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EA Guidance on Accreditation of Pesticide Residues Analysis in Food and Feed

PURPOSE

This paper provides guidance to develop harmonized accreditation of Pesticide Residues Analyses in Food and Feed.

Authorship
The publication has been written by the Laboratory Committee.

Official language
The text may be translated into other languages as required. The English languages version remains the definitive version.

Further information
For further information about this document, contact the EA Secretariat. Please check the EA website for up-to-date information (http://www.european-accreditation.org).

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1. INTRODUCTION

It is necessary to note that food and feed test reports generated in one country, and protected by the accreditation mark of an National Accreditation Body (NAB), may end up in other countries as part of the information required by operators for their trade within the EU.

On the other hand, the international dimension of the economy implies that the same organization of laboratories may be operating in different European countries for the same test activities in each country and apply for accreditation thereof.

In this document those criteria to be applied in the assessment of laboratories performing tests for pesticide residues in the agri-food area are specifically addressed, given the need for harmonization identified by both, users of test reports as many NABs; and even the European Commission itself.

Results of tests of pesticides residue analysis have a big impact in the regulatory and food safety fields. Therefore, application of technical criteria in the evaluation of the technical competence of the laboratories, clear established and recognized by all the interested parties is needed.

Fortunately, in this field of analysis a technical document is available “Guidance document on analytical quality control and method validation procedures for pesticides residue analyses in food and feed” (hereinafter, SANTE document) promoted by the European Commission and elaborated by representatives from recognized technical organizations like European Union Reference Laboratories, National Laboratories, universities and so on. There are, in addition, some specific technical documents concerning technical characteristics for analysis methods and their performance elaborated by Reference Laboratories of EU (EURL web page)

Therefore, technical criteria related to test methods, their validation, their uncertainty and their quality control shall be applied based on the SANTE document, that is regularly reviewed and according to its “Introduction and legal background” it gives support to compliance with the ISO/IEC 17025 standard.

Requirements established in EA-2/15 M document for the accreditation of flexible scopes shall be also taken into account. However, it is necessary to develop specific criteria for pesticide residue analysis that allow to dispose of harmonized rules to be applied by all NABs in the laboratory assessments with flexible scope.

It is also of interest to extend the field of harmonization that this document intends, to other key requirements of the ISO/IEC 17025, as are the contents of test reports and the review of requests. The criteria for assessing compliance with these requirements must necessary take into account the legal framework laid down in Regulation (EC) 396/2005, especially in the evaluation of compliance with the regulatory limits

It’s vital to note the extensive use of pesticide residue testing in the control activities of organic products covered by Regulation 834/2007 and 889/2008, accredited laboratories must consider the peculiarities associated to the analysis of samples from this type of production.

2. DEFINITIONS

Product family: generic name used to identify at the scope of accreditation the commodities covered by the flexible scope e.g. Fruit&Vegetables

Product group / matrices: set of commodities within the family, grouped according to criteria adopted by the laboratory conveniently e.g. High water content.

Representative matrix: matrix belonging to a group of commodities and selected according to appropriate technical criteria, with which the validation of the method is carried out in order to obtain results to be extrapolated to the product group e.g. apple.

Public List of Test (PLT): public document, prepared, reviewed and controlled by the laboratory, which include detailed description of matrices, pesticides and test method for which the laboratory can claim accreditation.

Reference document: document prepared by a technical organization or other kind enjoying the necessary degree of recognition and acceptance in the field in which it operates.

Pesticide residues: residues, including active substances, metabolites and degradation products or reaction products of active substances currently or formerly used in plant protection products, including those which may arise as a result of use in veterinary medicine and as a biocide

Maximum Residue Limit (MRL): the upper legal limit of concentration of a pesticide residue (as referred to Reg 396/2005 or National Legislation).

Limit of analytical determination (LOD): the validated lowest concentration of residue that can be quantified and reported by routine monitoring with validated control methods (as referred to Reg 396/2005).

Limit of quantification (LOQ): the lowest concentration of analyte validated that can be quantified with acceptable accuracy by applying the complete analytical method.

Limit of detection (LD): the lowest concentration of residue validated that can be detected and confirmed.

Reporting limit (RL): the lowest level at which residues will be reported as absolute numbers. It is equal to, or higher than LOQ (as referred to SANTE document).

3. PURPOSE

This document gives clarification and guidance to reach an harmonized approach to some requirements of ISO /IEC 17025 with the aim of ensuring a more reliable and transparent approach to the pesticides residue analysis offered by laboratories.

To achieve these ends is considered necessary that the NABs apply the principles of flexible accreditation set forth in the document EA-2/15.

Some aspects included in this document can be used in the accreditation of laboratories with fixed scope.

Main topics:

i) Technical issues: selection of test method, validation and internal quality control.

ii) Management issues: review of requests, tenders and contracts; content of test reports.

iii) Flexible scope requirements: Public list of testing and nonconforming work.

iv) Use of accreditation mark.

4. ACCREDITATION CRITERIA (TECHNICAL ISSUES)

This section is intended to optimize the work necessary to demonstrate the proper functioning of the test methods used by laboratories for analysis of pesticide residues, establishing a balanced and proportional system between the technical reliability thereof, the validation work and costs the laboratories have to assume and associated risks (eg. in terms of uncertainty of results)

Test Methods: in the selection of the test method for the analysis of pesticide residues, standardized methods (as defined in ILAC G 18) should preferably be considered due to they are widely accepted, known and applied in the sector as well as whenever it is possible the EURL- methods can be applied.

However, in this type of testing it is needed the application of technical criteria laid down in recognized technical documentation, which are called methods-criteria or collection of performance criteria for methods that allow the laboratory to verify the performance of their own methods for an intended use.

The use of methods-criteria should always be subject to performance criteria defined by a technical organization or recognized by a competent authority. Currently, the SANTE document establishes these criteria. Finally, publications from the EURL for pesticide residues available on EURL portal web establish technical criteria in the selection and use of methods of analysis of pesticide residues.

Therefore, for the selection of appropriate test method the laboratories have sufficient technical references for key processes: extraction, clean-up, chromatographic system, quantification and detection system. Currently, there is sufficient consensus on the detection system to be used: mass spectrometry and, for example, SANTE documents and Decision 2002/657/EC establish the criteria necessary for the spectral confirmation of the analyzed substances.

Any deviation from these criteria should be properly justified and demonstrated by the laboratory.

Finally, the laboratories can select both quantitative and qualitative methods. The latter have not necessarily to be consider as a screening and in any case its application should be based on the validation of a limit of detection suited to the specifications required by the applicable regulation, as in the case of organic production.

Validation: It must be carried out an initial validation to demonstrate the compliance with the performance requirements of the selected methods (as set out in the previous point) and the technical competence of the laboratory for validation. Validations should be evaluated from the records kept by the laboratory and from original observations.

This initial validation must be carried out on a sufficient number of representative matrices of the product family, so the NABs should ensure that the laboratories have established adequate criteria for their selection, which necessarily have to take into account the most frequently analyzed matrices.

The performance parameters of the selected methods, to be evaluated in the initial validation, and evaluation criteria should be based on SANTE document. Laboratories should document in a validation procedure both validation parameters to be determined and the evaluation criteria.

With regard to LOQ, it is considered that the result obtained in each pesticide should always be less than the legislated limit for the pesticide in the matrices analyzed, or any other limit, lower than the legislated one, defined by the client. In any case, shall be at least equal to the limit of analytical determination (LOD), currently established by EC Regulation 396/2005 at 0.01 mg / kg.

For qualitative methods, the validation parameters should be suitable for such methods, including the limit of detection and specificity / selectivity.

For pesticide residues defined as the sum of more than one substance ("complex residue definition"), the document SANCO 12574/2014 shall be taken into account. It is considered acceptable for the quantification limit of each analyte whenever it is less than or equal to the MRL for the complex residue definition. The result of this should be expressed using the validated limit of quantification for each analyte included in definition.

For flexible scope, NABs should ensure that the laboratory establishes an adequate strategy of validation to the flexibility introduced by the laboratory in the scope of accreditation (e.g. flexible products-fixed pesticides or flexible products and pesticides).

Quality Control: given the complexity of this type of testing it is essential that the NABs ensure that laboratories implement quality control procedures to guarantee the reliability of test results. A program of adequate quality control should consist of three types of activities:

a) Routine quality control activities included with each batch of test and therefore that must be documented in test procedure, consisting of at least one control of the process of extraction by testing recovery at the limit of quantification or at least Reporting limit or in the particular case of laboratory performing a confirmation analysis (a second analysis) at a concentration similar to the expected concentration in the sample; evaluation of "surrogate" in each sample of the batch; equip response verification, at least with a number of representative pesticides (in this case pesticides should change over time); control of calibration curve.

   It may also be necessary to include other routine activities, but not with each batch of test, depending on the analysis procedure such as control of blanks (reagent, matrix) etc.

b) Periodic internal control activities, which can be "blind samples", "double samples", reference materials and others; these activities should ensure to include positive samples, both at LOQ as other levels of interests (e.g. MRL, RL). The frequency of these controls should be based in the number and variety of samples usually analyzed by the laboratory.

The acceptance criteria for a) and b) should be established by the laboratory according to the results of the initial validation of the selected methods and those described in the SANTE document.

c) Participation in interlaboratory test comparisons: Currently there is an adequate offer of programs for analysis of pesticide residues and the laboratory might participate regularly in these programs, as a tool to demonstrate the validity of their testing results. If more than one food or feed groups be applied, at least a successful participation in a food or feed group and the application (s) for the other (s) the food and feed group (s) must be established, when sufficient proficiency tests are offered. In the surveillance period the laboratory should prove every year a successfully participated proficiency test. If the laboratory is accredited for more food or feed groups, an annual participation per food and feed group need to be sought, when appropriate proficiency tests are offered. For laboratories that are accredited for more than three food and feed groups, a two-year cycle is appropriate. If the participation is higher, then the activities referred to in paragraph b) can partially or fully replace.

If the laboratory has a flexible scope, NABs shall asses the quality assurance program (internal control and proficiency testing or interlaboratory test comparisons) in order to check if it covers the whole accreditation scope and not only limited to the samples and pesticides routinely tested.

According with SANTE document, it should be noted that proper planning of quality control activities can also be a very useful tool, which allows laboratories to gradually obtain data that can be leveraged for validation.

5. ACCREDITATION CRITERIA (MANAGEMENT ISSUES)

Review of requests, tenders and contracts: samples for testing can be from very different origin and with specific objectives. Usually samples are analyzed for compliance with legislated limits. It has to be noted that in some cases the limits have been established at national level, and therefore may differ from one country to another. Laboratories can also receive samples with the purpose of verifying compliance with residue limits for certain pesticides established by specific regulation or standards such as samples from organic farming, controlled production, etc. Therefore, in different circumstances the laboratory must clarify with the client as necessary the object of analysis and determine whether it can meet the limits required or issue the kind of desired result (eg. for organic farming is of interest to determine whether pesticide residues are detected or not, and not only if they can be quantified).

When the accreditation includes flexible scope, additional aspects should be considered in the review of the requests. So, laboratory should inform the customer about the need to perform any additional validation and the type of test report that can be issued based on the results of these validations and regarding the possibility that finally the report could not be covered by the accreditation. It should be noted that the flexible accreditation involves a commitment of the laboratory to offer accredited tests for any pesticide or matrix included in the scope of accreditation, even though at the time they are requested the tasks of validation and required verification have not been yet performed (see EA 2/15 5.1.3)

Testing reports: The following considerations should be taken into account:

- All requested pesticide residues that are part of laboratory methods shall be indicated. For results under the limit of quantification or reporting limit in the case of quantitative methods the limit of quantification or reporting limit shall be indicated. Similarly the detection limit in the case of qualitative methods shall be indicated for negative result.

- If reporting on the amount of a pesticide confirmed and quantified has taken into account a correction factor, this fact shall be stated in the report, the specification of the factor applied.

- In the case of pesticides residues with complex residue definition, the laboratory should preferably inform according to the regulated residue definition. However, the laboratory can report only those individual components that have been determined (e.g. active substance, degradation products, etc.) without declaring conformity in these cases with MRL, except when no questions may arise from the result obtained regarding the breach of MRL.

6. ACCREDITATION CRITERIA (FLEXIBLE SCOPE)

The NABs shall require to the laboratories to use a Public List of Testing (PLT).This document will be referred to in the scope of accreditation of the laboratory, and should be facilitated by laboratories to customers who request it. The laboratory should establish systematic update of PLT based on the incorporation of new matrices and / or pesticide residues according to the validations performed.

To ensure that this document is sufficiently homogeneous in terms of content, it should at least include:

a) Title: "Public List of Testing".

b) Revision and approval date.

c) Reference to the approved Scope of Accreditation.

d) Family (s) of products as listed in Scope of Accreditation, product groups established by the laboratory for each family, covering all of it, and within each group all the specific matrices that have been validated by the laboratory according to its procedure.

e) The pesticide residues for each of the product groups that have been validated by the laboratory according to its procedure and the limit of quantification or detection. The denomination of pesticide residues should be carried out in the same manner as in Scope of Accreditation (in the case of fixed pesticides scope the PLT only may include those listed in the Scope of Accreditation).

f) Test method detailing the analytical technique used and reference to specific procedure used by the laboratory.

It is necessary to inform the customer upon receiving the request about the laboratory capabilities based on the approved PLT.

The management of the flexible scope by laboratories necessarily requires conducting additional validation activities to incorporate new matrices and / or new pesticide residues in the PLT.

New matrices: in the event that the laboratory is asked to perform an analysis of a sample of a new matrix not included in the PLT, the laboratory validation procedures must indicate the verification of the method applied, previously to report accredited results. Such activities should at least include checking recovery on the limit of quantification and confirmation of pesticides included in the PLT for the product group in which the new matrix is included, as well as evaluates the results obtained in accordance with the requirements specified by the laboratory which should be in line with the initial validation and SANTE document.

New pesticides: for the inclusion of a new pesticide in the PLT a validation shall be performed. Similarly, if requested for a commodity of a group of matrices in which has not been previously validated, an initial validation must be also carried out, although this pesticide was included in the PLT for other groups.

The NABs should consider as an essential tool for laboratories to demonstrate their ability to effectively maintain the flexible scope, the proper management of nonconforming work arising out of the additional validation activities above. Thus, in the event that the laboratory has to perform additional validation, they must inform to the customer of the fact that can be given when the results obtained are not acceptable and that in that case it might not be possible to issue test results. When this situation happens the laboratory should ensure that management procedures for nonconforming work are implemented and that appropriate measures are taken (see EA-2/15 5.1.4).

7. USE OF ACCREDITATION MARK

Given the importance of testing pesticide residues, the NABs should reinforce the commitment by accredited laboratories for flexible scope to offer its customers all time only accredited results in test reports bearing the accreditation mark. So when a customer demands a test of this type within the scope of accreditation, it should be demanded by the NABs that the results issued are covered by the accreditation. Thus, for tests where it is proven that they are included in the current scope of accreditation, as it can be shown without any doubt by the identification of the matrix, analytes, method presented in the reports (as required in ISO/IEC 17025), the laboratory should always issue reports with accreditation mark or accreditation claim (depends on the policies of each NAB for the use of accreditation mark); it is without any question understood that these reports are accessible by NABs assessors and other NAB's surveillance tools as they are subject to services belonging to accredited scope of the laboratory. Therefore the widespread issuance of non-accredited results should be considered as a serious breach by NABs, even though they have customer acceptance. Such breaches should call into question the maintenance of the flexible scope.

8. REFERENCES

EA-2/15 M: 2008 *EA Requirements for the Accreditation of Flexible Scopes*

ILAC G 18:04/2010 Guideline for the Formulation of Scopes of Accreditation for Laboratories

SANTE/11813/2017 *Guidance document on analytical quality control and method validation procedures for pesticides residues analysis in food and feed*

SANCO/12574/2014 *Working document on the summing up of LOQs in case of complex residue definitions*

Regulation (EC) Nº 396/2005 *Maximum residue levels of pesticides in or on food and feed of plant and animal origin*

EURL Portal *(www.eurl-pesticides.eu)*

Regulation (EC) Nº 834/2007 *Organic production and labelling of organic products*

Decision 2002/657/EC *concerning the performance of analytical methods and the interpretation of results*