CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

Viale delle Terme di Caracalla, 00153 Rome, Italy - Tel: (+39) 06 57051 - Fax: (+39) 06 5705 4593 - E-mail: codex@fao.org - www.codexalimentarius.org

Agenda Item 8 **CX/PR 15/47/10**
February 2015

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON PESTICIDE RESIDUES

47th Session
Beijing, P.R. China, 13-18 April, 2015

PROPOSED DRAFT GUIDANCE ON PERFORMANCE CRITERIA FOR METHODS OF ANALYSIS FOR THE DETERMINATION OF PESTICIDE RESIDUES

(Prepared by the Electronic Working Group led by the United States of America and co-chaired by China and India)

(AT STEP 4)

Codex Members and Observers wishing to submit comments at **Step 3** on this document (**see Appendix I**), including possible implications for their economic interests, should do so in conformity with the *Uniform Procedure for the Elaboration of Codex Standards and Related Texts* (Codex Alimentarius Commission Procedural Manual) before **20 March 2015**. Comments should be directed:	
to: Mrs Lifang DUAN Institute for the Control of Agrochemicals Ministry of Agriculture Room 906, No. 18 building Maizidian Street, Chaoyang District, Beijing, 100125, P.R. China Email: ccpr@agri.gov.cn	with a copy to: Secretariat, Codex Alimentarius Commission, Joint FAO/WHO Food Standards Programme, Viale delle Terme di Caracalla, 00153 Rome, Italy Email:codex@fao.org

BACKGROUND

1. At the 46th session of the Committee on Pesticide Residues (May 2014), the Committee noted that additional work was still necessary on some areas of the document and therefore agreed to re-establish the electronic working group (eWG) to further revise the ***Guidance on Performance Criteria for Methods of Analysis for the Determination of Pesticide Residues*** taking into consideration relevant documents from the Committee on Residues of Veterinary Drugs in Foods (CCRVDF) and the Committee on Methods of Analysis and Sampling (CCMAS). This eWG was to be chaired by the United States of America and co-chaired by China and India.[1]

2. On September 1, 2014, a broadcast e-mail was sent by the Codex Secretariat to all member countries to provide name(s) of participants to this eWG. As a result, there were approximately 50 representatives from 25 countries who actively participated in two rounds of reviews that resulted in the final document submitted here. The list of participants is provided in Appendix II.

3. The goals were to provide the Codex community with a guidance document outlining methods and performance criteria on analytical multi-residue analysis testing for pesticide residues in food. Internationally there are many well written and comprehensive documents readily available on methodologies for analytical testing for pesticides and veterinary drug residues from ISO, IUPAC, CODEX, FAO, SANCO and multiple US government agencies (EPA, FDA, USDA, etc.). This guidance document was drafted by Codex to provide an internationally accepted framework based on ISO and IUPAC standards, rather than biasing protocols written to standards for an individual country or region. These guidelines are a general overview to allow flexibility for individual countries, while adhering and maintaining to the highest standards of scientific integrity.

[1] REP14/PR, paras 153-154.

4. The submitted CODEX **Guidelines on Performance Criteria Specific for Methods of Analysis for the Determination of Pesticide Residues in Food** (CCPR 47) takes into consideration that not all subscribing countries have the same financial resources, analytical laboratories, or scientific infrastructure. Comments submitted to the working group by all member countries (including those in reply to CL 2014/16-PR, Part B) were carefully reviewed and considered in this final draft as to allow this document to have relevance for current international conditions for laboratories in all ascribing CODEX countries. Comments to reference specific country or regional documents were reviewed, but referencing and preferential citing of methodologies was attributed to CODEX, ISO or IUPAC documents, when appropriate and available. The eWG also considered the caveats befalling technical translation into so many different languages that may result in subtle semantic differences and can cause major discrepancies for interpreting analytical results. Because the interpretation of results is critical for International trade with interpretation errors disrupting trade and resulting in economic losses, other documents are cited in this guidance to help support this framework.

5. The eWG wishes to thank all members who submitted comments; these perspectives and edits were greatly valued and appreciated, contributing to a more complete and high quality final document.

APPENDIX I

PROPOSED DRAFT GUIDELINES ON PERFORMANCE CRITERIA SPECIFIC FOR METHODS OF ANALYSIS FOR THE DETERMINATION OF PESTICIDE RESIDUES IN FOOD

CONTENTS:

PURPOSE

1. The purpose of this guidance document is to describe the performance criteria of methods to analyze pesticide residues in foods. It addresses the characteristics/parameters to provide scientifically acceptable confidence in the analytical methods to produce accurate/precise results and to reliably evaluate pesticide residues for either domestic monitoring and/or international trade.

SCOPE

2. This document is applicable to single and multiresidue methods (MRMs) to analyze target compounds in food commodities, including parent pesticide residues and/or their metabolites and degradates in food commodities, as per the residue definition.

3. In this document, a MRM is defined as a method which can determine three or more analytes in the same chemical class or in more than one class of pesticide. This guidance covers qualitative (screening, identification, confirmation) and quantitative analyses, each having their own specific method performance requirements. For qualitative purposes, method validation involves analysis of ≥20 each of diverse matrix blanks and matrix spikes at the reporting level to minimally assess rates of false positives and negatives.

PRINCIPLES FOR THE SELECTION AND VALIDATION OF METHODS

Identification of Methods Requirements

4. The intended purpose of the method is usually defined in a statement of scope which defines the analytes (residues), the matrices, and the concentration range. It also states whether the method is intended for screening, quantification, identification, and/or confirmation of analytes.

5. The MRL is expressed in terms of the “residue definition”, which may include the parent compound, a major metabolite, a sum of parent and/or metabolites, or a reaction product formed from the residues during analysis. Ideally, residue analytical methods should be able to measure all components of the residue definition.

6. Selection of methods is discussed in ENV/JM/MOMO(2007), “Guidance Document on Pesticide Residue Analytical Methods OECD”.

Implementing other Codex Alimentarius Commission Guidelines

7. The Codex Alimentarius Commission (CAC) has issued a guideline for laboratories involved in the import/export testing of foods which recommends that such laboratories should:

    a. use internal quality control procedures, such as those described in the "Harmonized Guidelines for Internal Quality Control in Analytical Chemistry Laboratories";

    b. participate in appropriate proficiency testing schemes for food analysis which confirm to the requirement laid out in "The International Harmonized Protocol for Proficiency Testing of (Chemical) Analytical Laboratories";

    c. comply with the general criteria for testing laboratories provided in ISO/IEC Guide 17025:2005 "General Requirements for the Competence of Calibration and Testing Laboratories"; and

    d. whenever available, use methods which have been validated according to principles provided by the CAC.

8. The methods should be used within the internationally accepted, approved, and recognized laboratory Quality Management System, following a guide such as ISO/IEC Guide 17025, to be consistent with the principles in the document for quality assurance (QA) and quality control (QC) referenced above. The on-going performance must be monitored through the Quality Management System in place in the laboratory.

Method Validation

9. The process of method validation is intended to demonstrate that a method is *fit-for-purpose*. This means that when a test is performed by a properly trained analyst using the specified equipment and materials and following the procedures described in the method, accurate and consistent results can be obtained within specified statistical limits for sample analysis. The validation should specify the analyte (identity and concentration), account for the matrix effects, provide a statistical characterization of the recovery results, and indicate if the rates of false positives and negatives are minimally acceptable. When the method protocol is followed using suitable analytical standards, results within the established performance limits should be obtained on the same or equivalent sample material by a trained analyst in any experienced residue testing laboratory. To ensure that validation of the method remains appropriate over time, the method should be continuously assessed using routine proficiency testing and appropriate quality control samples.

SUMMARY OF PERFORMANCE PARAMETERS TO BE CHARACTERISED AND DEFINED FOR ANALYTICAL METHODS

10. The general requirements for the individual performance characteristics of a method are summarized below from IUPAC's "Harmonized guidelines for single-laboratory validation of methods of analysis".

A. APPLICABILITY

11. After validation, the analysis documentation should provide, in addition to performance specifications, the following information:

    a. identity of the analyte, including speciation where appropriate (e.g., "cypermethrin, malathion, etc.");

    b. concentration range covered by the validation (e.g., "0.01–10mg/kg");

    c. specification of the range of matrices of the test material covered by the validation (e.g., "cucurbits, root vegetables, citrus, etc.");

    d. protocol, describing the equipment, reagents, procedure (including permissible variation in specified instructions, e.g., "heat at 100 ± 5°C for 30 ± 5 min"), calibration and quality procedures, any special safety precautions required, and intended application and critical uncertainty requirements;

    e. for many purposes, the measurement uncertainty (MU) does not need to be formally calculated;

    f. and if required, a quantitative result should be reported together with the expanded MU as follows: Result = x ± U (units), with x representing the measured value.

B. SELECTIVITY

12. Ideally, selectivity should be evaluated to demonstrate that no interferences occur that detrimentally affect the analysis. It is particularly important to check against common interferents on principal chemicals that are likely to respond to the test. It may be impracticable to consider or test every potential interferent; where that is the case, it is recommended that the likely most common interferents are checked. To minimally estimate rates of false positives and negatives during method validation, analyze ≥20 each of diverse matrix blanks (not from the same source) and spiked matrices at the analyte reporting level (e.g., 50% of the MRL). For methods of identification (see par. 46), no false positives are permissible, and false negatives should be ≤5%. Validations of screening methods (presence/absence analyses) are discussed in paragraphs 34-36. As a general principle, selectivity should be sufficiently good for any interference to be inconsequential. In many types of analysis, selectivity is essentially a qualitative assessment based on the significance of suitable tests for interferents.

C. CALIBRATION AND LINEARITY

13. With the exception of gross errors in preparation of calibration materials, calibration errors are usually (but not always) a minor component of the total uncertainty, and can be safely assigned into other categories. For example, random errors resulting from calibration are part of the run bias that is assessed as a whole, while systematic errors from that source may appear as laboratory bias, likewise assessed as a whole. Nevertheless, there are some characteristics of calibration that are useful to know at the outset of method validation, because they affect the strategy for the optimal development of the procedure. In this class are such questions as whether the calibration function plausibly (a) is linear, (b) passes through the origin, and (c) is unaffected by the matrix of the test material. The procedures described here relate to calibration studies in validation, which are necessarily more involved than calibration undertaken during routine analysis.

14. In general, the use of weighted-linear regression or weighted quadratic function is recommended rather than simply linear regression for the low part per billion (µg/kg) concentration level determination.

D. Linearity and Intercept

15. Linearity can be tested by examination of a plot of residuals produced by linear regression of the responses on the concentrations in an appropriate calibration set. Any curved pattern suggests *lack of fit* due to a nonlinear calibration function. If it is the case, other function such as quadratic should be tested and applied. Despite its current widespread use as an indication of quality of fit, the correlation coefficient is misleading and inappropriate as a test for linearity and should not be used.

16. Replicate measurements are needed to provide an estimate of pure error if there is no independent estimate. In the absence of specific guidance, the following should apply (for univariate linear calibration):

    a. there should be five or more calibration standards;

    b. the calibration standards should be evenly spaced over the concentration range of interest;

    c. the range should encompass 0–150% or 50–150% of the concentration likely to be encountered, depending on which of these is the more suitable; and

    d. the calibration standards should be run at least in duplicate, and preferably triplicate or more, in a random order, over the whole sequence.

17. The value of the intercept should be as close as possible to zero [e.g. less than 20% of the lowest calibration standard] to avoid errors in calculating sample concentrations at low residue levels.

E. Test for general matrix effect

18. Matrix-matched calibration is commonly used to compensate for matrix effects. Extracts of blank matrix, preferably of the same type as the sample, should be used for calibration. An alternative practical approach to compensate for matrix effects in gas chromatographic (GC) analyses is the use of analyte protectants that are added to both the sample extracts and the calibration solutions in order to equalize the response of pesticides in solvent calibrants and sample extracts. The most effective way to compensate for matrix effects is the use of standard addition or the use of isotopically labeled internal standards (IS). If standard calibration is used, a test for general matrix effects can be made by applying the method of analyte addition to a test solution derived from a typical test material. The test should be done in a way that provides the same final dilution as produced in the normal procedure, and the range of additions should encompass the same range as the procedure-defined calibration validation. If the calibration is linear, the slopes of the usual calibration function and the analyte additions plot can be compared for significant difference. A lack of significance means that there is no detectable general matrix effect. If the calibration is not linear, a more complex method is needed for a significance test, but a visual comparison at equal concentrations will usually suffice. A lack of significance in this test will often mean that the matrix variation effect will also be absent. If desired, total extractability can be measured by comparing the MRM with the official method provided by the registrants.

F. TRUENESS AND RECOVERY

19. Trueness is the closeness of agreement between a test result and the accepted reference value of the property being measured. Trueness is stated quantitatively in terms of “bias”, with smaller bias indicating greater trueness. Bias is typically determined by comparing the response of the method to a reference material with a known value assigned to the material. Significance testing is recommended. Where the uncertainty in the reference value is not negligible, evaluation of the results should consider the reference material uncertainty as well as the statistical variability.

20. Recovery refers to the proportion of analyte remaining at the point of the final determination, following its addition (usually to a blank sample) immediately prior to extraction, generally expressed as a percentage. Routine recovery refers to the determination(s) performed with the analysis of each batch of samples.

G. PRECISION

21. Precision is the closeness of agreement between independent test results obtained under stipulated conditions. It is usually specified in terms of standard deviation or relative standard deviation. The distinction between precision and bias is fundamental, but depends on the level at which the analytical system is viewed. Thus, from the viewpoint of a single determination, any deviation affecting the calibration for the run would be seen as a bias. From the point of view of the analyst reviewing a year’s work, the run bias will be different every day and will act like a random variable with an associated precision, incorporating any stipulated conditions for the estimation of this precision.

22. For single-laboratory validation, two types of precision sets of conditions are relevant: (a) repeatability, which is the variability of measurements within the same analytical sequence, and (b) reproducibility, which is the variability of results among multiple sample sets. It is important that the precision values are representative of likely test conditions. First, the variation in conditions among the runs must represent what would normally happen in the laboratory under routine use of the method. For instance, variations in reagent batches, analysts, and instruments should be representative. Second, the test material used should be typical, in terms of matrix and (ideally) the state of comminution, of the materials likely to be encountered in routine application.

23. Precision often varies with analyte concentration. Typical assumptions are that (i) there is no change in precision with analyte level, or (ii) that the standard deviation is proportional to, or linearly dependent on, analyte level. In both cases, the assumption needs to be checked if the analyte level is expected to vary substantially (*e.g.*, by more than 30% from its central value).

24. Precision data may be obtained for a wide variety of different sets of conditions in addition to the minimum of repeatability and between-run conditions indicated here, and it may be appropriate to acquire additional information. For example, it may be useful to the assessment of results, or for improving the measurement, to have an indication of separate operator and run effects between or within-day effects, or to have an indication of the precision attainable using one or several instruments. A range of different designs and statistical analysis techniques is available, and careful experimental design is strongly recommended in all such studies. The initial validation should be carried at the targeted limit of quantification (LOQ) or reporting limit of the method, and at least one other higher level, for example, 2-10x the targeted LOQ or the MRL.

H Analytical Range

25. The validated range is the interval of analyte concentration within which the method can be regarded as validated. It is important to realize that this range is not necessarily identical to the useful range of the calibration. While the calibration may cover a wide concentration range, the remainder of the validation (and usually much more important part in terms of uncertainty) will cover a more restricted range. In practice, most methods will be validated for at least two levels of concentration. The validated range may be taken as a reasonable extrapolation from these points on the concentration scale. The lowest level should be at or below the current Maximum Residue Levels (MRL) established by the Codex Alimentarius Commission (CXL) and the validation level should cover the existing CXL, or when a CXL does not exist the lowest level may be established by a national regulatory authority (MRLs).

I. LIMIT OF QUANTIFICATION (LOQ)

26. The common accepted definition of LOQ is the concentration at which signal/noise ratio is 10. This reflects 95% confidence (19 out of 20 times) that an analyte at that concentration will be determined. The LOQ is typically only an estimate because determination of the precise LOQ takes many analyses of spiked samples and matrix blanks to accurately determine signal/noise, which is typically a fruitless exercise because the LOQ changes from day-to-day depending on the state of the instrument. Some validation guidelines require that the LOQ be verified to meet method performance criteria via spiking experiments at the LOQ, but a better term for use of this concept is lowest validated level (LVL). Furthermore, quantification of analytes should not be made below the lowest calibrated level (LCL) in the same analytical sequence. The Signal to noise (S/N) ratio at the LCL must be ≥10 (conc. ≥ LOQ), which can be set as a system suitability check required for each analytical sequence. A quality control matrix spike can also be included in each sequence to verify that the reporting limit is achieved in the analysis (an action level is typically greater than the LCL). In essence, the point of the validation is not to determine the LOQ, but to demonstrate that the lowest reported concentration meeting the need for the analysis will be equal to or greater than the LOQ.

27. It is preferable to try to express the uncertainty of measurement as a function of concentration and compare that function with a criterion of fitness for purpose agreed between the laboratory and the client or end-user of the data.

J. SENSITIVITY

28. The sensitivity of a method is the gradient of the calibration function. As this is usually arbitrary, depending on instrumental settings, it is not useful in validation. (It may be useful in quality assurance procedures, however, to test whether an instrument is performing to a consistent and satisfactory standard.)

K. RUGGEDNESS

29. The ruggedness of an analytical method is the resistance to change in the results produced by an analytical method when minor deviations are made from the experimental conditions described in the procedure. The limits for experimental parameters should be prescribed in the method protocol (although this has not always been done in the past), and such permissible deviations, separately or in any combination, should produce no meaningful change in the results produced. (A “meaningful change” here would imply that the method could not operate within the agreed limits of uncertainty defining *fitness for purpose*.) The aspects of the method that are likely to affect results should be identified, and their influence on method performance evaluated by using ruggedness tests.

30. Examples of the factors that a ruggedness test could address are: changes in the instrument, operator, or brand of reagent; concentration of a reagent; pH of a solution; temperature of a reaction; time allowed for completion of a process, and/or other pertinent factors.

L. FITNESS-FOR-PURPOSE

31. *Fitness-for-purpose* is the extent to which the performance of a method describes the end-user's needs, and matches the criteria agreed between the analyst and the end-user of the data. For instance, the errors in data should not be of a magnitude that would give rise to incorrect decisions more often than a defined small probability, but they should not be so small that the end-user is involved in unnecessary expenditure. *Fitness-for-purpose* criteria could be based on some of the characteristics described here, but ultimately will be expressed in terms of acceptable combined uncertainty.

M. MEASUREMENT UNCERTAINTY

32. The formal approach to measurement uncertainty estimation is a calculated estimate from an equation or mathematical model, around which the true value can be expected to lie within a defined level of probability. The procedures described in method validation are designed to ensure that the equation used to estimate the result, with due allowance for random errors of all kinds, is a valid expression embodying all recognized and significant effects upon the result. "Guidelines on Estimation of Uncertainty of Results" are provided in CAC/GL 59-2006.

PERFORMANCE CHARACTERISTICS OF SCREENING METHODS

33. Screening methods are usually either qualitative or semi-quantitative in nature, with the objective being to discriminate samples which contain no detectable residues above a threshold value ("negatives") from those which may contain residues above that value ("indicated positives"). The validation strategy therefore focuses on establishing a threshold concentration above which results are "potentially positive", determining a statistically based rate for both "false positive" and "false negative" results, testing for interferences and establishing appropriate conditions of use. Screening methods should be checked for their selectivity and sensitivity. They can be based on test kits and their selectivity may be increased when a detection system is used after chromatographic or other separation techniques. Another approach is to use screening methods that involve automated mass spectrometry-based (MS) detection systems, which are very selective. These methods offer laboratories a cost-effective means to extend their analytical scope to analytes which potentially have a low probability of being present in the samples. Analytes that occur more frequently should continue to be sought and measured using validated quantitative MRMs.

34. The selectivity of screening methods should be adequate and must be able to distinguish the presence of the target compound, or group of compounds, from other substances that may be present in the sample material. Selectivity of screening methods is normally not as great as that of a quantitative method. Screening methods often take advantage of a structural feature common to a group or class of compounds and may be based on microbiological growth inhibition, immunoassays, or chromogenic responses which may not unambiguously identify a compound. Mass spectrometric techniques also are used for screening purposes. The selectivity of a screening method may be increased when it is used as a detection system after chromatographic or other separation techniques.

35. The validation of a screening method based on a screening detection limit (SDL) can be focused on detectability. For each commodity group, a minimal validation should involve analysis of at least 20 samples spiked at the estimated SDL. The samples and at least 20 matrix blanks from different sources (more replicates of greater diversity provides better validation) from the commodity group, with a minimum of two different samples for each commodity category and should be representative for the intended scope of the laboratory. Additional validation data can be collected from on-going QC-data and method performance verification during routine analysis. The SDL of the qualitative screening method is the lowest level at which an analyte has been detected (not necessarily meeting the MS-identification criteria) in at least 95% of the samples (e.g. an acceptable false-negative rate of 5%).

PERFORMANCE CHARACTERISTICS OF QUANTITATIVE METHODS

36. Selectivity is of particular importance in defining the performance characteristics of quantitative methods used in regulatory control programs for pesticide residues in foods. Ideally, the method needs to provide a signal response that is free from interferences from other analytes and matrix compounds and may be present in a sample or sample extract. Chromatographic analyses based on peaks, which are not fully resolved, provide less reliable quantitative results. Use of element-specific detectors or different detection wavelengths or mass-selective detectors which are better able to distinguish a particular compound or structure, combined with chromatographic separation, improves the selectivity of quantitative methods.

37. The requirement to recover a range of different pesticide residues in one extraction increases the potential for compromised selectivity in MRMs compared to single analyte methods. Using less selective extraction and clean-up procedures is likely to result in greater co-extracted matrix material in the final extract. The nature and quantities of such co-extracted material can vary markedly based on the particulars and method of the individual sample. Particular care is therefore required when setting criteria for the precision and trueness of MRMs to ensure that quantification will not be affected by interference from other compounds present in the sample matrix.

38. In addition to the selectivity of a method, the ability of the method to provide a reliable quantitative result must be demonstrated (i.e. trueness –see F p.6-and precision –see G p.6).

39. Acceptability criteria for a quantitative analytical method should be demonstrated at both initial and extended validation stages, as being capable of providing acceptable mean recovery values at each spiking level. A minimum of 5 replicates is required (to check the recovery and precision) at the targeted LOQ or reporting limit of the method, and at least one additional higher level, for example, 2-10x the targeted LOQ or the MRL. Where the residue definition includes two or more analytes, then wherever possible, the method should be validated for all analytes. The method-LVL is the lowest spike level of the validation meeting these method performance acceptability criteria. In certain cases and typically with MRMs, recoveries outside this range may be accepted. In situations, where recovery is low but consistent (e.g. demonstrating good precision) and the basis for this is well established (e.g. due to analyte distribution in a partitioning step), a mean recovery below 70% may be acceptable. However, a more accurate method should be used, if practicable. Within-laboratory reproducibility, which may be determined from on-going quality control data in routine analyses, should be ≤ 20%, excluding any contribution due to sample heterogeneity. Acceptable mean recoveries range from 70-120% with a RSD ≤20%.

40. The trueness of a method may be determined by analysis of a certified reference material, by comparison of results with those obtained using another method for which the performance parameters have previously been rigorously established (typically, a collaboratively studied method), or by determination of the recovery of analyte fortified into known blank sample material. The latter determination of trueness as recovery is frequently used in validation of methods for pesticide residues in foods, as both certified reference materials and methods validated by an inter-laboratory trial are often not available. The trueness of a measurement is closely related to random error (repeatability error or within-lab reproducibility error), systematic error (analytical method bias), and analyte recovery (measured as percent recovery). Recovery should be assessed over concentrations covering the analytical range of the method. For interpreting recoveries, it is necessary to recognize that analyte added to a sample may not behave in the same manner as the same biologically incurred analyte (pesticide residue). In many situations, the amount of an extracted (the yield or recovered fraction) incurred residue is less than the total incurred residues present. This may be due to losses during extraction, intra-cellular binding of residues, the presence of conjugates, or other factors that are not fully represented by recovery experiments conducted with analyte-fortified blank matrices. At relatively high concentrations, analytical recoveries are expected to approach 100%. At lower concentrations, particularly with methods involving extensive extraction, isolation, and concentration steps, recoveries may be lower.

41. In general, residues data do not have to be adjusted for recovery when the mean recovery is within the range of 70-120%. In situations where recovery is low but consistent (e.g. demonstrating good precision) and the basis for this is well established (e.g. due to analyte distribution in a partitioning step), a mean recovery below 70% may be acceptable. However, a more accurate method should be used, if practicable. If available and affordable, participation in a proficiency testing program should be done. Recovery corrections should be made consistent with the guidance provided by the CAC/GL 37-2001. It is of over-riding importance that all data, when reported, should (a) be clearly identified as to whether or not a recovery correction has been applied and, if a recovery correction has been applied, (b) include the amount of the correction and the method by which it was derived. This will promote direct comparability of data sets. Correction functions should be established on the basis of appropriate statistical considerations, and documented, archived and made available to the client.

42. Quantitative methods are usually based on a comparison of the response from an analyte in a sample with the response from standards of the analyte in solution or in a matrix at known concentrations. In method development and validation, the calibration curve should first be determined to assess the detector response to standards over the range of concentrations of analytical interest. Possible matrix enhancement or suppression effects of sample co-extractives on the chromatography system or detection system response should be addressed both in gas chromatography (GC) and liquid chromatography (LC) based methods. When appropriate, the detection system may be calibrated using standard solutions in a blank matrix similar to that of the sample to be analyzed (matrix-matched standards), which compensate for matrix effects and if present, acceptable interference. An alternative practical approach to compensate for matrix effects in GC-analyses is the use of analyte protectants that are added to both the sample extracts and the calibration solutions in order to equalize the response of pesticides in solvent calibrants and sample extracts. When no suitable blank commodity is available for the preparation of the matrix-matched standards, the most effective way to compensate for matrix effects is the use of standard addition or the use of isotopically labeled internal standards. The standard addition approach may compensate for matrix effects and for recovery of the analytical procedure, but does not overcome chromatographic interferences. To achieve accurate results using a standard addition approach, it is essential to assure a linear response in the concentration range investigated.

PERFORMANCE CHARACTERISTICS OF METHODS FOR ANALYTE IDENTIFICATION AND CONFIRMATION

43. The development of a separate confirmatory method is not generally needed when the original method is based on mass spectrometry or another highly specific technique. By far, gross error (mistakes) is the greatest source of misidentifications in MS-based methods. For this reason, all regulatory enforcement actions require confirmation of the result via re-extraction of a replicate test portion of the original sample and re-analysis, ideally using different chemistries of sample preparation and/or analysis. Millions of dollars, international relations, and personal/business reputations may be at stake in regulatory determinations, and the laboratory must be sure of that all reports of residue violations are correct and validated.

44. Selectivity is the primary consideration for methods of identification. The method should be sufficiently selective to provide unambiguous identification. Mass spectrometry coupled to a chromatographic separation method is a very powerful combination for identification of an analyte in the sample extract. GC-MS and LC-MS tools (full-scan, selected ion mode, high-resolution, tandem MS/MS, hybrid systems, among other advanced techniques) provide many measurable parameters, such as retention times, chromatographic peak shapes, ion intensities and relative abundances/ratios, mass accuracies, and other useful aspects to help make analyte identifications.

Multiple options exist as appropriate identification criteria

45. The following chromatography-MS/MS identification criteria, or similar established criteria as shown in Tables 1-2, should be met for regulatory purposes:

    a. the retention time of the detected analyte peak must be within ±0.2 min of the contemporaneously analyzed analyte reference standard peak;

    b. the different ion transitions for the analyte should co-elute with similar peak shapes;

    c. the ratios of peak areas for each ion transition should match the ratios of the standard(s) within specified criteria. Options include using ±10% absolute for one transition or ±20% absolute for two or more transitions, or following the criteria stated in Table 2;

d. reagent and matrix blanks must be shown to be free of carry-over, contamination, and/or interferences above an appreciable level;

e. signal/noise ratios for measured peaks must be greater than 3;

f. the signal must exceed the threshold intensity level as compared to the signal of a suitable reference standard or control encompassing the level of interest; and

g. the ion transitions chosen for identification purposes should make chemical/structural sense.

46. Methods based on high-resolution mass spectrometry are considered to give a greater reliability through more precise measurement of mass than can be obtained using low-resolution mass spectrometry techniques. Different types and models of mass spectrometric detectors provide different degrees of selectivity, which relates to the confidence in identification. The requirements for identification are given in **Table 1.** They should be regarded as guidance criteria for identification, not as absolute criteria to prove presence or absence of a compound.

Table 1: Identification requirements for different types of mass spectrometers

MS mode	**Single MS (unit mass resolution)**	**Single MS (high-resolution/high mass accuracy)**	**MS/MS**
Typical systems (examples)	Quadrupole, ion trap, time-of-flight (TOF)	TOF, Orbital ion, FTMS, magnetic sector	Triple quadrupole ion trap, hybride MS (e.g. Q-TOF, Q-trap)
Acquisition	Full scan, Limited mass-to-charge ratio value (*m/z* range), Selected ion monitoring (SIM)	Full scan, Limited *m/z* range, Selected ion monitoring (SIM)	Selected/multiple reaction monitoring (SRM/MRM), full scan product-ion spectra
Requirements for identification	≥ 3 diagnostic ions, (preferably including quasi molecular ion)	≥ 2 diagnostic ions (preferably including the quasi molecular ion). Mass accuracy < 5 ppm. At least one diagnostic ion.	≥ 2 product ions

47. The relative abundances (intensities) or ratios of selective ions (full-scan MS or SIM) or product ions (MS/MS), expressed as a ratio relative to the most intense (product) ion, should correspond to those of the calibration standard at comparable concentrations and measured under the same conditions. Matrix-matched calibration solutions may need to be used. Table 2 indicates the commonly acceptable tolerances for ion ratios. The tolerances given in **Table 2** should not be taken as absolute limits and automated data interpretation based on the criteria without complementary interpretation by an experienced analyst is not recommended.

Table 2: Recommended maximum (default) tolerances for ion ratios using different MS techniques

Ion ratio (least/most intense ion)	Maximum tolerance (relative) for GC-EI-MS	Maximum tolerance (relative) for LC-MS^n, LC-MS, GC-MS^n, GC-CI-MS
0.5-1.0	±10%	±30%
0.2-0.5	±15%	±30%
0.1-0.2	±20%	±30%
<0.10	±50%	±30%

48. Additional confidence is provided with the use of high-resolution mass spectrometers (or detection using mass spectrometers with high resolving power, typically > 20,000 full width half maximum (FWHM) that offer more precise identification of the mass and may be used to predict the elemental composition of each fragment). In addition, at least one diagnostic ion must also be measured to eliminate the potential for fragments of the same mass arising from isobaric compounds of similar structure.

49. The minimum acceptable retention time for the analyte(s) under examination should be at least twice the retention time corresponding to the void volume of the column. The retention time of the analyte in the extract should correspond to that of the calibration standard (may need to be matrix-matched) with a tolerance of ±0.2 min, for both gas chromatography and liquid chromatography. Greater retention time deviations are acceptable where both retention time and peak shape of the analyte match with those of a suitable isotopically labeled internal standard (IS), or evidence from validation studies is available.

50. For enforcement actions, confirmation that analytes are present in the samples must be made by a second analysis, and one of the confirmatory methods should involve analyte identification, typically using MS techniques. Moreover, the confirmatory methods should use independent approaches based on different chemical mechanisms, such as liquid and gas chromatography (LC and GC) separations. In some situations, confirmation by independent laboratories may be appropriate. Examples of analytical techniques that may be suitable to meet criteria for confirmatory analytical methods are summarized in **Table 3**.

51. Whenever chromatographic techniques are used in screening or confirmation, proper settings of the retention time windows is pivotal. Care should be taken that the instrument is adjusted correctly before starting the analysis; a system suitability test should be performed prior to each batch of analysis. Retention time data base should be adjusted for the current conditions. In tolerance intervals of 1.5 to 3% of the absolute retention time may be applied for capillary GC depending on the peak shape. For confirmation of the retention time, the absolute tolerance intervals will increase at higher retention time. The tolerance interval should be less than 0.2 minutes or 0.2% relative retention time (RRT). For higher retention times, 6 seconds is a suitable interval. Additional guidance is given in CAC/GL 56-2005, “Guidelines on the Use of Mass Spectrometry for Identification, Confirmation and Quantitative Determination of Residues”. Other chromatographic systems (applying stationary and/or mobile phases of different selectivity) or other techniques may also be readily available and applied.

Table 3: Examples of detection methods suitable for the confirmatory analysis of substances, as recommended by the Miskolc Consultation

Detection method	**Criterion**
LC or GC and MS	If sufficient number of fragment ions are monitored
LC-DAD	If the UV spectrum is characteristic
LC – fluorescence	In combination with other techniques
2-D TLC – (spectrophotometry)	In combination with other techniques
GC-ECD, NPD, FPD	Only if combined with two or more separation techniques
Derivatisation	If it was not the first choice method
LC-immunogram	In combination with other techniques
LC-UV/VIS (single wavelength)	In combination with other techniques

APPENDIX I

DEFINITIONS

Analyte: the chemical substance sought or determined in a sample.

Analyte protectant: compounds that strongly interact with active sites in the gas chromatographic (GC) system, thus decreasing degradation, adsorption, or both of co-injected analytes.

Confirmatory method: a method that provides complementary information in agreement with a previous result. Ideally, a different subsample is analyzed with a method involving a different chemical mechanism than in the first analysis, and one of the methods meets analyte identification criteria with an acceptable degree of certainty at the level of interest

Determination: quantitative result of a method, but which has not yet met identification or confirmation criteria.

False positive: a result wrongly indicating that the analyte concentration is present or exceeds a specified value

False negative: a result wrongly indicating that the analyte concentration is not present or does not exceed a specified value.

Identification: process of unambiguously determining the chemical identity of a pesticide or metabolite in experimental or analytical situations.

Incurred residue: residue identified in a commodity resulting from specific use of a pesticide or from consumption by an animal or environmental contamination in the field, as opposed to residues identified from laboratory fortification of samples.

Interference: intrinsic or extrinsic response unrelated to an analyte (noise) due to electronic, chemical, or other factors related to the instrumentation, environment, method, or sample.

Internal standard (IS): a chemical added at a constant amount to samples and/or standards in a chemical analysis, including the blank and calibration standards. This substance can then be used for calibration by plotting the ratio of the analyte signal to the internal standard signal as a function of the analyte concentration of the standards. This ratio for the samples is then used to obtain their analyte concentrations from a calibration curve. The internal standard used needs to provide a signal that is similar to the analyte signal in most ways but sufficiently different so that the two signals are readily distinguishable by the instrument.

Limit of quantification (LOQ): [See paragraph 27].

Matrix: the material or component sampled for pesticide residue studies.

Matrix blank: sample material containing no detectable concentration of the analytes of interest.

Matrix-matched standards: standard solutions prepared in a matrix extract similar to that of the sample to be analyzed which compensate for matrix effects if present.

Maximum residue level/limit (MRL/CXL): maximum concentration of a residue that is legally permitted or recognized as acceptable in, or on, food commodities as set by Codex (CXL) or a national regulatory authority (MRL). The term tolerance used in some countries is, in most instances, synonymous with MRL (normally expressed as mg/kg fresh product weight).

Multiresidue method (MRM): a method which can determine three or more analytes in the same chemical class or in more than one class of pesticide.

Precision: degree of variability of a measurement around a mean.

Quantitative method: a method capable of producing analyte concentration (determinative) results with trueness and precision that comply with established criteria.

Relative Standard Deviation (RSD): the standard deviation, divided by the absolute value of the arithmetic mean, expressed in percentage. It refers to the precision of the method. Considering a single laboratory, the precision is expressed in terms of repeatability and reproducibility within the laboratory.

Repeatability: for an analytical method, the closeness of agreement between results of measurements on identical test material subject to the following conditions: same analyst, same instrumentation, same location, same conditions of use, repetition over a short period of time.

Reproducibility: for an analytical method, the closeness of agreement between results of measurements on identical test material where individual measurements are carried under changing conditions such as: analyst, instrumentation, location, conditions of use, and time.

Residue: amount of pesticide (or other contaminant) on the sample, typically originating from application of the farmer in the field, but it may results from drift, environmental exposure, or other forms of contamination.

Sample preparation: Involves the extraction of a test portion of the sample, its cleanup and other steps in the method that leads to a final extract for analysis.

Sample processing: procedure to yield a test portion for analysis that is representative of the collected sample and maintains the integrity of the analytes. This involves cutting, homogenization, comminunition, blending, or other means using appropriate techniques and equipment depending on the sample type and sizes of the collected sample and test portions.

Screening detection Limit (SDL): the screening detection limit of a qualitative screening method is the lowest concentration for which it has been demonstrated that a certain analyte can be detected (not necessarily meeting unequivocal identification criteria) in at least 95% of the samples (e.g. a false negative rate of 5% is accepted).

Screening method: a method that meets predetermined criteria to detect the presence, or absence, of an analyte or class of analytes at or above the minimum concentration of interest.

Selectivity: the extent to which the method can be used to determine particular analytes in mixtures or matrices without interferences from other components of similar behavior. Some regulatory authorities use the term specificity to refer to selectivity.

Sensitivity: quotient of the change in the indication of a measuring system and the corresponding change in the value of the quantity being measured.

Standard addition: the method of standard addition is a type of quantitative analysis approach often used in analytical chemistry whereby the standard is added directly to the aliquots of analyzed sample.

Uncertainty: a parameter associated with the result of a measurement that characterizes the dispersion of values that could reasonably be attributed to the measurement.

Trueness: refers to the closeness of agreement between a test result and the accepted reference value of the property being measured

APPENDIX II

REFERENCES

1	CAC/GL 27-1997	Guidelines for the Assessment of the Competence of Testing Laboratories Involved in the Import and Export Control of Food
2	CAC/GL 37-2001	Harmonized IUPAC Guidelines for the use of Recovery Information in Analytical Measurement. *Pure & Appl. Chem.*, **71**,1999; 337 – 348
3	CAC/GL 40-1993	Guidelines on Good Laboratory Practice in Pesticide Residue Analysis
4	CAC/GL 49-2003	Harmonized IUPAC Guidelines for Single-Laboratory Validation of Methods of Analysis *Pure & Appl. Chem.*, **74**,2002; 835–855
5	CAC/GL 56-2005	Guidelines on the Use of Mass Spectrometry (MS) for Identification, Confirmation and Quantitative Determination of Residues
6	CAC/GL 59-2006	Guidelines on Estimation of Uncertainty of Results
7	CAC/GL 64-1995	Protocol for the Design, Conduct and Interpretation of Method Performance Studies
8	CAC/GL 65-1997	Harmonized Guidelines for Internal Quality Control in Analytical Chemistry Laboratories, *Pure & Appl. Chem.*, **67,**1995; 649-666
9	CAC/GL 72-2009	Guideline on Analytical Terminology
10	CAC/STAN 193 -1995	General Standard for Contaminants and Toxins in Food and Feed

11	Guidance Document on Pesticide Residue Analytical Methods OECD	Guidance Document on Pesticide Residue Analytical Methods ENV/JM/MOMO(2007)17 OECD Environment, Health and safety Publications, Series on Testing and Assessment, No. 72, Series on Pesticides No. 39
12	Guidance Document on the Definition of Residue OECD	OECD Guidance Document on the Definition of Residue ENV/JM/MONO(2009)30
13	Identification and Confirmation of Chemical Residues in Food by Chromatography	Identification and Confirmation of Chemical Residues in Food by Chromatography – Mass Spectrometry and Other Techniques *Trends in Analytical Chemistry,* 27(11), 2008;1070-1090
14	ISO/IEC 17025 -General requirements for the competence of testing and calibration laboratories_	ISO 9000:—1), Quality management systems — Fundamentals and vocabulary ISO 9001:2000, Quality management systems — Requirements
15	ISO VIM	ISO International Vocabulary of Basic and General Terms in Metrology (VIM)
16	IUPAC Glossary of Terms Relating to Pesticides	Glossary of Terms Relating to Pesticides *Pure & Appl. Chem.*, 68 (5), 1996; 1167-1193
17	IUPAC Harmonized Guidelines For Single-Laboratory Validation Of Methods Of Analysis	Harmonized Guidelines For Single-Laboratory Validation Of Methods Of Analysis *Pure & Appl. Chem.*, 74(5), 2002; 835 – 855
18	IUPAC Selectivity in Analytical Chemistry	Selectivity in analytical chemistry *Pure & Appl. Chem.,* 73(8), 2001; 1381-1386
19	Miskolc, Hungary Nov 1999	Miskolc, Hungary in Nov 1999 to develop “Guidelines for Single-Laboratory Validation of Analytical Methods for Trace-level Concentrations of Organic Chemicals,” which cover pp. 179-252
20	Principles and Practices of Method Validation	Principles and Practices of Method Validation. A. Fajgelj and Á. Ambrus (Eds.), Royal Society of Chemistry, Cambridge, UK, 2000.
21	IUPAC Selectivity in Analytical Chemistry	Selectivity in analytical chemistry *Pure & Appl. Chem.,* 73(8), 2001; 1381-1386

22	SANCO/825/00rev8.1	Guidance document on pesticide residue analytical methods
23	SANCO/12571/2013	SANCO Analytical quality control and validation procedures for pesticide residues analysis in food and feed update of SANCO/12495/2011

APPENDIX II

LIST OF PARTICIPANTS

Electronic Working Group on the

Performance Criteria for Suitability Assessment

of Methods of Analysis for Pesticide Residues

CHAIR

Dr. Parthapratim (Pat) Basu
Alternate US Delegate to CCPR
US Department of Agriculture,
Food Safety Inspection Service, OPHS
1200 Independence Ave., SW,
Washington, DC 20250, USA
Phone: +1 202-690-6558
Fax: +1 202-690-2364
Email: pat.basu@fsis.usda.gov

CO-CHAIRS

Dr. Canping Pan (CO-CHAIR)
Professor
College of Science
China Agricultural University
10093 Beijing, CHINA
Tel: 86-10-62731978
Fax: 86-10-62733620
E-mail: Panc@cau.edu.cn

Dr. Krishan Kumar Sharma (CO-CHAIR)
Network Coordinator
All India Network Project on Pesticide Residues
Indian Agricultural Research Institute
New Delhi, INDIA
Email: kksaicrp@yahoo.co.in

==========

AUSTRALIA

Ms Karina Budd
Director
Residue Chemistry & Laboratory Performance
Evaluation Section, National Residue Survey
Department of Agriculture, Fisheries and Forestry
GPO Box 858
2601 Canberra, ACT, Australia
Phone: +61 2 6272 5795
E-mail: karina.budd@daff.gov.au

BAHRAIN

Dr. Abbas Ali
Sr. chemist
Public Health Laboratory
Kingdom of Bahrain
Tel: 00973-33917936
Email: asalman@health.gov.bh

Co-Chair: Abdul Nabi AL-Natei
Sr. Chemist
Public Health Laboratory –Chemical Analysis
Group (CAG)
Ministry of Health
Kingdom of Bahrain
Tel. 00973 17279241 Ext 2062
Mobile 00973 39259420
Fax 00973 17279228
Email: Anatie@health.gov.bh

BRAZIL

Mr. Carlos Venâncio
Head of Pesticide Registration
Ministry of Agriculture Livestock and Food Supply
Esplanada dos Ministerios - Bloco D - Edificio Anexo - 3° Andar - Sala 325 –
Ala A
Brasilia, BRAZIL
Tel: + 55 61 3218-2445
carlos.venancio@agricultura.gov.br:
codex@agricultura.gov.br

CANADA

Dr. Jian Wang
Head, Research and Development
Calgary Laboratory
Canadian Food Inspection Agency
Phone: (403) 338-5273-Email:
jian.wang@inspection.gc.ca

CHILE

Soraya Sandoval
National Coordinator - CCMAS
Institute of Public Health (ISP), Ministry of Health
Email: soraya@ispch.cl

Soledad Ferrada
National Coordinator - CCPR
Agriculture and Livestock Service (SAG), Ministry of Agriculture
Email: soledad.ferrada@sag.gob.cl

CHINA

Dr. Camping Pan (**CO-CHAIR**)
Professor, College of Science
China Agricultural University
10093 Beijing, CHINA
Tel: 86-10-62731978
Fax: 86-10-62733620
E-mail: Panc@cau.edu.cn

COSTA RICA

Ms. Verónica Picado Amador
Jefe Laboratorio de Análisis de Residuos de Agroquímicos
Servicio Fitosanitario del Estado
Ministerio de Agricultura y Ganadería
San José- Costa Rica
Tel: (506) 25493604
E-mail: vpicado@sfe.go.cr

Ms. Amanda Lasso Cruz
Ministerio de Economía Industria y Comercio
Departamento Codex
San Jose- Costa Rica
Tel. 25491434
E-mail: alasso@meic.go.cr

EUROPEAN UNION

Ms Almut Bitterhof
Email: Almut.bitterhof@ec.europa.eu

Ms Veerle Vanheusden
Email: Veerle.vanheusden@ec.europa.eu

FRANCE

Mr Frédéric HOMMET
National expert
French Agency for Food, Environmental and Occupational Health & Safety
27-31 avenue du Général Leclerc 94701 Maisons-Alfort Cedex, FRANCE
Tel: +33 (0) 1 49 77 13 50
E-mail: frederic.hommet@anses.fr

GERMANY

Dr. (Mrs.) Nadja Buchner
Federal Office of Consumer Protection and Food Safety
Unit 504 “NRL for Pesticide Residues”
Diedersdorfer Weg 1
12277 Berlin
Germany
Phone: +49 30 18445-8124
E-mail: nadja.buchner@bvl.bund.de

Dr. Jochen Heidler
Federal Institute for Risk Assessment
Max-Dohrn-Straße 8–10
10589 Berlin
Germany
Phone: +49 30 18412-3478
E-mail: jochen.heidler@bfr.bund.de

GHANA

Dr. Augustine Donkor
Codex Contact Point, Ghana
Email: adonkor@ug.edu.gh

GREECE

Dr Konstantinos S. Liapis
Head of Pesticides Residues Laboratory (National Reference Laboratory)
Head of Department for Pesticides Control & Phytopharmacy
Benaki Phytopathological Institute
7 Ekalis Street, Kifissia, Athens 145 61, GREECE
Tel +302108180366
E-mail: k.Liapis@bpi.gr & codex@efet.gr

Dr Chris Anagnostopoulos
Researcher
Laboratory of Pesticide Residues (National Reference Laboratory)
Department of Pesticides Control and Phytopharmacy,
Benaki Phytopathological Institute,
7 Ekalis Street, Kifissia, Athens 145 61, GREECE
Tel. +30-210-8180364
email: c.anagnostopoulos@bpi.gr

INDIA

Dr. K.K. Sharma (**CO-CHAIR**)
Principal Scientist & Network Coordinator (Pesticide Residues)
All India Network Project on Pesticide Residues
Indian Agricultural Research Institute
New Delhi, India
Email: kksaicrp@yahoo.co.in

Dr. P. Nagaraj
Scientist, Quality Evaluation Laboratory
Spices Board of India
E-mail: ccschnagaraj@gmail.com

INDONESIA

Mr. Flan Hernadi
Supervisor for Agricultural Product Quality
Ministry of Agriculture
E-mail: akh.hanif@gmail.com; and codex_kementan@yahoo.com

IRAN

Mrs. Roya Noorbakhsh
Secretary of CCPR in Iran
Expert on Pesticide Residues in Food
Fax: +98 26 32803889
Email: roybakhsh@yahoo.com

JAPAN

Mr. Yoshiyuki TAKAGISHI
Section Chief
Agricultural Chemicals Office
Plant Products Safety Division
Food Safety and Consumer Affairs Bureau
Ministry of Agriculture, Forestry and Fisheries
1-2-1 Kasumigaseki, Chiyoda-ku, Tokyo 100-8950, Japan
Phone: +81-3-3502-5969
E-mail: yoshiyuki_takagishi@nm.maff.go.jp, codex_maff@nm.maff.go.jp

Mr. Kazuyoshi KOJIMA
Director, Precision Management
Planning and Coordination Department
Food and Agricultural Materials Inspection Center
(Incorporated Administrative Agency)
Saitama Shintoshin National Government Building Kensato BLDG.
2-1, Shintoshin, Chuo-ku, Saitama-shi, Saitama 330-9731 JAPAN
Phone: +81-50-3797-1827
E-mail: kazuyoshi_kojima@nm.famic.go.jp

Ms. Asako OGAWA
Assistant Director
Standards and Evaluation, Department of Food Safety,
Ministry of Health, Labour and Welfare
1-2-2 Kasumigaseki, Chiyoda-ku Tokyo 100-8916, japan
Phone:+81-3-3595-2341
E-mail: codexj@mhlw.go.jp

Dr. Satoru NEMOTO
Section Chief
Organization: Division of Foods, National Institute of Health Sciences
1-18-1, Kamiyoga, Setagaya-ku, Tokyo 158-8501, japan
Phone:+81-3-3700-1141
E-mail: nemoto@nihs.go.jp

LUXEMBOURG

Danny Zust
Chargé de mission
Présidence luxembourgeoise 2015
Direction de la santé
Service de la sécurité alimentaire
9, avenue Victor Hugo L-1750 - Luxembourg
Tel: +352 247-75632
Fax: +352 27 47 80 86
danny.zust@ms.etat.lu

NETHERLANDS

Dr. Henk van der Schee
Scientific Officer
Dutch Food and Consumer Product Safety Authority
Catharijnesingel 59
3511 GG Utrecht, PO box 43006
3540 AA Utrecht
Tel: + 31 (0) 900 0388
Mobile: +31 (0) 615036231
Email: h.a.vanderschee@nvwa.nl

NIGERIA

Mr. B.I. Urulor
National Agency for Food Drugs Administration and Control (NAFDAC)
Phone: 08037584580
E-mail: urubik@yahoo.com; codexsecretariat@son.gov.ng

PARAGUAY

José Eduardo Giménez Duarte
Agronomist
Head, Department of Vegetable Traceability
National Quality and Plant Protection
SENAVE- Paraguay
Emai: jose.gimenez@senave.gov.py

REPUBLIC OF KOREA

Sohn Yong-Wook
Deputy Director, Food Standard Division
Ministry of Food and Drug Safety (MFDS)
Email: s9918@korea.kr

Chang Moon-Ik
Deputy Director, Pesticide & Veterinary Drug Residue Division
Ministry of Food and Drug Safety (MFDS)
Email: 1004@korea.kr

Kwon Chan-Hyeok
Scientific Officer, Food Standard Division
Ministry of Food and Drug Safety (MFDS)
Email: chkwon@korea.kr

Kim Hyo-Chin
Scientific Officer, Food Standard Division
Ministry of Food and Drug Safety (MFDS)
Email: hckim77@korea.kr

Kim Hee-Jung
Deputy Director, Pesticide & Veterinary Drug Residue Division
Ministry of Food and Drug Safety (MFDS)
Email: heejung731@korea.kr

Codex Korea Contact Point
Ministry of Food and Drug Safety (MFDS)
Email: codexkorea@korea.kr

RUSSIA

Prof. Konstantin Eller
Head of the Laboratory
Institute of Nutrition
Russian Academy of Medical Sciences
Moscow, Russia
Email: eller@ion.ru

SLOVAK REPUBLIC

Dr. Jarmila Ďurčanská
Veterinárny a potravinový ústav Bratislava (Veterinary and Food Institute in Bratislava)
Botanická 15
842 52 Bratislava, Slovak Republic
E-mail: durcanska@svuba.sk

SWEDEN

Mrs. Tuija Pihlstrom
Chemist, National Food Agency
Sweden
E-mail: Tuija.pihlstrom@slv.se

SWITZERLAND

Ms. Henri Diserens
Early Warning Group
Food Safety & Quality Competence Pillar
Nestlé Ltd
Nestlé Research Center
PO Box 44
CH-1000 Lausanne 26, Switzerland
E-mail: henri.diserens@rdls.nestle.com

THAILAND

Mrs. Kanokporn Atisook
Medical Scientist, Expert Level
Bureau of Quality and Safety of Food, Department of Medical Sciences,
Ministry of Public Health
88/7 Moo 4 Tiwanon Road, Tambon Talad Kwan
Amphur Muang Nonthaburi 11000, Thailand.
Tel: + 662 951 0000 ext. 99602
E-mail: katisook@yahoo.com & codex@acfs.go.th

Miss Panpilad Saikaew
Standards Officer
Office of Standard Development
National Bureau of Agricultural Commodity and Food Standards
50 Phaholyothin Road, Ladyao, Chatuchak, Bangkok 10900 Thailand
Tel: (662) 561 2277 ext 1427
E-mail: panpilad@acfs.go.th

UNITED STATES OF AMERICA

Dr. Parthapratim (Pat) Basu - Chair
Alternate US Delegate to CCPR
Senior Leader, Chemistry, Toxicology & Related Sciences
US Department of Agriculture
Food Safety Inspection Service, OPHS
1200 Independence Ave., SW
Washington, DC 20250
Phone: +1 202-690-6558
Fax: +1 202-690-2364
Email: pat.basu@fsis.usda.gov

Marie Maratos
International Issues Analyst
U.S. Codex Office
Food Safety Inspection Service
US Department of Agriculture
Room 4865 South Building
1400 Independence Blvd, SW
Washington, DC, USA
Phone: +1.202.690.4795
Email: Marie.Maratos@fsis.usda.gov

Charles E Pixley, DVM, PhD
OPHS, LQAS
Food Safety Inspection Service
US Department of Agriculture
Russell Research Center
950 College Station Rd.
Athens, GA 30605
Tel: +1 706-546-3559
Email: charles.pixley@fsis.usda.gov

Louis Bluhm, Ph.D.
Chemistry Team Leader
Laboratory Quality Assurance Staff
Administrator, Accredited Laboratory Program
USDA FSIS OPHS
950 College Station Rd., Athens, GA 30605
Phone: 706-546-2359
Fax: 706-546-3453
Email: louis.bluhm@fsis.usda.gov

Dr. Steve J. Lahotay
Lead Scientist
Eastern Regional Research Center
Agricultural Research Service
US Department of Agriculture
600 East Mermaid Lane
Wyndmoor, PA 19038
Tel: +1 215-233-6433,
Fax: +1 215-233-2642
Email: Steven.Lehotay@ars.usda.gov

E. Scott Weber III VMD, MSc
AAAS Science and Technology Policy Fellow
Scientific Analyst, International Regulations and Standards Division
Office of Agreements and Scientific Affairs
Foreign Agricultural Service
US Department of Agriculture
Tel: (202) 720-6868
Fax: (202) 720-0433
Email: scott.weber@fas.usda.gov

John J. Johnston, PhD, MBA
Scientific Liaison
U.S. Department of Agriculture
Food Safety and Inspection Service
Office of Public Health Science
Fort Collins, CO 80526
Tel: +1- 202-365-7175
Email: John.Johnston@fsis.usda.gov

Thuy Nguyen
Director, Analytical Chemistry Laboratory
Environmental Science Center
701 Mapes Road
Ft. Meade, MD 20755
Tel: +1-410-305-2905
Email: nguyen.thuy@epa.gov

Sara Kucenski
Agricultural Scientific Analyst
International Regulations & Standards Division
International Standards Group
Office of Agreements & Scientific Affairs
Foreign Agricultural Service
U.S. Department of Agriculture
1400 Independence Ave., SW
Washington, DC 20250
Tel: +1 202-720-6741
Email: SaraKucenski@fas.usda.gov

Terry Councell
U.S. Food and Drug Administration
Center for Food Safety and Nutrition
Office of Food Safety
5100 Paint Branch Parkway
College Park, MD 20740
Phone: +1 240-402-1180
Email: Terry.Councell@fda.hhs.gov

==========

INTERNATIONAL ATOMIC ENERGY AGENCY (IAEA)

Dr. Johannes Corley
International Atomic Energy Agency
Vienna International Centre
PO Box 100
1400 Vienna, Austria
Phone: (+43-1) 2600-21695
E-mail: j.s.corley@iaes.org

INTERNATIONAL FROZEN FOOD ASSOCIATION

Ms. Maia M. Jack, Ph.D.
Director, Regulatory and International Affairs
American Frozen Food Institute
International Frozen Food Association Secretariat
Tel: +1 703-821-0770
Email: mjack@affi.com

INTERNATIONAL ORGANIZATION OF VINE AND WINE (OIV)

Dr Jean- Claude RUF
OIV, Scientific Coordinator
18, rue d'Aguesseau
F-75008 Paris, France
Tel: +33 (0) 1 44 94 80 94
Fax: +33 (0) 1 42 66 90 63
Mobile: +33 674 663 451
E-mail: jruf@oiv.int