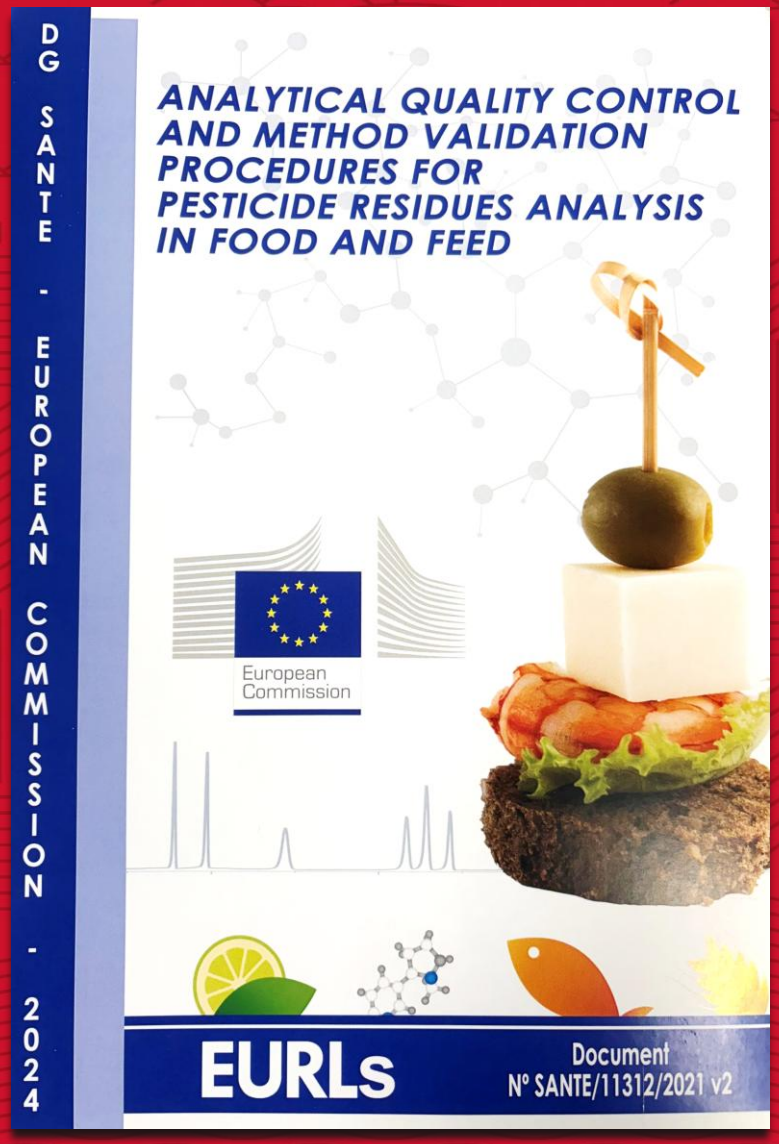
GOBIERNO DE ESPAÑA
MINISTERIO DE AGRICULTURA, PESCA Y ALIMENTACIÓN

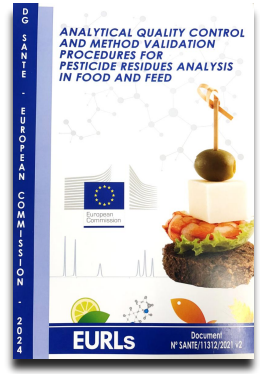


Cambios propuestos para la nueva versión (v3) del documento SANTE 11312/2021

Jornadas de Referencia del CNA
10 de Junio de 2025

Ana Lozano
Responsable del Dpto. Residuos y Contaminantes No Metálicos
Laboratorio Arbitral Agroalimentario

Documento SANTE 11312/2021

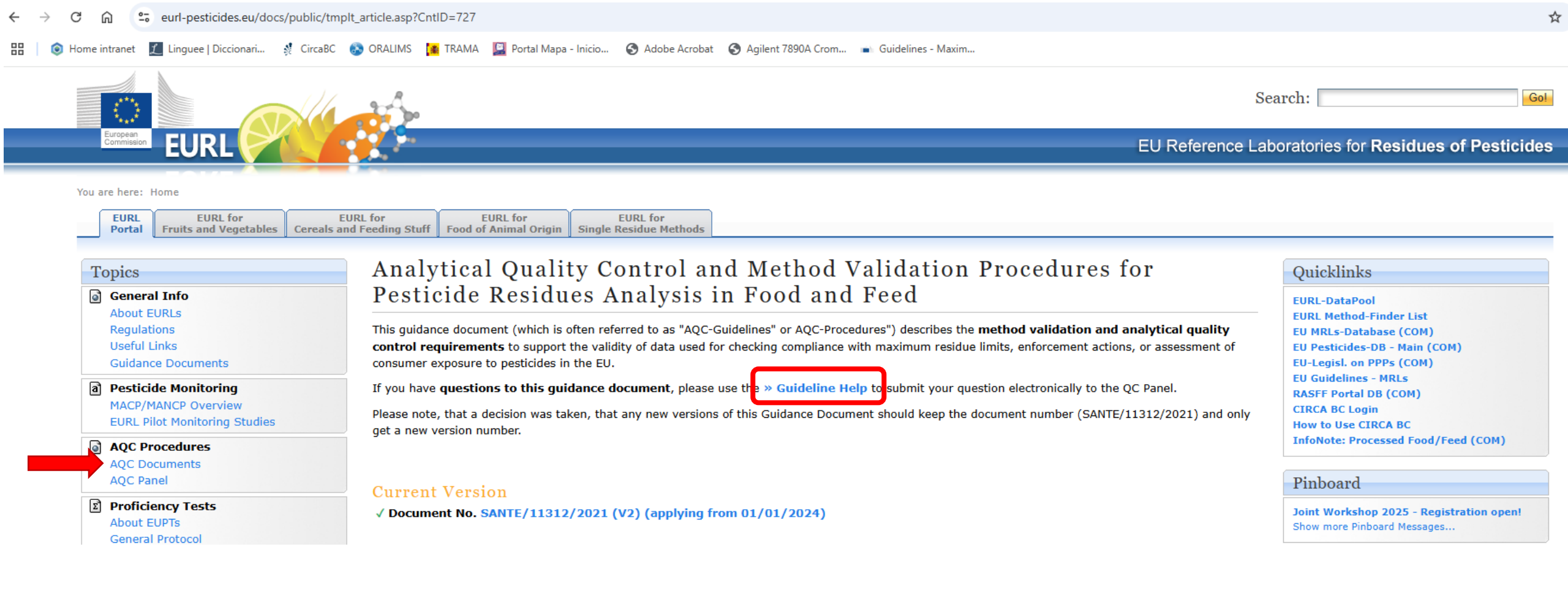


- Es un documento guía harmonizado entre todos los EEMM de la UE que describe la validación de métodos y los requerimientos de control de calidad para el control oficial en la UE.

- Se realiza una revisión del documento cada 2 años, teniendo una nueva versión a primeros de los años pares.

- En marzo de este año los coordinadores del documento pidieron propuestas de cambios a los EMM a través de los NRLs y nos enviaron una recopilación de cambios y comentarios que los enviamos a los OfLs españoles.

- Se recibieron propuestas de cambios de 4 OfLs.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About EUPTs
General Protocol
Analytical Quality Control and Method Validation Procedures for Pesticide Residues Analysis in Food and Feed
This guidance document (which is often referred to as "AQC-Guidelines" or AQC-Procedures") describes the method validation and analytical quality control requirements to support the validity of data used for checking compliance with maximum residue limits, enforcement actions, or assessment of consumer exposure to pesticides in the EU.
If you have questions to this guidance document, please use the » Guideline Help to submit your question electronically to the QC Panel.
Please note, that a decision was taken, that any new versions of this Guidance Document should keep the document number (SANTE/11312/2021) and only get a new version number.
Current Version
✓ Document No. SANTE/11312/2021 (V2) (applying from 01/01/2024)
Quicklinks
EURL-DataPool
EURL Method-Finder List
EU MRLs-Database (COM)
EU Pesticides-DB - Main (COM)
EU-Legisl. on PPPs (COM)
EU Guidelines - MRLs
RASFF Portal DB (COM)
CIRCA BC Login
How to Use CIRCA BC
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Compilación de propuestas de cambios - EU

		Compilation 20241208		
Paragraph	Comment number	Comment	More information	Measure/comment
C14-19	1	During initial validation: how to assess/validate the linearity of the calibration curve? How to choose the best weighting function? Some labs apply the same criterion as in routine (BCC ≤ 20%), others use R^2 as a criterion (but may be tricky), others use statistical tests.		In this document, back calculated concentrations are used to predict true concentrations and to check the calibration function. If the deviation is more than ±20 % an alternative calibration function must be used. This approach (BCC) is considered more robust than the use of R^2. The document will be adjusted as regard "linearity" (C24, D9, D11 and Table 4) since in the present document, the BCC is the key requirement instead of to proof linearity.

Compilación de propuestas de cambios - EU

		Compilation 20241208		
Paragraph	Comment number	Comment	More information	Measure/comment
C21	2	Mixture of matrices for matrix matched calibration		Mixture of matrices for matrix matched calibration is not to be recommended. It might be an option for the first analysis. For second (confirmatory) analysis, in case of MRL exceedance, then exact matrix matching is needed.
C39	3	Verification of new automated workflow steps (or instruments). What if we could only make a small verification and skip the full validation if such methods are implemented in the labs? The same with new instruments in the lab, do we have to validate again? Maybe use PT verification or QC data (C39 onwards).		Appendix A (alt H. Additional recommendations) will be completed with recommendation for performance check to change the instrument. A survey will be sent to NRLs to gather information from accreditation bodies about existing policies in this matter.

Compilación de propuestas de cambios - EU

		Compilation 20241208		
Paragraph	Comment number	Comment	More information	Measure/comment
C21	2	Mixture of matrices for matrix matched calibration		Mixture of matrices for matrix matched calibration is not to be recommended. It might be an option for the first analysis. For second (confirmatory) analysis, in case of MRL exceedance, then exact matrix matching is needed.
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Propusimos que como la mayoría de veces los cambios son pequeños (etapa de limpieza, equipamiento nuevo), estaría bien tener unas recomendaciones sobre los controles de calidad a tener en cuenta, pero que según nuestro punto de vista sería suficiente con que los QC diarios pasasen los criterios aceptables o analizar PTs.

Compilación de propuestas de cambios - EU

		Compilation 20241208		
Paragraph	Comment number	Comment	More information	Measure/comment
E12	8	MU of 50% to apply to residue definition.	If there are several components, you can calculate the MR of individual ones, but how to calculate for the complex residue definition?	In case of complex residue definition, the individual analytes are measured separately including calculation of individual MU. For practical reasons, when summing up the MU of individual results the exp. MU should be lower or equal to 50%. For summing up the induvial analytes for residue definition, please see Appendix B A presentation on how to calculate the MU for SUM (complex RD) will be held at the meeting.

Compilación de propuestas de cambios - EU

		Compilation 20241208		
Paragraph	Comment number	Comment	More information	Measure/comment
F6	10	It´s written in the document that we can store stds up to 10 years, but are those 10 years from the expiry date of the supplier or from the purchase or opening of the std?		We think that the day when the laboratory receives the neat standard is a suitable starting point to check the stability. New tool to Data Pool is in progress in order to gather more data about stability.

Compilación de propuestas de cambios - EU

		Compilation 20241208		
Paragraph	Comment number	Comment	More information	Measure/comment
G1-G5	12	Give guidance on how to establish LOQs in cases where background levels do not allow validation at levels (e.g. variability of multiple injection should be low, linearity should be acceptable)		We might need different criteria for difficult analyte/matrix combinations? Repeatability at lowest level is the best measure. The topic will be discussed.
G2	13	Complex matrices, if there is no blank, how to validate at LOQ level?		Pls, see comment no 12 (reference C41)
Annex A	14	Unique commodities: pickled cucumbers, would it be possible to add a new commodity group for processed food?		New commodity group for processed food is not possible due to huge amount of possible commodity combinations. Processed food can be classified under the main commodity group i.e. pickled cucumbers are high water and acid (with acetic acid) resulting in group 2.

Compilación de propuestas de cambios - EU

		Compilation 20241208		
Paragraph	Comment number	Comment	More information	Measure/comment
Appendix A G6	16	Absolute recovery	It should be clarified, that the absolute recovery using matrix matched calibration must (should, shall) be in the range of 30-140 %. At the moment G6 says:„…but the mean absolute recovery should not be lower than 30 % or above 140 %“), or add a sentence in Appendix F Glossary Recovery (absolute) is determined via matrix matched calibration.	Pls see the revised text in Appendix A

Remark/Note: for calibration procedures that intrinsically correct/account for losses of analytes during extraction and/or cleanup (e.g. procedural calibration, ILIS or standard addition added before extraction/cleanup, see Appendix E, Table E1), it is required to assess the extraction/cleanup yield (absolute recovery) to verify it is within the range 30-140% (see E6).

The absolute recovery is obtained by a recovery experiment using matrix-matched calibration for quantification.

Compilación de propuestas de cambios - EU

		Compilation 20241208		
Paragraph	Comment number	Comment	More information	Measure/comment
Appendix A	19	In the example of the experimental set : repeatability or reproducibility conditions (or both)?		"Example experimental set" 1.Initial full validation ►repeatability 4. On going validation / performance verification ►within-laboratory reproducibility (RSD_{wR})

Compilación de propuestas de cambios - EU

		Compilation 20241208		
Paragraph	Comment number	Comment	More information	Measure/comment
Appendix A	19	In the example of repeatability or re conditions (or bot		"Example experimental set" .Initial full validation - repeatability . On going validation / performance verification - within-laboratory reproducibility (RSD_{wR})

Indicamos que en esta medida de precisión no quedaba claro que en la validación inicial se habla de la repetibilidad, por lo que pedimos que se especificase mejor el texto en el apéndice A,

Compilación de propuestas de cambios - EU

		Compilation 20241208		
Paragraph	Comment number	Comment	More information	Measure/comment
	25	Copper analysis ICPMS: there is no guidance for ID, validation, MU….		Analysis of copper will be presents at the Joint meeting.

Comentarios ES a los cambios de la UE

20250402				
Paragraph	Comment number	Comment	More information	Measure/comment
Appendix A	NEW	In the example of the experimental set a reagent blank is specified as one of the samples to extract. Reagent blank could be deleted.	Reagent blank is not necessary at the first step of the initial validation as long as the blank sample is not contaminated.	

Propuestas de cambios - ES

20250402				
Paragraph	Comment number	Comment	More information	Measure/comment
Section 3 and 4 of Appendix A	NEW	It is necessary to specify the acceptance criteria for extension of the scope of the method for new matrices.	For initial validation of the representative matrix of a commodity group, 5 replicates for recovery are necessary with an acceptance criteria range of 70-120%. But for including new matrices to that commodity group, usually only one recovery in that matrix is carried out with the sample set. In that case, as the uncertainty is higher, the acceptance criteria should be the specified in paragraph C43 (60-140%), but this paragraph is intended for routine recoveries. Section 3 of appendix A (extension of the scope of the method: new matrices) lead us to section 4, which indicates that the acceptance criteria are those set in Table 4 (70-120%). If a mean recovery is needed even when on-going validation is used, how many data are required? For some routine labs it could take too much time, by that it is made mostly with only one data (one recovery at the LOQ). So, we ask to specify at least in Appendix A that for extension of new matrices, when n=1 the acceptance criteria should be 60-140% and for n>1 should be 70-120%. The Spanish Accreditation Body (ENAC) interprets that the acceptance criteria for validation recovery must be the same both for initial validation and for complementary validation (extension to new matrices of the same commodity group). As long as this extension can be achieved with routine recovery data (on-going validation), the recovery is usually performed once. So, the uncertainty is higher and there are a lot of cases that would be accepted as routine recovery checks (criteria 60-140%), but don't match the 70-120% range. We would like to ask a clarification on how to deal with this. That is, an explicit numerical range that takes this higher uncertainty into account.	

Propuestas de cambios - ES

20250402				
Paragraph	Comment number	Comment	More information	Measure/comment
F8	11	Stability of stock solutions. There are different approaches among the labs to check stabilities, in some cases depending on the accreditation bodies. Suggestion to have some rules for checking the stock solutions.	Based on our experience with more than 400 pesticides, fulfilling the 10% variation for all of them is very restrictive. May the advisory group consider setting a new limit of 15%? In the case of our lab, the stability is checked every 3 months and some of the solutions are prepared fresh every 3 months and even like that sometimes being below 10% may be an issue.	

Propuestas de cambios - ES

20250402				
Paragraph	Comment number	Comment	More information	Measure/comment
Appendix A G6	16	Absolute recovery. It should be clarified, that the absolute recovery using matrix matched calibration must (should, shall) be in the range of 30-140 %. At the moment G6 says:"…but the mean absolute recovery should not be lower than 30 % or above 140 %"), or add a sentence in Appendix F Glossary Recovery (absolute) is determined via matrix matched calibration.	We do not agree with the new remark on the calculation of the absolute recovery when working with procedural calibration. As long as it can be ensured that the quality control criteria are met during the extraction and the LOQ is detectable for the analytes working with procedural calibration, the absolute recovery has no meaning, as that is not how routine work will be carried out. This means an extra work during validation that is not worthy. If the absolute recovery must be done, it will be nice an explanation of the reasons.	

GRACIAS POR SU ATENCIÓN

#alimentosdespaña

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