P	
	Root and tuber vegetable, wet pomace	high water content	P	feed item
	Root and tuber vegetable, puree	high water content	P	
	Root and tuber vegetable, cooked/canned	high water content	P	
	Root and tuber vegetable, dried	dry	P	
016 Root and tuber vegetables: Sugar beet and chicory root	Sugar beet root	high water content	R	
	Sugar beet, ensiled pulp	high water content	P	feed item
	Sugar beet, molasses	high water content	P	feed item and intermediate in food production
	Sugar beet pulp, dried	high water content	P	feed item
	Sugar from sugar beet, refined	dry high sugar content	P	storage stability: dry
	Chicory root	high water content	R	
	Chicory root, milled	dry	P	
	Chicory root, roasted	dry	P	
016 Root and tuber vegetables: Potatoes	Potato	high water content	R	
	Sweet potato	high water content	R	
	Potato tuber, peeled	high water content	P	
	Potato tuber, canned	high water content	P	
	Potato tuber, cooked (with or w/o peel, with or w/o microwave)	high water content	P	
	Potato tuber, baked (with or w/o peel)	high water content	P	
	Potato tuber, steamed (with or w/o peel)	high water content	P	
	Potato peel	high water content	P	feed item
	Potato tuber, dried	dry	P	
	Potato flakes/granules	dry	P	

Codex Commodity group	Commodity (examples from the group including typical processed commodities)	Matrix group	R/P	Comments
	Potato peel, dried	dry	P	feed item
	Potato pulp, dried	dry	P	feed item
	Potato starch	dry	P	
	Potato crisps (with or w/o peel)	high oil content	P	composite: contains oil
	Potato tuber, fried or deep-fried (with or w/o peel)	high oil content	P	composite: contains oil
017 Stalk and stem vegetables	Cardoon stems	high water content	R	
	Celery stalk	high water content	R	
	Florence fennel bulb	high water content	R	
	Rhubarb stems	high acid content	R	
	Rhubarb stems, cooked	high acid content	P	
	Asparagus stems	high water content	R	
	Bamboo shoot	high water content	R	
	Globe artichoke flower head	high water content	R	
	Palm heart stems	high water content	R	
	Stalk and stem vegetable (except rhubarb), canned	high water content	P	
	Stalk and stem vegetable (except rhubarb), cooked	high water content	P	
018 Edible Fungi	Cultivated fungi	high water content	R	
	Wild fungi	high water content	R	
	Fungi, canned	high water content	P	
	Fungi, dried	dry	P	
020 Cereal grains	Amaranth	dry	R	
	Quinoa	dry	R	
	Rye grain	dry	R	
	Rye, bran	dry	P	
	Rye, flour	dry	P	
	Wheat grain	dry	R	
	Wheat, bran	dry	P	
	Wheat, distiller's grain, wet	high water content	P	feed item
	Wheat, distiller's grain, dried	dry	P	feed item
	Wheat, flour	dry	P	
	Wheat, germs	dry	P	
	Wheat, gluten	dry	P	
	Wheat, gluten feed meal	dry	P	feed item
	Wheat, grit	dry	P	
	Wheat, malt	dry	P	
	Wheat, starch	dry	P	
	Barley grain	dry	R	
	Barley, beer	high water content	P	composite: contains malt and hops
	Barley, bran	dry	P	

Codex Commodity group	Commodity (examples from the group including typical processed commodities)	Matrix group	R/P	Comments
	Barley, brewer's grain	high water content	P	feed item
	Barley, distiller's grain, wet	high water content	P	feed item
	Barley, distiller's grain, dried	dry	P	feed item
	Barley, flour	dry	P	
	Barley, malt	dry	P	
	Barley, malt sprouts	high water content	P	
	Pot/pearl barley	dry	P	
	Barley, spirit	high water content	P	
	Buckwheat	dry	R	
	Oat grain	dry	R	
	Oat, bran	dry	P	
	Oat, flour	dry	P	
	Oat, hulls	dry	P	
	Rolled oat	dry	P	
	Rice grain	dry	R	
	Rice, bran	dry	P	feed item
	Rice, flour	dry	P	
	Rice, hulls	dry	P	feed item
	Rice, polished	dry	P	
	Rice, spirit	high water content	P	
	Common millet/proso millet grain	dry	R	
	Sorghum grain	dry	R	
	Sorghum, flour	dry	P	
	Sorghum, starch	dry	P	
	Maize/corn, grain	dry	R	
	Maize, distiller's grain	high water content	P	feed item
	Maize, extracted meal	dry	P	feed item
	Maize, flour	dry	P	
	Maize, germs	high oil content	P	
	Maize, gluten meal	dry	P	feed item
	Maize, grits	dry	P	
	Maize, oil	high oil content	P	
	Maize, starch	dry	P	
	Sweet corn	high water content	R	
	Sweet corn, cooked	high water content	P	
	Sweet corn, canned	high water content	P	
021 Grasses for sugar or syrup production	Sugar cane	high water content	R	
	Sugar cane, molasses	high water content	P	feed item and intermediate in food production
	Sugar from sugar cane, raw	dry high sugar content	P	storage stability: dry
	Sugar from sugar cane, refined	dry high sugar content	P	storage stability: dry

Codex Commodity group	Commodity (examples from the group including typical processed commodities)	Matrix group	R/P	Comments
022 Tree nuts	Almond nutmeat	high oil content	R	
	Almond, hulls	dry	P	feed item
	Brazil nut nutmeat	high oil content	R	
	Cashew nut nutmeat	high oil content	R	
	Chestnut nutmeat	high water content	R	
	Coconut nutmeat	high oil content	R	
	Coconut, dried meat (copra)	high oil content	P	
	Coconut, meal	dry	P	feed item
	Hazelnut nutmeat	high oil content	R	
	Macadamia nut nutmeat	high oil content	R	
	Pecan nutmeat	high oil content	R	
	Pine nut nutmeat	high oil content	R	
	Pistachio nut nutmeat	high oil content	R	
	Walnut nutmeat	high oil content	R	
023 Oilseeds and oilfruits	Borage seed	high oil content	R	
	Gold of pleasure seed	high oil content	R	
	Linseed	high oil content	R	
	Mustard seed	high oil content	R	
	Poppy seed	high oil content	R	
	Rapeseed/canola seed	high oil content	R	
	Sesame seed	high oil content	R	
	Safflower seed	high oil content	R	
	Sunflower seed	high oil content	R	
	Cotton seed	high oil content	R	
	Castor bean	high oil content	R	
	Grape seed	high oil content	P	
	Hemp seed	high oil content	R	
	Kapok seed	high oil content	R	
	Palm nut/palm kernel nutmeat	high oil content	R	
	Palm kernel meal	dry	P	feed item
	Peanut/groundnut	high oil content	R	
	Peanut butter	high oil content	P	
	Peanut, roasted	high oil content	P	
	Pumpkin seed	high oil content	R	
	Oilseed, extracted meal	dry	P	feed item
	Oilseed, hulls	dry	P	feed item
	Oilseed oil	high oil content	P	
	Olive for oil production	high oil content	R	
	Olive oil	high oil content	P	
	Olive, wet pomace	high oil content	P	
	Oil palm fruit	high oil content	R	
	Oil palm fruit, extracted meal	dry	P	feed item
	Palm oil	high oil content	P	

Codex Commodity group	Commodity (examples from the group including typical processed commodities)	Matrix group	R/P	Comments
024 Seeds for beverages and sweets	Cacao bean (fermented or dried)	difficult	R	high matrix load, storage stability: high oil content
	Cacao bean, roasted	difficult	P	high matrix load, storage stability: high oil content
	Cacao butter	difficult	P	high matrix load, storage stability: high oil content
	Cacao powder	difficult	P	high matrix load, storage stability: high oil content
	Coffee bean (green)	difficult	R	high matrix load, storage stability: high oil content
	Coffee bean, roasted	difficult	P	high matrix load, storage stability: high oil content
	Instant coffee	difficult	P	high matrix load, storage stability: high oil content
	Coffee, infusion	high water content	P	
027 Herbs	Fresh herbs and edible flowers	high water content	R	
	Herbs and edible flowers, dried	difficult	P	high matrix load, storage stability: dry
	Peppermint oil	high oil content	P	
028 Spices	Seed spices	difficult	R	high matrix load, storage stability: dry
	Fruit or berry spices	difficult	R	high matrix load, storage stability: dry
	Bark spices	difficult	R	high matrix load, storage stability: dry
	Root or rhizome spices, dried	difficult	P	high matrix load, storage stability: dry
	Root or rhizome spices, fresh (e.g. ginger)	high water content	R	
	Bud spices, dried	difficult	R	high matrix load, storage stability: dry
	Bud spices, pickled (e.g. capers)	high acid content	P	
	Flower or stigma spices	difficult	R	high matrix load, storage stability: dry
	Aril spices	difficult	R	high matrix load, storage stability: dry

Codex Commodity group	Commodity (examples from the group including typical processed commodities)	Matrix group	R/P	Comments
	Citrus peel, dried	dry	P	
	Dried chili peppers	dry	P	
050 Legume feed products	Forage (for example alfalfa, clover, vetch, bean)	high water content	R	feed item
	Vine (for example pea)	high water content	R	feed item
	Silage (for example alfalfa, clover, vetch, beans, peas)	high water content	P	feed item
	Hay (for example alfalfa, clover, vetch, beans, peas)	dry	R	feed item
	Straw (for example alfalfa, clover, vetch, beans, peas)	dry	R	feed item
051 Cereal grains (including pseudocereals) and grass feed products	Forage (for example barley, grass, maize, millet, oat, rice, rye, triticale, wheat)	high water content	R	feed item
	Silage (for example barley, grass, maize, millet, oat, rice, rye, triticale, wheat)	high water content	P	feed item
	Hay (for example barley, grass, maize, millet, oat, rye, triticale, wheat)	dry	R	feed item
	Straw (for example barley, grass, maize, millet, oat, rye, triticale, wheat)	dry	R	feed item
	Stover (for example maize)	dry	R	feed item
052 Miscellaneous Feed Products	Beet leaves or tops (for example fodder beet, sugar beet, turnip)	high water content	R	feed item
	Fodder beet root	high water content	R	feed item
066 Teas	Tea (Camellia sinensis, dried leaves, stalks and flowers, fermented or otherwise treated)	difficult	R	high matrix load, storage stability: dry
	Tea, green, dried	difficult	R	high matrix load, storage stability: dry
	Instant tea	difficult	P	high matrix load, storage stability: dry
	Tea, infusion	high water content	P	
	Herbal infusion (dried, flowers)	difficult	R	high matrix load, storage stability: dry
	Herbal infusion (dried, leaves and herbs)	difficult	R	high matrix load, storage stability: dry
	Herbal infusion (dried, roots)	difficult	R	high matrix load, storage stability: dry
	Herbal infusion, infusion	high water content	P	

Codex Commodity group	Commodity (examples from the group including typical processed commodities)	Matrix group	R/P	Comments
079 Miscellaneous processed foods of plant origin	Hops, green cones	difficult	R	high matrix load, storage stability: high water content
	Hops (dried), hop pellets and unconcentrated powder, hop extract	difficult	R	high matrix load, storage stability: dry
	Beer	high water content	P	composite: contains malt and hops
	Hop extract (aqueous solutions)	difficult	P	high matrix load, storage stability: high water content
	Spent hops	difficult	P	high matrix load, storage stability: high water content feed item