

Open/flexible scope accreditation  
(pesticide residue tests) following  
EN-ISO 17025: The Spanish experience

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ENAC (Spanish Accreditation Body)  
Technical Note 19 (NT-19)

- *The Spanish Accreditation Body (ENAC) is applying since 2004 some specific criteria for “flexible scope” accreditation of pesticide residue tests in food products, which are established in its “Technical Note 19” (NT-19)*

ENAC (Spanish Accreditation Body)  
Technical Note 19 (NT-19)

- *Revision 2 of the NT-19 was published in April 2006, and was elaborated by ENAC in a working group with representatives from public and private laboratories as well as the Spanish Food Safety Authority.*

(ENAC) NT-19 Rev. 2 April 2006

*Test Laboratories: Accreditation of Pesticide Residues Tests in Food Products*

- Objectives and field of application
- Applicable documents and references
- Accreditation scope (scope definition / Public list of Tests)
- Test Method Validation (initial validation / additional activities)
- Quality Assessment
- Revision of requests and test reports
- Assessment process
- Maintenance of the accreditation

# Objectives of the new revision

- Improve the public list of tests
- Validation criteria; Additional activities; new matrices, new pesticides
- Maintenance of the accreditation

# 1. Objective and field of application

- ✓ Applications for open scope accreditation generally expressed as:
  - ✓ Product testing (*matrix*)
  - ✓ Parameter testing (*analyte-pesticide*) to analyze
- DOES NOT APPLY to accreditation requests for analysis of: specific products and single residue method

## 2. Applicable documents and references

- ✓ UNE-EN-ISO/IEC 17025: General Requirements for the Competence of Calibration and Testing Laboratories.
- ✓ CGA-ENAC-LEC rev.3: General criteria for accreditation of calibration and testing laboratories according to UNE-EN-ISO/IEC 17025

## 3. Accreditation scope

### 3.1 Scope definition

The accreditation scope is described in the Technical Annex:

- ✓ Product/Material to test:
  - Product family.
- ✓ Test:
  - Analytical technique, pesticide residues.
- ✓ Testing method:
  - Testing procedure(s) applicable
- ✓ Reference to the "Public list of tests"

# LABORATORY

## Scope for the Accreditation Commission.

Request accreditation from the national accreditation body, conform with the criteria included in the standard UNE-EN-ISO/IEC 17025: 2005 and (CGA-ENAC-LEC), for the realization of :

### **Analysis of pesticides residues in fruits and vegetables**

Category 0 (Tests in the permanent laboratory)

Product/Material	Test	Method
Fruits and vegetables	Pesticide residues by gas chromatography and mass spectrometry detection (GC/MS/MS)	Internal Procedure PNT 04 MA 01 ed. 3 20/04/06

## 3. Accreditation scope

### 3.2 Public list of tests

- ✓ Public document, improved, revised and controlled by the laboratory.
- ✓ Lists the techniques which they can use as accredited.
- ✓ Aides the clients who request an analysis.

# 3. Accreditation scope

## 3.2 Public list of tests (Cont.)

### ✓ **Content:**

- a) Title: "Public list of test methods".
- b) Revision/edition number y approval date.
- c) Reference to the code and revision of the technical annex of ENAC.
- d) Family(s) of products, groups of products for each family and within each group all the specific matrices which have been validated/verified.
- e) The specific pesticides which have been validate by the laboratory for each group of products.
- f) Applicable testing procedure for each category, detailed analytical technique and reference to the specific testing procedures.

	Public list of analysis
Edition and approval date	Rev.2 (24-05-2006)
ENAC Technical annex	Acreditaion n°_ /LE Technical annex rev.___ Date
Procedure	MET-XX. Determination of pesticide residues by GC/MS/MS
Product family applicable	Fruit and vegetables

- *Group 1: Fruits and vegetables with high water content and low sugar content: beets, celery, marrow, endives.*
  - *Pesticides: dicofol, fenamifos, fenarimol, fenpropatrin, metalaxil, etc*
- *Group 2: Fruits and vegetables with high water content and high sugar content: grape, peach, nectarine, apple.*
  - *Pesticides: acrinatrin, buprofezin, ciprodinil, clozolinato, diazinon, etc.*

	Public list of analysis
Edition and approval date	Rev.2 (24-05-2006)
ENAC Technical annex	Acreditaion n°_ /LE Technical annex rev.___ Date
Procedure	MET-XX. Determination of pesticide residues by GC/MS/MS
Product family applicable	Fruits and vegetables

- *Group 3: Fruits and vegetables with high water content and high acidity: orange, lemon, grapefruit.*
  - **Pesticides:** *acrinatrin, buprofezin, ciprodinil, clozolinato, diazinon dicofol, fenamifos, fenarimol, fenpropatrin, metalaxil, etc*
- *Group 4: Fruits and vegetables with high fat content: olives, avocado.*
  - **Pesticides:** *procimidona, quinometionato tebuconazol, tetradifon, vinclozolina.*

# 4. Validation

- 4.1 Complete Initial validation:
- A) Demonstration of the ADJUSTMENT of the test method to the INTENDED USE:
  - Validation of representative matrices for each of the product groups defined in the family of products.  
Eg. Lettuce for the high water content group for the family "Leafy Vegetables"
  - Validation of representative analytes.
  - Representability: demand, analytical technique and complexity of the matrix/analyte combination.

# 4. Validation

- 4.1 Complete initial validation:

B) Demonstration of TECHNICAL COMPETENCE for TN-19 (management of open scope):

- Validation of at least 3 representative matrices
- Validation of at least 20 representative pesticides per matrix.

# 4. Validation

- Validation Procedure:
  - ✓ Validation strategy to follow.
  - ✓ Definition of the product groups.
  - ✓ Selection criteria for representative matrices and representative pesticides for each group
  - ✓ Validation parameters:
    - ✓ Limit of quantification( $\leq$ MRL) or application interval
    - ✓ linearity
    - ✓ precision and recuperation at different levels including the LoQ
    - ✓ selectivity
  - ✓ Requirements: specified (eg. SANCO in preference to others) and verification of fulfilment.

# 4. Validation

- New MATRICES:
  - New Matrix not included in the Public list tests (for the product group with complete validation) at least:
    - Recovery at LoQ
    - Confirm identity of the pesticides included in the public list for this group
- New PESTICIDES:
  - New Pesticide not included in the Public list
    - Complete validation in the matrix of interest
  - Pesticide included in the public analyses list but not validated for the requested product group:
    - Complete validation in the matrix of interest

# 5. Quality Assessment

- Quality control programme:  
should include at least three types of different control activities:
- Routine internal control activities:
  - For each analytical run
  - Documented in the test procedure.
  - At least to include: recovery (to meet the acceptance criteria), blank control, instrumental response verification with a series of representative pesticides from the Public analytical technique list (with rotation to evaluate each of them annually).

# 5. Quality Assessment

- Quality control programme:
- Periodic internal control activities:
  - Blind samples, double samples, RMC, others.
  - Inclusion of positive samples (at LoQ as the level or interest MRL)
  - The 'verified' matrices from the Public analytical technique list should be taken into account
  - Frequency based on the number and variety of received samples.
- External control activities (Proficiency Tests):
  - Periodic activity

Obtaining data to complete the validation of "verified" matrices

## 6. Revision of requests and test reports

- **Open scope (TN-19) implies commitment to offer accredited test from the technical annex.**
  
- **Contract revision:**
  - Should be kept on file
  - **Should clarify:**
    - i) Objective of the analysis (national legislation or international MRLs, integrated agriculture, ecological),
    - ii) laboratory offer (Technical annex and Public analytical technique list),
    - iii) Additional validation and places
    - iv) Type of test report which can be produced following the validation results.
  
  - Explicit acceptance of the client to publish pesticide results not covered by the accreditation and to document the reasons.