SLOVENSKI STANDARD
SIST EN 18082:2026

01-maj-2026

Živila živalskega izvora - Multimetoda za določanje ostankov pesticidov z uporabo LC-analize po acetonitrilni ekstrakciji/ločevanju in čiščenju z disperzijsko SPE

Foods of animal origin - Multimethod for the determination of pesticide residues using LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE

Tierische Lebensmittel - Multiverfahren zur Bestimmung von Pestizidrückständen mit LC nach Acetonitril-Extraktion/Verteilung und Reinigung mit dispersiver SPE

Aliments d'origine animale - Multiméthode de détermination des résidus de pesticides par analyse CL après extraction/partition avec de l'acétonitrile et purification par SPE dispersive

Ta slovenski standard je istoveten z: **EN 18082:2026**

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Foods of animal origin - Multimethod for the determination of pesticide residues using LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE

Aliments d'origine animale - Multiméthode de détermination des résidus de pesticides par analyse CL après extraction/partition avec de l'acétonitrile et purification par SPE dispersive

Tierische Lebensmittel - Multiverfahren zur Bestimmung von Pestizidrückständen mit LC nach Acetonitril-Extraktion/Verteilung und Reinigung mit dispersiver SPE

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Page

European foreword

This document (EN 18082:2026) has been prepared by Technical Committee CEN/TC 275 “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of an identical text or by endorsement, at the latest by September 2026, and conflicting national standards shall be withdrawn at the latest by September 2026.

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1 Scope

This document specifies a method for the analysis of pesticide residues in foods of animal origin with a low fat content, such as meat/muscle, egg or milk by LC-MS/MS. Because of the low material requirements for miniaturized processing and the few work steps, the process is particularly time and cost-saving with high reliability and effectiveness. The method has been collaboratively studied on a number of commodity/pesticide combinations. Precision data are summarized in Table B.1. 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*

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Principle

Water is added to the homogeneous sample and is extracted with the help of acetonitrile. After addition of magnesium sulfate, sodium chloride and buffering citrate salts, the mixture is shaken intensively and centrifuged for phase separation. An aliquot of the organic phase is cleaned-up by dispersive solid phase extraction (D-SPE) with amino-sorbents (e.g. primary secondary amine sorbent, PSA) as well as magnesium sulfate for the removal of residual water. Following clean-up extracts are centrifuged. Fat contained in the sample is removed by freezing out by storing the purified acetonitrile extract in the freezer. The pesticides contained in the fat-free solution can be determined directly by LC-based analysis. For the analysis with LC hyphenations with tandem mass-spectrometry (LC-MS/MS) or high resolution mass-spectrometry (LC-HR-MS) are particularly suitable. Quantification may be performed using an internal standard, which is added to the test portion before the first extraction, but this is not mandatory. Details for calibration, see 7.2. Abbreviations and symbols are listed in Annex C.

5 Preparation and storage of the samples

5.1 General

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 portion to portion (sub-sampling) 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, the extraction shall be carried out with the aid of a mechanical shredding device (e.g. a homogenizing rod).

5.2 Laboratory sample

A laboratory sample that is wholly or extensively spoiled or degraded should not be analysed. When possible, prepare 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 prepared without delay, it should be stored under appropriate conditions to keep it fresh and to avoid deterioration. In general, laboratory samples should not be stored for more than 3 days in total, with the exception of dried or processed samples that are to be analysed within the specified minimum shelf life.

5.3 Partly-prepared test sample

For preparation of the partly-prepared test sample take only the portion of the laboratory sample to which the maximum residue level applies, e.g. bones or shells should be removed.

5.4 Test sample

Where the homogeneity of the test sample is not sufficient or the extraction of residues could be significantly compromised due to large particle sizes, intensive comminution should be performed using appropriate means. This is possible at ambient temperature, if separation of flesh and juice or degradation of target pesticides does not occur to a significant extent. Comminution of samples in a frozen state can significantly reduce losses of chemically labile pesticides and usually results in smaller particle sizes and a higher degree of homogeneity. Cutting the samples coarsely (e.g. 3 cm × 3 cm) with a knife and putting them into the freezer (e.g. −18 °C overnight) prior to comminution facilitates processing. Processing can be also assisted and improved by cryogenic milling (using dry ice or liquid nitrogen) by keeping the temperature below 0 °C. When processing test samples at low temperatures, condensation caused by high humidity should be avoided. Residual carbon dioxide should be allowed to sufficiently dissipate so that its contribution to weigh of the sample will be negligible.

5.5 Test portion

Individual test portions each sufficient for one analysis should be taken from the comminuted test sample. These test portions should be analysed immediately. If test portions cannot be analysed directly, the test sample or the test portions shall be frozen until required. If it is noted that homogeneity of the test sample has been compromised during storage, the test sample shall be mixed before taking test portions to ensure that homogeneity has been re-established.

6 Procedure

Extraction of samples is specified in module E. Extraction is usually followed by a clean-up of the obtained raw extracts (Freezing out of the fat module C1 and /or cleaning with PSA module C2) and analysed by module D. All modules are described in detail in Annex A. Complementary information is given in Annex B.

Table 1 contains brief descriptions of the modules as well as application notes and examples of use. For the calculation of residue concentrations in the sample extracts all of the calibration procedures and quantification methods described in options Q1 to Q7 are applicable.

Table 1 — Overview of possible modules

Module	Description	Preferred application
E	After addition of water a test portion of 5 g is extracted with acetonitrile in the presence of buffering salts	Meat, milk and eggs with a fat content of up to approx. 10 %
C1	Freezing-out	Removal of co-extracted fat (even in combination with further clean-up steps, e.g. C2)
C2	Dispersive SPE with amino-sorbent (PSA)	Clean-up of raw-extracts prior to the determination of basic and neutral pesticides
D	LC-MS	Extracts from module E subsequently cleaned-up with modules C1 to C2
Q1	Quantification using external standards in solvent	Determinations where matrix-effects are assumed to be negligible
Q2	Quantification using external standards in matrix	Determinations where matrix-effects shall be considered
Q3	Quantification using a procedural internal standard and standards in solvent	Determinations where matrix-effects are assumed to be negligible
Q4	Quantification using standard addition to the final extract	Determinations where matrix effects shall be considered and suitable blank matrices are not available
Q5	Quantification using a procedural internal standard and standards in matrix or isotope-labelled internal standards	Determinations where matrix-effects shall be considered for compensation of low recovery
Q6	Quantification using standard addition to the sample	Determinations where matrix-effects shall be considered without availability of blank (control) samples or incomplete extractions of the analyte occur
Q7	Quantification by calibration of the entire procedure	Determinations where matrix-effects shall be considered or incomplete extractions of the analyte occur

7 Evaluation of results

7.1 Identification and quantification

A number of parameters can be employed to determine the identity of an analyte present in the sample extract. This includes:

— retention time of the analyte in question (R_t) or, even better, the retention time ratio against the ISTD ($R_{t(A)}/R_{t(ISTD)}$) obtained from the same run;

— in case of MS or MS/MS detection, the relative abundance of the recorded masses or transitions respectively (in general 2 selected reaction monitoring (SRM) transitions are required in MS/MS and 3 ions in MS applications), see also [1], [2], [3], [4];

— characteristic peak shape/pattern of the analyte.

The parameters obtained for the analyte to be identified in the sample extract are compared with those obtained for the pesticides in the calibration solution(s). Should a higher degree of certainty be required for the confirmation of the analyte identity, additional measures can be necessary, such as the use of different chromatographic separation conditions or the evaluation of additional *m/z* or SRM transitions. For more information about the required confirmation criteria (e.g. the recommended maximum tolerances for ion ratios using different MS techniques) see the guidelines described in EU Quality Control Procedures [2]. Table A.1 gives a list of the ISTDs that can be employed. The use of more than one ISTD will provide some backup information.

Use standard solutions to check linearity and to determine the calibration functions for each analyte. The use of matrix-matched standards is to be preferred, however, for a first estimate of the residue level of pesticides in the food or to show their absence, the standard solutions in pure solvent can be used. They can be also used for quantification if preliminary experiments indicate that any suppression or enhancement effects experienced do not significantly affect the results obtained. As soon as relevant residue concentrations are detected (e.g. suspected maximum residue level (MRL) violations), a more precise determination using matrix-matched standards or the standard addition method should be used.

NOTE Matrix effects influence the response of target analytes in sample extracts compared to the response of standard solutions in pure solvent.

The calibration range should be appropriate to the residue concentrations to be quantified. Thus, it may be necessary to construct more than one calibration graph from the results of calibration measurements.

This document contains the option to use an internal standard for quantification and identification. Nevertheless, it is still possible to quantify without ISTD. Without ISTD, the volume of the acetonitrile phase is assumed to be identical to the volume of acetonitrile added to the sample (10 ml).

In this method, the internal standard is basically used for process control of sample preparation, but by default it is not used for quantification. However, there is the option to use an internal standard for quantification, for which the validity shall be ensured.

7.2 Calibration

The analytical method shall be calibrated according to CEN/TS 17061:2019. In addition, it may be calibrated according to the EU Quality Control Procedures [2]. A suitable calibration procedure should be selected from one of the quantification options Q1 to Q7 in A.6.

7.3 Calculation of residue concentration

The mass fraction w_A of each identified analyte depends on the mass concentration of the sample in the final extract $\rho_{\text{sample}}^{\text{final extract}}$ after application of clean-up (modules C1 to C2) and the concentration ρ_A of the substance in this extract (determined according to one of the options Q1 to Q7 given in A.6). It is expressed in mg/kg and is calculated by Formula (1).

$$w_A = \frac{\rho_A}{\rho_{\text{sample}}^{\text{final extract}}} \quad (1)$$

where

ρ_A is the mass concentration of the analyte in the sample extract (option Q, see A.6.1.3, A.6.2.3, A.6.3.3, A.6.5.3, and A.6.7.3) in µg/ml;

$\rho_{\text{sample}}^{\text{final extract}}$ is the mass concentration of the sample in the final extract (modules C, see A.4.1.3, A.4.2.3), in g/ml.

7.4 Validity of the method

The recoveries obtained from experiments (spiking levels 0,01 mg/kg or 0,05 mg/kg to 0,1 mg/kg) were usually between 70 % and 110 %.

Interlaboratory method validation studies covered a multitude of analytes using representative commodities (meat, egg and milk). Furthermore, extensive individual validation has been carried out. All validation data provided by laboratories are published in the Data Pool of the European Union Reference Laboratories [3].

Validity of the method is confirmed for any specific commodity/pesticide combination if at least four laboratories conducted independently validation studies with the same matrix at two fortification levels with at least five replicates per level and obtained a recovery between 70 % and 120 %. Furthermore, the relative standard deviation had to be below or equal 20 % for both spiking levels in each laboratory.

For the respective calculation of the relative standard deviation, each laboratory had to have at least 5 additional tests for each concentration level. Based on these conditions, it was found that sufficient validation data are available for 41 pesticides or pesticide metabolites. An overview is shown in Table 2. In addition to the active substances listed in Table 2, fenpropimorph, the metabolites flusilazole-IN-F7321 and ioxynil octanoate were also investigated. These substances could not be validly analysed with the presented method.

Detailed validation results are given in Table B.1. This table also contains successful validations which have not been carried out by at least four laboratories for the same analyte-matrix combination or for which values are not available for at least two concentration levels. Table B.1 contains also validation data for analyte-matrix combinations with average recoveries and/or RSD outside the acceptable range. This data should be considered as supportive information.

The limit of quantification of an analyte depends on the performance of the analytical equipment used and the sample matrix. With modern analytical equipment, residues at 0,01 mg/kg (in most cases the lowest maximum residue level) can typically be analysed (see Table B.1).

Table 2 shows the confirmed validation data of the method for specific analytes and commodity groups.

Table 2 — Validation data

No.	Analyte	CAS-No.	Commodity group (see [2])		
			Meat[a] (muscle) and seafood	Milk and milk products[b]	Eggs
1	Azinphos-ethyl	2642–71–9	X	X	X
2	Bifenthrin	82657–04–3	X	X	–
3	Bixafen	581809–46–3	X	X	X
4	Boscalid	188425–85–6	X	X	X
5	Boscalid-M 510F01	-	X	X	X
6	Carbendazim	10605–21–7	X	X	X
7	Chlorpyrifos	2921–88–2	X	X	–
8	Chlorpyrifos-methyl	5598–13–0	X	X	–
9	Cyfluthrin	68359–37–5	X	X	–
10	Cypermethrin	52315–07–8	X	X	X
11	Deltamethrin	52918–63–5	X	X	–
12	Diazinon	333–41–5	X	X	X
13	Etofenprox	80844–07–1	X	X	–
14	Famoxadone	131807–57–3	X	X	X
15	Fenthion	55–38–9	X	X	X
16	Fenthion-oxon	6552–12–1	X	X	X
17	Fenthion-oxon-sulfone	14086–35–2	X	X	X
18	Fenthion-oxon-sulfoxid	6552–13–2	X	X	X
19	Fenthion-sulfone	3761–42–0	X	X	X
20	Fenthion-sulfoxid	3761–41–9	X	X	X
21	Fenvalerate/Esfenvalerate	51630–58–1	X	X	–
22	Fluopyram	658066–35–4	X	X	X
23	Fluquinconazole	136426–54–5	X	X	X
24	Flusilazole	85509–19–9	X	X	X
25	Indoxacarb	144171–61–9	X	X	X
26	Ioxynil	1689–83–4	X	X	X
27	Metaflumizone	139968–49–3	X	X	X
28	Methidathion	950–37–8	X	X	X

No.	Analyte	CAS-No.	Commodity group (see [2])		
			Meat[a] (muscle) and seafood	Milk and milk products[b]	Eggs
29	Paraoxon-methyl	950–35–6	X	X	X
30	Parathion-methyl	298–00–0	X	X	X
31	Permethrin	52645–53–1	X	X	–
32	Pirimiphos-methyl	29232–93–7	X	X	X
33	Prochloraz	67747–09–5	X	X	X
34	Profenofos	41198–08–7	X	X	X
35	Pyrazophos	13457–18–6	X	X	X
36	Resmethrin	10453–86–8	X	X	–
37	Spiroxamine	118134–30–8	X	X	X
38	Tau-Fluvalinate	102851–06–9	X	X	X
39	Tetraconazole	112281–77–3	X	X	X
40	Thiophanate-methyl	23564–05–8	X	X	X
41	Triazophos	24017–47–8	X	X	X

[a] Pork with a fat content of 10 % was used for the validation.

[b] Milk with a fat content of 3,8 % was used for the validation.

8 Precision

Details of the inter-laboratory test and the precision of the method are summarized in Table B.1. The values derived from the inter-laboratory test are not applicable to pesticide concentration ranges and commodities other than those given in Table B.1.

9 Test report

The test report shall contain at least the following:

— all information necessary for the identification of the sample;

— a reference to this document (including its year of publication);

— the results and the units in which the results have been expressed;

— the date and type of sampling procedure (if possible);

— the date of receipt of sample in the laboratory;

— the date of test;

— any particular observations made in the course of the test;

— any operations not specified in the method or regarded as optional which might have affected the results.