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BSI Standards Publication

Foodstuffs — Determination of patulin in fruit juice and fruit based purée for infants and young children — HPLC method with liquid/liquid partition cleanup and solid phase extraction and UV detection

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee AW/-/3, Food analysis - Horizontal methods.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Foodstuffs - Determination of patulin in fruit juice and fruit based purée for infants and young children - HPLC method with liquid/liquid partition cleanup and solid phase extraction and UV detection

Denrées alimentaires - Dosage de la patuline dans le jus de fruits et la compote de fruits en alimentation infantile - Méthode par CLHP avec purification par partition liquide-liquide et extraction en phase solide et détection UV

Lebensmittel - Bestimmung von Patulin in Fruchtsaft und Obstbrei für Säuglinge und Kleinkinder - HPLC-Verfahren mit Reinigung durch Flüssig/Flüssig-Verteilung, Festphasenextraktion und UV-Detektion

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Contents

Page

Foreword

This document (EN 15890:2010) 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of an identical text or by endorsement, at the latest by March 2011, and conflicting national standards shall be withdrawn at the latest by March 2011.

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WARNING — The use of this standard can involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This European Standard specifies a method for the determination of patulin in fruit juices and fruit-based purée, such as baby food purée, using high performance liquid chromatography with ultra-violet detection (HPLC-UV). Using naturally contaminated and spiked samples this method has been validated for the determination of patulin in apple juice, at levels ranging from 3,0 µg/kg to 15,5 µg/kg, and in fruit-based baby food purée, at levels ranging from 3,4 µg/kg to 17,9 µg/kg. Baby food fruit purée used in this study contained a mixture of the following ingredients which are commercially available on the European market: blueberry; apple; banana; lemon; wheat biscuits; wheat syrup; whole milk; and vegetable oil. A detailed listing, including the fractions, of each product used in this study is given in [1].

Further information on validation, see Clause 9 and Annex B.

2 Normative references

The following referenced documents are indispensable for the application 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.

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Principle

Patulin is extracted from apple juice, or fruit-based purée, with a mixture of ethyl-acetate and hexane in the presence of sodium sulfate and sodium hydrogen carbonate. An aliquot of the extract is purified by solid-phase extraction and evaporated. The residue is re-dissolved in water of pH = 4 and patulin is separated by reverse phase (RP)-HPLC and quantitatively determined by UV detection.

4 Reagents

4.1 General

Use only reagents of recognized analytical grade and water complying with grade 1 of EN ISO 3696:1995, unless otherwise specified. Solvents shall be of quality for HPLC analysis, unless otherwise specified. Commercially available solutions with equivalent properties to the reagents listed may be used.

4.2 Perchloric acid, the mass fraction $w(HClO_4) \geq 60$ % in water.

4.3 Sand, 50 mesh to 70 mesh particle size.

4.4 Silicagel solid phase extraction (SPE) cartridges (500 mg SiO_2).

4.5 Sodium sulfate anhydrous, Na_2SO_4.

4.6 Sodium hydrogen carbonate, $NaHCO_3$.

4.7 Glacial acetic acid, $w(CH_3COOH) \approx 98$ % in water.

4.8 Water of pH = 4.

Adjust water to pH = 4 with glacial acetic acid (4.7).

4.9 Absolute ethanol, $w(CH_3CH_2OH) \geq 99{,}7$ % in water.

4.10 Acetonitrile.

WARNING — Acetonitrile is hazardous and samples shall be blended using an explosion proof blender which is housed within a fume cupboard. After blending, samples shall be filtered inside a fume cupboard.

4.11 Ethyl acetate.

4.12 *n*-Hexane.

4.13 Extraction solvent.

Add 60 ml of ethyl acetate (4.11) to 40 ml of *n*-hexane (4.12).

4.14 Mixture of glacial acetic acid and ethyl acetate.

Add 3 ml of glacial acetic acid (4.7) to 97 ml of ethyl acetate (4.11).

4.15 HPLC mobile phase.

Mix 990 parts per volume of water with up to ten parts per volume of acetonitrile (4.10) and one part per volume of perchloric acid (4.2). The amount of acetonitrile will depend upon the type of samples analysed and their characteristic pattern of interferences after clean-up (see Annex A for typical chromatograms) and the HPLC column chosen for analysis. Degas this solution before use.

NOTE A mobile phase of 990 part of water with one part of perchloric acid has been found to give sufficient separation between patulin and other interfering substances (in particular 5-hydroxymethylfurfural when used in combination with a Synergy® [1)] column of 250 mm length and 4,6 mm diameter with a particle size of 4 µm and 8 nm porosity (see 5.13.4)).

4.16 Patulin.

WARNING — Patulin is a suspect mutagen and has been reported to have immunotoxic and neurotoxic properties. Gloves and safety glasses should be worn at all times and all standard and sample preparation stages should be carried out in a fume cupboard.

4.17 Patulin stock solution.

Dissolve 5 mg of patulin or the contents of one ampoule (if patulin has been obtained as a film) in ethyl acetate (4.11). Transfer the solution to a 25 ml volumetric flask and dilute to volume with ethyl acetate to produce a solution containing approximately 200 µg/ml of patulin.

1) Synergy® is a trade name of a suitable product available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.

Store this solution in a freezer at approximately - 18 °C. Confirm the mass concentration of the solution if it is older than six weeks. Ensure the solution is allowed to reach room temperature before use to avoid incorporation of water by condensation.

4.18 Patulin standard solution.

Evaporate 1 000 µl of the stock solution (4.17) to dryness under nitrogen and then immediately dissolve it in 20 ml of ethanol (4.9) to obtain a mass concentration of approximately 10 µg/ml of patulin.

To determine the exact mass concentration, record the absorption curve between 250 nm and 350 nm in a 1 cm quartz cell with ethanol as reference. Identify the wavelength for maximum absorption. Calculate the mass concentration of patulin, ρ_{pat}, in micrograms per millilitre, using Equation 1:

$$\rho_{pat} = \frac{A_{max} \times M \times 100}{\varepsilon \times b} \quad (1)$$

where

A_{max} is the absorption determined at the maximum of the absorption curve (here: at approximately 276 nm);

M is the molar mass, in grams per mole, of patulin (M = 154 g/mol);

ε is the molar absorption coefficient, in square metres per mole, of patulin in ethanol (here: 1 460 m^2/mol, see [2]);

b is the optical path length, in centimetres, of the quartz cell.

Store this solution in a freezer at approximately - 18 °C. A solution stored in this way is stable for several months. Ensure that the standard solution is allowed to reach room temperature before use to avoid incorporation of water by condensation. Confirm the concentration of the solution if it is older than six weeks.

4.19 Spiking solutions.

For spiking experiments at levels of 10 ng/ml and 25 ng/ml patulin in the sample, prepare spiking solutions of patulin in water of pH = 4 (4.8) at mass concentrations of 200 ng/ml and 500 ng/ml, respectively.

These solutions can be obtained by evaporating exactly 100 µl and 250 µl respectively of the stock solution (4.17) to dryness under nitrogen in a 100 ml volumetric flask, followed by immediate dissolution in water of pH = 4 (4.8) to obtain a mass concentration of approximately 200 ng/ml and 500 ng/ml respectively of patulin, depending on the exact mass concentration of patulin in the stock solution. Make sure that the patulin is completely dissolved in the water of pH = 4 before the volumetric flask is filled up to the mark.

In case the patulin standard solution (4.18) has a different mass concentration than 10 µg/ml, adjust spiking solutions by calculating the correct aliquots in order to take account of the actual mass concentration of the standard solution determined in 4.18.

Store this solution in a refrigerator at 4 °C. A solution stored in this way is stable for at least eight weeks.

5 Apparatus

5.1 General

Usual laboratory apparatus and, in particular, the following.

5.2 Displacement pipettes, of e.g. 5 ml, 1 ml , 200 µl and 50 µl capacity with appropriate pipette tips.

5.3 Analytical balance, capable of weighing to 0,1 mg.

5.4 UV spectrometer, double beam and recording suitable for measurement at 250 nm to 350 nm.

5.5 Quartz cells, with an optical path length of 1 cm.

5.6 Centrifuge, capable of operating at 400 *g*.

5.7 Centrifuge tubes, of 25 ml capacity with screw cap lids.

5.8 Mechanical shaker.

5.9 Evaporation block, capable of maintaining a temperature of 40 °C, with nitrogen supply.

5.10 Glass vial, of 6 ml capacity with screw cap.

5.11 Syringe, gas tight with a polytetrafluoroethylene (PTFE) plunger and with a volume of 3 ml to 5 ml.

5.12 Disposable syringe filters, of 0,2 µm pore size (optional).

Test each batch before use to ensure that patulin is not adsorbed onto the filter.

5.13 HPLC apparatus, comprising the following:

5.13.1 Injection system, a valve injection system with a 200 µl injection loop.

5.13.2 Pump, isocratic, pulse free, capable of maintaining a volume flow rate of 1 ml/min.

5.13.3 UV detector, fitted with an analytical flow cell and set at 276 nm.

5.13.4 Analytical reverse-phase HPLC separating column, capable to run with 100 % water as mobile phase such as a polar end-capped or polar embedded alkyl phases (for example columns of the type Synergy® [2], Atlantis® [2] or Luna® [2] or similar).

The column dimensions may vary depending on the obtained peak separation of patulin from interfering peaks such as 5-hydroxymethylfurfural (5-HMF). The maximum height of overlapping peak shoulders shall be less than 10 % of the maximum peak height. It could be necessary to adjust the mobile phase for sufficient baseline resolution. A suitable pre-column should be used.

Columns with a length of 250 mm, an inner diameter of 4,6 mm and a particle size of approximately 4 µm, with an 8 nm porosity have been shown to be suitable to meet these requirements when used in combination with the mobile phase proposed the Note under 4.15.

5.13.5 Data system.

2) Synergy®, Atlantis® and Luna® are trade names of suitable products available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5.13.6 Switching valve or second HPLC pump, optional, to allow washing of analytical column between injections.

6 Procedure

6.1 Extraction

To a clean centrifuge tube (5.7) add 2 g of sand (4.3), 15 g of sodium sulfate (4.5), 2 g of sodium hydrogen carbonate (4.6) and shake. Add 10 ml (V_1) of the extraction solvent (4.13) to the prepared tube and close tightly. In this way the centrifuge tubes can be prepared in advance prior to analysis.

Weigh to the nearest 0,1 g, 10 g of sample and transfer it into the prepared centrifuge tube and shake vigorously by hand for a few seconds, and then for 5 min to 6 min on a mechanical shaker (5.8). Subsequently centrifuge the extraction mixture at low speed (about 50 *g* to 100 *g* depending on the type and stability of centrifuge tube in use) for 30 s to force layer separation.

Patulin is not stable in alkaline solutions (e.g. sodium hydrogen carbonate), therefore this stage of the method shall be carried out as quickly as possible to avoid any losses.

6.2 Solid phase extraction cleanup

This step is to remove interfering acidic compounds in the sample. Add 50 µl of the mixture of acetic acid and ethyl acetate (4.14) to a 6 ml glass vial with screw cap (5.10) and place it under an unconditioned silicagel SPE column (4.4). Immediately transfer exactly 2,50 ml (V_2) of the centrifuged extract onto the unconditioned SPE column (4.4). Collect the eluate in the glass vial (5.10) at a speed of one drop per second (e.g. by gravity or slight air pressure). Immediately wash the SPE column with 3 ml of the extraction solvent (4.13) to elute the patulin-containing extractant quantitatively from the column. When most of the eluent has passed through, push with an air filled syringe the remaining solvent from the column into the vial.

Evaporate the collected eluate at maximum 40 °C under a gentle stream of nitrogen just to dryness using the evaporation block (5.9). This should not take longer than 10 min. Do not prolong the evaporation after having obtained dryness, in order to avoid any patulin losses. Add 1 ml (V_3) of water of pH = 4 (4.8) to the vial (5.10) and close it with the screw cap. Shake (or vortex) the vial for at least 3 min to ensure that the patulin is fully re-dissolved. If the solution is clear, transfer it directly into a suitable injector vial (e.g. 2 ml or smaller) and inject, or when turbid, filter it prior injection to the HPLC with a suitable syringe filter (5.12).

6.3 Spiking procedure

6.3.1 Fruit juice

Weigh to the nearest 0,1 g, 19 g of the juice into a 50 ml beaker and add exactly 1,00 ml of the 200 ng/ml or the 500 ng/ml spiking solution (4.19) to obtain spiking levels of 10 µg/kg or 25 µg/kg respectively. Stir the solution until completely mixed. From this 20,0 g fortified solution, use exactly 10,0 g for analysis according to 6.1 and 6.2.

6.3.2 Fruit purée

Weigh to the nearest 0,1 g, 19 g of the purée into a 50 ml beaker. Add exactly 1,00 ml of the 200 ng/ml or the 500 ng/ml spiking solution (4.19) to obtain spiking levels of 10 µg/kg or 25 µg/kg respectively. Mix thoroughly with a spatula for at least 3 min and scratch any purée traces from the beaker wall to include these in the mixing process. After pre-stirring with a spatula, use a plastic syringe and mix the spiked purée by repeated aspiration and rapid dispensing. Make sure that the contents are mixed completely. From the resulting 20,0 g fortified purée, use exactly 10,0 g for analysis according to 6.1 and 6.2.

6.3.3 Spike calculation

For spiking experiments, use 10 g for juice and purée as basis for calculation.

7 HPLC analysis

7.1 HPLC operating conditions

When the column specified in 5.13.4 and the mobile phase specified in 4.15 were used, the following settings were found to be appropriate, see also Figures A.1 and A.2:

— Flow rate mobile phase (column): 0,6 ml/min to 1,0 ml/min;

— UV detection wavelength: 276 nm;

— Injection volume: 100 µl to 200 µl.

7.2 Preparation of calibration solutions for HPLC

Pipette for example amounts of 60 µl, 120 µl, 180 µl, 240 µl, 300 µl, 360 µl and 420 µl of the patulin standard solution (4.18) into different 50 ml volumetric flasks. Fill the volumetric flasks up to the mark with water of pH = 4 (4.8) and shake. This will result in patulin solutions with mass concentrations of 12 ng/ml, 24 ng/ml, 36 ng/ml, 48 ng/ml, 60 ng/ml, 72 ng/ml and 84 ng/ml. These mass concentrations reflect contamination levels of 4,8 µg/kg, 9,6 µg/kg, 14,4 µg/kg, 19,2 µg/kg, 24,0 µg/kg, 28,8 µg/kg and 33,6 µg/kg patulin in the sample based on the assumption that a patulin standard solution of 10 µg/ml (4.18) was used for preparation and they can be used directly for injection into the HPLC system.

In case the patulin standard solution (4.18) has a different mass concentration than 10 µg/ml, adjust calibration solutions by calculating the correct aliquots in order to take account of the actual mass concentration of the standard solution determined in 4.18.

Store this solution in a refrigerator at 4 °C. Solutions stored in this way are stable for at least eight weeks.

7.3 Calibration curve

Prepare a calibration curve by injecting 100 µl of the calibration solutions (7.2) at the beginning of every day of the analysis.

Plot the peak area against the mass of the injected patulin and check the curve for linearity.

In case the content of patulin in the sample is outside of the calibration curve, prepare another calibration curve with an appropriate range to cover the mass concentration of the sample. Alternatively the injection solution for HPLC analysis can be diluted to a patulin content appropriate for the established calibration curve.

7.4 Determination of patulin in sample test solutions

Inject aliquots of the sample test solution prepared as described in 6.1 and 6.2 into the chromatograph using the same conditions applied for the preparation of the calibration curve.

8 Calculation

Quantitative determination is carried out by the integration of the peak height or peak area. Determine the mass fraction, w_{pat}, of patulin in the test sample in micrograms per kilogram, directly from the calibration curve (7.3) or calculate it from the mass concentration of the injected solution, using Equation (2):

$$w_{pat} = \frac{\rho_{pat} \times V_1 \times V_3}{V_2 \times m_s} \quad (2)$$

where

ρ_{pat} is the mass concentration of patulin, in nanograms per millilitre, in the sample test solution injected calculated from linear regression;

V_1 is the volume, in millilitres, of the solvent taken for extraction (here: 10 ml);

V_2 is the volume, in millilitres, of the aliquot of sample extract taken for SPE cleanup (here: 2,5 ml);

V_3 is the volume, in millilitres, of water of pH = 4 (4.8) used to re-dissolve (here: 1,0 ml);

m_s is the mass, in grams, of sample material taken for analysis (here: 10 g).

Report the result to three significant figures.

9 Precision

9.1 General

Details of the inter-laboratory test of the precision of the method are summarized in Annex B. The values derived from the interlaboratory test may not be applicable to analyte concentration ranges and matrices other than given in Annex B.

9.2 Repeatability

The absolute difference between two single test results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability limit r in not more than 5 % of the cases.

The values for apple juice are:

$\bar{x}$ = 7,4 µg/kg r = 1,60 µg/kg
$\bar{x}$ = 15,5 µg/kg r = 5,07 µg/kg
$\bar{x}$ = 3,0 µg/kg r = 1,01 µg/kg
$\bar{x}$ = 6,0 µg/kg r = 2,40 µg/kg
$\bar{x}$ = 10,7 µg/kg r = 3,61 µg/kg

The values for baby food fruit purée are:

$\bar{x}$ = 7,4 µg/kg r = 0,73 µg/kg
$\bar{x}$ = 17,9 µg/kg r = 2,91 µg/kg
$\bar{x}$ = 3,4 µg/kg r = 0,90 µg/kg
$\bar{x}$ = 7,1 µg/kg r = 1,06 µg/kg
$\bar{x}$ = 10,1 µg/kg r = 2,13 µg/kg

9.3 Reproducibility

The absolute difference between two single test results found on identical test material reported by two laboratories will exceed the reproducibility limit *R* in not more than 5 % of the cases.

The values for apple juice are:

$\bar{x}$ = 7,4 µg/kg *R* = 6,54 µg/kg
$\bar{x}$ = 15,5 µg/kg *R* = 12,29 µg/kg
$\bar{x}$ = 3,0 µg/kg *R* = 3,28 µg/kg
$\bar{x}$ = 6,0 µg/kg *R* = 3,30 µg/kg
$\bar{x}$ = 10,7 µg/kg *R* = 7,28 µg/kg

The values for baby food fruit purée are:

$\bar{x}$ = 7,4 µg/kg *R* = 3,05 µg/kg
$\bar{x}$ = 17,9 µg/kg *R* = 6,30 µg/kg
$\bar{x}$ = 3,4 µg/kg *R* = 3,39 µg/kg
$\bar{x}$ = 7,1 µg/kg *R* = 3,61 µg/kg
$\bar{x}$ = 10,1 µg/kg *R* = 5,54 µg/kg

10 Test report

The test report shall contain at least the following data:

a) all information necessary for the identification of the sample (kind of sample, origin of sample, designation);

b) a reference to this European Standard;

c) the date and type of sampling procedure (if known);

d) the date of receipt;

e) the date of test;

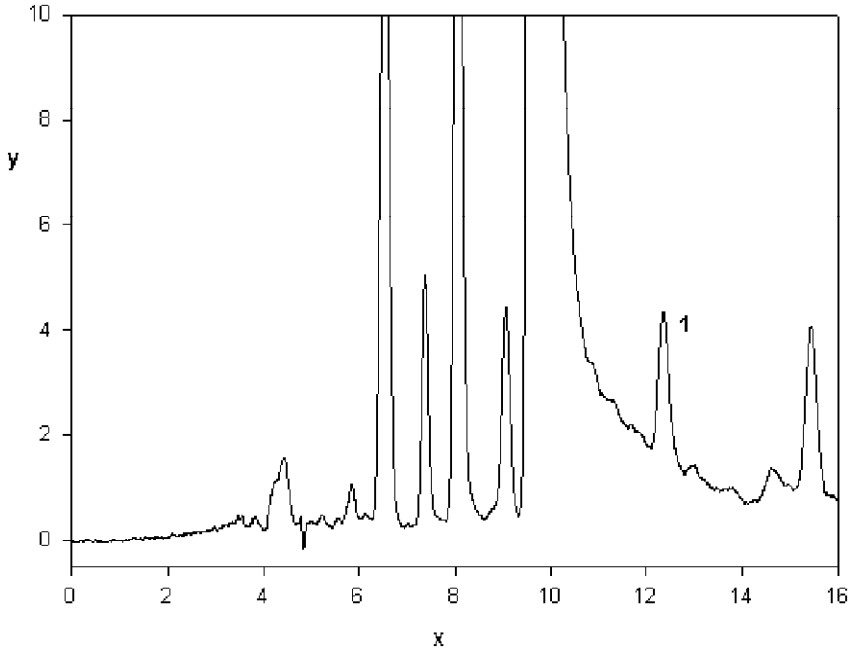
f) the test results and the units in which they have been expressed;

g) any particular points observed in the course of the test;

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

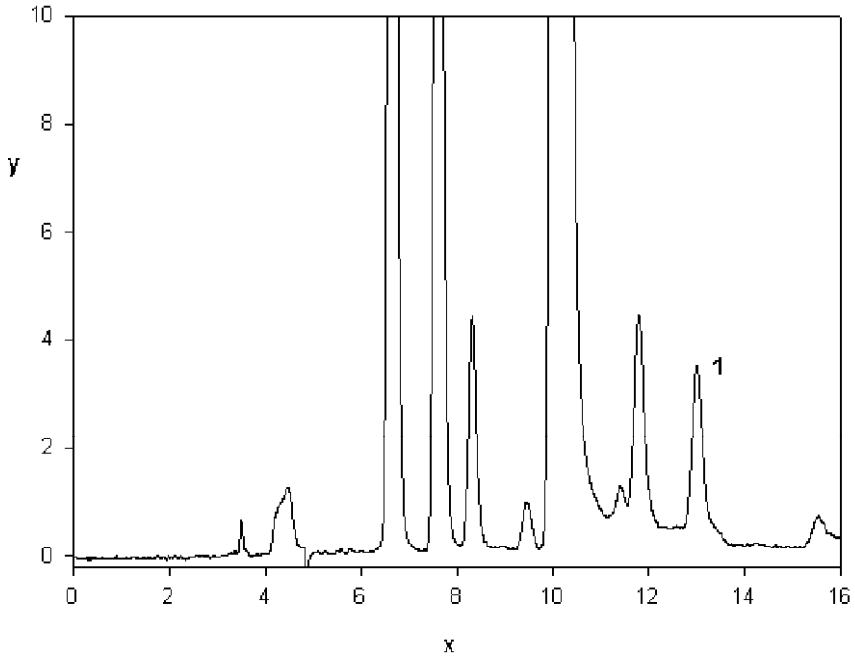
Annex A
(informative)

Typical chromatograms



Key
x time, in minutes
y signal, in millivolts
1 patulin

Figure A.1 — Typical chromatogram of apple juice with patulin at approximately 13 µg/kg



Key
x time, in minutes
y signal, in millivolts
1 patulin

Figure A.2 — Typical chromatogram of apple purée with patulin at approximately 15 µg/kg

Annex B
(informative)

Precision data

The data given in Table B.1 and Table B.2 were obtained in an inter-laboratory test [1] according to an IUPAC protocol for method performance studies [3].

Table B.1 — Precision data inter-laboratory study for apple juice

Sample	**1**	**2**	**3**	**4**	**5**
Target levels, µg/kg	10	25	3,6 [a]	8,1 [a]	12,2 [a]
Year of inter-laboratory test	2003	2003	2003	2003	2003
Number of laboratories	14	14	13	13	15
Number laboratories retained after eliminating outliers	14	14	12	11	15
Number of outliers (laboratories)	0	0	1	2	0
Number of accepted results	14	14	12	11	15
Mean value, $\bar{x}$, µg/kg	7,4	15,5	3,0	6,0	10,7
Repeatability standard deviation s_r, µg/kg	0,57	1,81	0,36	0,86	1,29
Repeatability relative standard deviation, RSD_r, %	7,7	11,7	12,2	14,3	12,1
Repeatability limit r [$r = 2{,}8 \times s_r$], µg/kg	1,60	5,07	1,01	2,40	3,61
Reproducibility standard deviation s_R, µg/kg	2,34	4,39	1,17	1,18	2,60
Reproducibility relative standard deviation, RSD_R, %	31,6	28,3	39,5	19,8	24,3
Reproducibility limit R [$R = 2{,}8 \times s_R$], µg/kg	6,54	12,29	3,28	3,30	7,28
Recovery, %	74	62	-	-	-
HorRat value, according to [4]	0,94	0,94	1,03	0,57	0,77
HorRatR value, according to [5]	1,44	1,29	1,80	0,90	1,10

[a] Naturally contaminated material, target values determined during homogeneity study of material.

Table B.2 — Precision data inter-laboratory study for baby food fruit purée

Sample	**6**	**7**	**8**	**9**	**10**
Target levels, µg/kg	10	25	2,8 [a]	6,5 [a]	10,5 [a]
Year of inter-laboratory test	2003	2003	2003	2003	2003
Number of laboratories	15	15	13	15	14
Number laboratories retained after eliminating outliers	13	13	11	14	14
Number of outliers (laboratories)	2	2	2	1	0
Number of accepted results	13	13	11	14	14
Mean value, $\bar{x}$, µg/kg	7,4	17,9	3,4	7,1	10,1
Repeatability standard deviation s_r, µg/kg	0,26	1,04	0,32	0,38	0,76
Repeatability relative standard deviation, RSD_r, %	3,5	5,8	9,3	5,3	7,5
Repeatability limit r [$r = 2{,}8 \times s_r$], µg/kg	0,73	2,91	0,90	1,06	2,13
Reproducibility standard deviation s_R, µg/kg	1,09	2,25	1,21	1,29	1,98
Reproducibility relative standard deviation, RSD_R, %	14,7	12,5	35,2	18	19,6
Reproducibility limit R [$R = 2{,}8 \times s_R$], µg/kg	3,05	6,30	3,39	3,61	5,54
Recovery, %	74	72	-	-	-
HorRat value, according to [4]	0,44	0,43	0,94	0,53	0,61
HorRatR value, according to [5]	0,67	0,57	1,60	0,82	0,89

[a] Naturally contaminated material, target values determined during homogeneity study of material.

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