SLOVENSKI STANDARD
SIST EN 18032:2025

01-december-2025

Živila - Hitra metoda za analizo več visokopolarnih pesticidov in njihovih metabolitov v živilih z ekstrakcijo z zakisanim metanolom in merjenjem z LC- ali IC-MS/MS (metoda QuPPe)

Foodstuff - Quick Method for the Analysis of Multiple Highly Polar Pesticides and their Metabolites in Foodstuff Involving Extraction with Acidified Methanol and Measurement by LC- or IC-MS/MS (QuPPe-Method)

Lebensmittel - Schnellmethode zur Bestimmung mehrerer hochpolarer Pestizide und ihrer Metaboliten in Lebensmitteln nach Extraktion mit angesäuertem Methanol und Messung mittels LC- oder IC-MS/MS (QuPPe-Methode)

Produit alimentaire - Méthode rapide pour l'analyse de plusieurs pesticides hautement polaires et de leurs métabolites dans les aliments impliquant une extraction avec du méthanol acidifié et une mesure par LC- ou IC-MS/MS (QuPPe-Methode)

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Ta slovenski standard je istoveten z: **EN 18032:2025**

ICS:

67.050	Splošne preskusne in analizne metode za živilske proizvode	General methods of tests and analysis for food products

SIST EN 18032:2025 **en,fr,de**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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SIST EN 18032:2025

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 18032

October 2025

ICS 67.050

English Version

Foodstuff - Quick Method for the Analysis of Multiple Highly Polar Pesticides and their Metabolites in Foodstuff Involving Extraction with Acidified Methanol and Measurement by LC- or IC-MS/MS (QuPPe-Method)

Produits alimentaires - Méthode rapide pour l'analyse de plusieurs pesticides hautement polaires et de leurs métabolites dans les aliments impliquant une extraction avec du méthanol acidifié et une analyse par LC- ou CI-SM/SM (méthode QuPPe)

Lebensmittel - Schnellmethode zur Bestimmung mehrerer hochpolarer Pestizide und ihrer Metaboliten in Lebensmitteln nach Extraktion mit angesäuertem Methanol und Messung mittels LC- oder IC-MS/MS (QuPPe-Methode)

This European Standard was approved by CEN on 11 August 2025.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (EN 18032:2025) has been prepared by Technical Committee CEN/TC275 “Food analysis - Horizontal methods”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2026, and conflicting national standards shall be withdrawn at the latest by April 2026.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

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1 Scope

This document specifies a procedure for the analysis of residues of highly polar pesticides and metabolites, which are not amenable to common multiresidue methods, in various food commodities of plant and animal origin, including fruits, vegetables, cereals, pulses, oily seeds, nuts, milk, liver and honey. The method was developed at the EURL-SRM hosted at CVUA Stuttgart [1], [2], [3] and has been collaboratively studied on a large number of commodity/pesticide combinations. Guidelines for calibration are outlined in CEN/TS 17061:2019.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

CEN/TS 17061:2019, *Foodstuffs - Guidelines for the calibration and quantitative determination of pesticide residues and organic contaminants using chromatographic methods*

SANTE/11312/2021v2,[1] *Analytical quality control and method validation procedures for pesticide residues analysis in food and feed*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in SANTE/11312/2021v2 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp/

— IEC Electropedia: available at https://www.electropedia.org/

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4 Principle

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Residues are extracted from the homogeneous test portion following water adjustment and addition of acidified methanol. In the case of fruits and vegetables, the mixture is centrifuged, filtered and directly analysed by LC-MS/MS or IC-MS/MS. In the case of cereals, pulses, nuts, oily seeds and foods of animal origin, EDTA is added for the complexation of metal ions, such as calcium and magnesium, which positively affects the analysis of certain compounds (e.g. glyphosate and AMPA). As such commodities also contain a substantial content of protein, they are additionally diluted with acetonitrile to precipitate proteins. Samples with high lipid content are subjected to dispersive SPE with C18-sorbent. Various LC- and IC-MS/MS methods, allowing simultaneous analysis of different combinations of pesticides, are provided in this document. Quantification is performed employing isotope-labelled analogues of the target analytes as internal standards (IL-ISs), so far these are available. When adding IL-ISs directly to the test portion at the beginning of the procedure, they compensate for any factors having an influence on recovery-rates, such as volume-deviations and analyte losses during sample preparation. Matrix-effects during measurement are also corrected. The use of IL-ISs, ensures good accuracy and reproducibility. Quantification without IL-ISs is possible, but careful water adjustment and other approaches for addressing recovery losses and matrix effects are required in this case. The analytical procedure entails few working steps and involves little material consumption. A brief overview of the method is shown in the flowcharts within the “On-line Supplement” linked in Annex C. Abbreviations and symbols are listed in Annex D.

[1] Version 2 of 2021. Implemented as of 01/01/2024.
Available at: https://www.eurl-pesticides.eu/docs/public/tmplt_article.asp?CntID=727

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5 Preparation and storage of the samples

5.1 General considerations

Sample processing and storage procedures should be demonstrated to have no significant effect on the residues present in the test sample (sometimes also called “analytical sample”). Processing should also ensure, that the test sample is homogeneous enough so that sub-sampling (portion-to-portion) variability is acceptable. If a single analytical portion is unlikely to be representative of the test sample, larger or replicate portions shall be analysed, to provide a better estimate of the true value. The degree of comminution should support a quantitative residue extraction, otherwise, extraction shall involve supplementary comminution, e.g. through a homogenizing device (A.2.2) or grinding aids (e.g. metal balls (A.2.3)).

5.2 Laboratory sample

A laboratory sample is the sample arriving to the laboratory for analysis and should ideally be sampled according to international sampling protocols [4], [5]. A laboratory sample that is extensively spoiled or degraded should not be analysed. Samples associated with a shelf life should be analysed within their stated shelf life. If possible, process laboratory samples immediately after arrival and in any event, before any significant physical or chemical changes have taken place. If a laboratory sample cannot be processed without delay, it should be stored under appropriate conditions to keep it fresh and to minimize deterioration.

If the laboratory sample is in a state that does not require milling prior to analysis (e.g. juices, milk, and cereal flour), stir or shake the sample well and then withdraw the analytical test portions directly. Where the homogeneity of the sample is, however, not sufficient or the extraction of residues is expected to be significantly compromised due to the presence of larger particles, intensive comminution should be performed using appropriate means.

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5.3 Treatment of laboratory samples prior to milling

For preparation of the analytical sample, take only the portion of the laboratory sample to which the maximum residue levels apply [6], [7]. If a reduction of the laboratory sample is required, out of practical reasons, it shall be carried out in a way ensuring representativeness. This, for example, applies when samples are made up of larger units, and the capacity of the available mixer is too small to process the required number of units in one go. In this case, parts of the sample can be used, i.e. wedge-shaped sections (e.g. melons) or cross sections (e.g. cucumbers) that include the skin (outer surface). Opposite sections from each unit (e.g. quarters) should then be used for mixing (see e.g. [8]). For samples of small units (e.g. small fruits such as berries, legumes, cereals), the sample shall be thoroughly mixed before taking any aliquot for further processing.

Any parts that would cause difficulties with the homogenization process may be removed prior to milling (e.g. in the case of stone fruits, the stones). The mass of the sample before and after removing the interfering sample portions shall be recorded. Precautions should be taken to avoid any losses of juice or flesh. The sample obtained in this way is referred to as the test sample. Calculation of the residue shall be based on the mass of the original test sample (including the stones and assuming that these parts are residue free).

In case of cryogenic comminution using dry ice, cutting the samples coarsely (e.g. 3 × 3 cm) with a knife and putting them into the freezer (e.g. at ≤ −18 °C overnight) prior to comminution facilitates processing.