SLOVENSKI STANDARD
SIST EN 15662:2009

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Hrana rastlinskega izvora - Doloļ anje ostankov pesticidov z uporabo GC-MS in/ali LC-MS(/MS) po delitvi in izpiranju acenotrila z disperzivno SPE - Metoda QuEChERS

Foods of plant origin - Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and clean-up by dispersive SPE - QuEChERS-method

Pflanzliche Lebensmittel - Bestimmung von Pestizidrückständen mit GC-MS und/oder LC -MS/MS nach Acetonitril-Extraktion/Verteilung und Reinigung mit dispersiver SPE - QuEChERS-Verfahren

Aliments d'origine végétale - Méthode polyvalente de détermination des résidus des pesticides par CG-SM et CL/SM(/SM) avec extraction/partition avec de l'acétonitrile et nettoyage par SPE dispersée - Méthode QuEChERS

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

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Foreword

This document (EN 15662:2008) 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of an identical text or by endorsement, at the latest by May 2009, and conflicting national standards shall be withdrawn at the latest by May 2009.

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1 Scope

This European Standard describes a method for the analysis of pesticide residues in foods of plant origin, such as fruits (including dried fruits), vegetables, cereals and processed products thereof. The method has been collaboratively studied on a large number of commodity/pesticide combinations.

2 Principle

The homogeneous sample is extracted with the help of acetonitrile. Samples with low water content (< 80 %) require the addition of water before the initial extraction to get a total of approximately 10 g of water. 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) employing bulk sorbents as well as magnesium sulfate for the removal of residual water. Following clean-up with amino-sorbents (e.g. primary secondary amin sorbent, PSA) extracts are acidified by adding a small amount of formic acid, to improve the storage stability of certain base-sensitive pesticides. The final extract can be directly employed for GC- and LC-based determinative analysis. Quantification is performed using an internal standard, which is added to the extract after the initial addition of acetonitrile. A brief overview of the method is shown in the flowchart in Annex C.

3 Reagents

3.1 General and safety aspects

Unless otherwise specified, use reagents of recognized analytical grade. Take every precaution to avoid possible contamination of water, solvents, sorbents, inorganic salts, etc.

DISCLAIMER — This standard refers to several trade names products and instruments which are commercially available and suitable for the described procedure. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the products named. Equivalent products may be used if they can be shown to lead to equivalent results.

3.2 Water, HPLC quality

3.3 Acetonitrile, HPLC quality

3.4 Methanol, HPLC quality

3.5 Ammonium formate

3.6 Magnesium sulfate, anhydrous, grit, e.g. Fluka No. 63135

Phthalates may be removed in a muffle furnace by heating to 550 °C (e.g. overnight).

3.7 Magnesium sulfate, anhydrous, fine powder

Phthalates may be removed in a muffle furnace by heating to 550 °C (e.g. overnight).

3.8 Sodium chloride

3.9 Disodium hydrogencitrate sesquihydrate

3.10 Trisodium citrate dihydrate

3.11 Sodium hydroxide solution, substance concentration c = 5 mol/l

Dissolve 2 g of sodium hydroxide in approximately 5 ml of water and dilute to 10 ml.

3.12 Buffer-salt-mixture for second extraction and partitioning:

Weigh 4 g ± 0,2 g of magnesium sulfate anhydrous (3.6), 1 g ± 0,05 g of sodium chloride, 1 g ± 0,05 g of trisodium citrate dihydrate and 0,5 g ± 0,03 g of disodium hydrogencitrate sesquihydrate into a cup (4.11). These amounts refer to approximately 10 ml water in the sample.

For highly acidic samples (with pH < 3) the pH-value achieved after the addition of buffering salts is usually below 5. To better protect acid labile compounds the pH-value can be elevated by adding 5 mol/l sodium hydroxide solution (3.11): For lemons, limes and currants add 600 µl and for raspberry 200 µl of sodium hydroxide solution directly to the salt mixture.

NOTE It is advisable to prepare a sufficient number of buffer-salt-mixtures in advance so that extraction series can be performed quickly without interruption. The preparation of the salt mixtures can be enormously facilitated using a sample divider (4.12). The amounts of salts given above are to be used for sample portions containing approximately 10 g water.

3.13 Formic acid solution in acetonitrile, volume fraction φ = 5 ml formic acid/100 ml

Dilute 0,5 ml of formic acid (mass fraction w = > 95 %) to 10 ml with acetonitrile (3.3).

3.14 Primary secondary amin sorbent

For example, Bondesil-PSA® 40 µm Varian No. 12213023[1)].

Other amino sorbents may be used, but investigations may be necessary to prove equivalency especially regarding analyte losses and pH value of the end extracts.

3.15 Graphitised Carbon Black sorbent (GCB), e.g. Supelco Supelclean Envi-Carb® 1) SPE Bulk Packing, No. 57210U

Other graphitised carbon sorbents may be used, but investigations will be necessary to prove equivalency especially regarding analyte losses.

3.16 Sorption mixture 1: GCB (3.15)/ magnesium sulfate anhydrous fine powder (3.7)-mixture, 1 + 59 mass portions

Mix the two components intensively to form a visually homogeneous mixture.

3.17 Sorption mixture 2: GCB (3.15)/ magnesium sulfate anhydrous fine powder (3.7)-mixture, 1 + 19 mass portions

Mix the two components intensively to form a visually homogeneous mixture.

NOTE It is highly advisable to prepare the sorption mixtures 1 (3.16) and 2 (3.17) in advance and store them in sealable vessels. For the extract clean-up according to 5.4.3 the pre-mixed sorption mixtures 1 or 2 are weighed into the centrifuge tubes (4.4).

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1) Bondesil-PSA® is a product supplied by Varian, Inc. (Palo Alto, CA, USA). Envi-Carb is a product supplied by Supelco. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the products named. Equivalent products may be used if they can be shown to lead to the same results.

3.18 C-18-sorbent (Octadecyl-silyl-modified silica gel), Bulk material 50 µm

3.19 Internal standard and quality control standard solutions in acetonitrile, ρ = 10 µg/ml to 50 µg/ml

Table 1 shows a list of potential internal standards (ISTDs) and quality control (QC) standards that may be used in this method. The suggested concentration values (C_{ISTD}) listed refers to the ISTD solutions that should be added at the first extraction step (5.2). An appropriate dilution of this solution ($C_{ISTD}^{cal\ mix}$) should be prepared to be used for the preparation of the standard solutions. For more details see 3.22.

Table 1 — Potential internal standards (ISTDs) or quality control (QC) standards

Name of the compound	Log P (octanol-water partition coefficient)	Chlorine atoms	Suggested concentration C_{ISTD} [µg/ml][a]	GC				LC	
				ECD	NPD	MSD EI (+)	MSD CI (-)	MS/MS ESI (+)	MS/MS ESI (-)
Potential Internal Standards									
PCB 18	5,55	3	50	+++	-	++	+++	-	-
PCB 28	5,62	3	50	+++	-	++	+++	-	-
PCB 52	6,09	4	50	+++	-	++	+++	-	-
Triphenyl phosphate	4,59	-	20	-	+++	+++	-	+++	-
Tris-(1,3-dichlorisopropyl)-phosphate	3,65	6	50	+++	+++	+++	+++	+++	+
Triphenylmethane	5,37	-	10	-	-	+++	-	-	-
Bis-nitrophenyl urea (nicarbazin)	3,76	-	10	-	-	-	-	-	+++
Potential Quality Control Standards (may be contained in the same mixture as the other ISTDs used or added at a different stage of analysis to detect and localize sources of error)									
PCB 138[b]	6,83	6	50	+++	-	++	+++	-	-
PCB 153[b]	7,75	6	50	+++	-	++	+++	-	-
Anthracene (or its d10 analogue)[c]	4,45	-	100	-	-	++	-	-	-

a Exemplary concentrations of the ISTD solutions to be added to the test samples in 5.2, use acetonitrile as solvent.

b Recoveries of PCB 138 and 153 drop as lipid amount in the sample increases, recoveries of those two compounds exceeding 70 % indicate that no unacceptable partitioning losses have occurred even for the most lipophilic pesticides.

c Recoveries of anthracene exceeding 70 % will indicate that no unacceptable losses of pesticides with high carbon affinity have occurred during dispersive SPE with GCB.

3.20 Pesticide stock solutions

Prepare individual stock solutions of analytical standards at concentrations that are sufficient to allow the preparation of complex pesticide working solutions (3.21) that are used for the preparation of standard solutions.

Usually, store stock solutions at ≤ -18 °C. Check the stability of stock solutions during storage regularly [2]. In some cases the addition of acids or bases can be helpful to enhance stability and extend the acceptable

storage period. Before withdrawing any aliquot from this solution redissolve any precipitation that may have occurred.

3.21 Pesticide working solutions

Because of the broad applicability of this method and due to the partly divergent pH-stability of pesticides, more than one working solution each containing one or more pesticides can be needed to cover the entire pesticide spectrum of interest. These are prepared by mixing together defined volumes of the required pesticide stock solutions (3.20) and appropriately diluting them with acetonitrile. The pesticide concentrations in these mixtures should be sufficient to allow the preparation of the required matrix matched standards (see 3.22.2) with moderate dilution of the blank sample extract (e.g. less than 20 %).

Usually, store pesticide working solutions at ≤ -18 °C. Check the stability of pesticides contained in these mixtures during storage regularly [2]. In some cases the addition of acids or bases can be helpful to enhance stability and extend acceptable storage times.

3.22 Standard solutions (calibration mixtures)

3.22.1 Solvent-based standards

Solvent-based standards are prepared by mixing known volumes of the pesticide working solutions ($V_{pest}^{cal\ mix}$ see 3.21) and the ISTD solution ($V_{ISTD}^{cal\ mix}$ see 3.19) and filling up to volume with acetonitrile.

The volume of the ISTD solution to be employed ($V_{ISTD}^{cal\ mix}$) will depend on the volume of the standard solution to be prepared ($V^{cal\ mix}$) and should be such to ensure an ISTD concentration similar to that in the sample test solutions (5.3, 5.4).

EXAMPLE If 1 ml solvent-based standard is prepared the volume of ISTD solution to be added should contain a mass of ISTD ($m_{ISTD}^{cal\ mix} = C_{ISTD}^{cal\ mix} \times V_{ISTD}^{cal\ mix}$) which is 10-fold smaller than the mass of ISTD added to the test portions in 5.2.3, where 10 ml of acetonitrile are used for extraction. It is thus indicated to appropriately dilute the concentration of internal standard solution (in this case $C_{ISTD}^{cal\ mix}$ = 0,1 × C_{ISTD}). Then the same pipette volume can be used to add ISTDs to spike test samples and for the preparation of standard solutions. Table 2 shows exemplarily the ratio of the ISTD mass that should be added to the test portions (5.2.3) and the standard solutions (3.22).

The preparation of multiple standard solutions covering a broad concentration range will allow the construction of a calibration curve (see 6.2).

NOTE A pesticide concentration of 1 µg/ml correlates to a residue level of 1 mg/kg when a 10 g sample is employed (e.g. samples with water content > 30 %) or 2 mg/kg when 5 g sample is employed (e.g. cereals).

3.22.2 Matrix-matched standards

Prepare matrix-matched standards in the same way as solvent-based standards, however, instead of pure acetonitrile use extracts of blank samples (prepared as described in 5.1 to 5.4, but without ISTD addition). To minimize errors caused by matrix induced effects during chromatography, it is best to choose similar commodities (e.g. apple for apple samples, carrot for carrot samples, etc.). Should the dilution of the blank sample extract upon addition of the pesticide working solutions exceed 20 %, a volume adjustment may be necessary to avoid errors caused by differences in the matrix-induced enhancement effect between sample extract and matrix-matched standard.

The stability of pesticides in matrix-matched standards can be lower than that of standards in pure acetonitrile and has to be checked more thoroughly.

Table 2 — Ratios of the masses of ISTD added to the test-portion and to the standard solutions (calibration mixtures)

Volume of standard solution $V^{cal\ mix}$ ml	$\frac{m_{ISTD}^{sample}}{m_{ISTD}^{cal\ mix}} = \frac{C_{ISTD} \times V_{ISTD}^{sample}}{C_{ISTD}^{cal\ mix} \times V_{ISTD}^{cal\ mix}}$
1	10
2	5
5	2
10	1
NOTE The values given in this table refer to sample extract volumes of ca. 10 ml (i.e. following addition of 10 ml acetonitrile in 5.2.3). The blank sample employed to prepare the matrix-matched standard should be extracted in the same way as the sample.	

3.23 Cold water (< 4 °C)

3.24 Dry ice

3.25 Mobile phase A_1: Ammonium formate solution in water, c = 5 mmol/l

3.26 Mobile phase B_1: Ammonium formate solution in methanol, c = 5 mmol/l

3.27 Mobile phase A_2: Acetic acid solution in water, φ = 0,1 ml glacial acetic acid /l

3.28 Mobile phase B_2: Acetic acid solution in acetonitrile, φ = 0,1 ml glacial acetic acid /l

3.29 Mobile phase A_3: Methanol/water 2+8 (*V/V*) with 5 mmol/l ammonium formate

3.30 Mobile phase B_3: Methanol/water 9+1 (*V/V*) with 5 mmol/l ammonium formate

4 Apparatus

Usual laboratory apparatus and, in particular, the following:

4.1 Sample processing equipment, e. g. Stephan UM 5 universal

4.2 High speed dispersing device

Diameter of the dispersing elements should fit the openings of the centrifuge tubes (4.4) used.

4.3 Automatic pipettes, suitable for handling volumes of 10 µl to 100 µl, 200 µl to 1 000 µl and 1 ml to 10 ml.

NOTE Instead of the latter, 10 ml graduated glass pipettes may be used alternatively.

4.4 Centrifuge tubes with screw caps, 50 ml

EXAMPLES a) 50 ml centrifuge tubes made of poly-tetrafluoroethylene with screw caps, or

b) disposable 50 ml polypropylene centrifuge tubes with screw caps

4.5 Polypropylene-single use centrifuge tubes with screw caps, 10 ml or 12 ml

4.6 10 ml solvent-dispenser for acetonitrile, to be employed in 5.2.3

4.7 Centrifuges suitable for the centrifuge tubes employed in the procedure (4.4 and 4.5) and capable of achieving at least 3 000 *g*.

4.8 Powder funnel, to fit the openings of the centrifuge tubes

4.9 Injection vials, 1,5 ml, suitable for GC and LC autosampler, if necessary with micro-inserts

4.10 Screw capped glass vials, e.g. 20 ml, for the storage of excessive amounts of the final extract, if necessary

4.11 Plastic cups (stackable), 25 ml, used for the storage of buffer-salt mixture portions (3.12).

4.12 Sample divider, to automatically portion salts and sorbents

For example from Retsch/Haan, PT 100 or Fritsch/Idar-Oberstein, Laborette 27 or Bürkle/Lörrach, Repro high-precision sample divider[2)]. Their use is optional but highly recommended when dealing with high numbers of samples.

NOTE The first two are better for portioning the buffer-salt-mixture (3.12) while the Bürkle Repro is designed for smaller amounts of solids and is much more suitable for portioning the PSA (3.14) / magnesium sulfate (3.6) mixture needed for „dispersive SPE" (5.4.2). The 10 ml polypropylene tubes from Simport Canada, 17 mm x 84 mm, article-no. T550-10AT[2)] (4.5) perfectly fit the Bürkle Repro.

4.13 Vibration device, e.g. Vortex (used for recovery studies)

4.14 LC-MS/MS system equipped with electrospray ionisation (ESI) interface (see Annex A)

4.15 GC-MS system, equipped with appropriate detectors e.g. MS, MS/MS, TOF and with PTV-injector with solvent vent mode (see GC-MS equipment described in Annex A)

2) PT 100, Laborette 27, Repro high-precision sample divider and T550-10AT are examples 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 these products.

5 Procedure

5.1 Preparation and storage of the samples

5.1.1 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 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.

5.1.2 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. Generally, laboratory samples should not be stored longer than 3 days before preparation. Dried or similarly processed samples should be analysed within their stated shelf life.

5.1.3 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. No further plant-parts may be removed.

The reduction of the laboratory sample shall be carried out in such a way that representative portions are obtained (e. g. by sub-division into four and selection of opposite quarters). For samples of small units (e. g. small fruits such as berries, legumes, cereals), the sample must be thoroughly mixed before weighing out the partly-prepared test sample. When the samples are made up of larger units, take wedge-shaped sections (e.g. melons) or cross sections (e. g. cucumbers) that include the skin (outer surface) from each unit [2].

5.1.4 Test sample

From each partly-prepared test sample, any parts that would cause difficulties with the homogenisation process should be removed. In the case of stone fruits, the stones shall be removed. A record of the plant-parts that have been removed shall be kept. Precautions should be taken to avoid any losses of juice or flesh. This is the test sample. Calculation of the residue shall be based on the mass of the original test sample (including the stones).

Where the homogeneity of the test sample is not sufficient or the extraction of residues may 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 x 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. Especially in the case of fruits and vegetables, cryogenic milling is much more effective at homogenising commodities that have tough skins (e.g. tomatoes or grapes) compared to milling at ambient temperature. Given the fact that non-systemic pesticides often predominantly occur on the skin, cryogenic milling significantly reduces sub-sampling variability. 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.

5.1.5 Test portion

Individual test portions each sufficient for one analysis should be abstracted 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 test portions are taken from test samples after being stored frozen, the test samples shall be mixed before taking test portions to ensure that homogeneity has been re-established.

5.1.6 Homogenization of dried fruit and similar commodities (< 30 % water content)

Add 850 g of cold water (3.23) to 500 g frozen dried fruits and homogenize the mixture (if possible by adding dry ice).

5.2 First extraction step

5.2.1 Weighing

Transfer a representative test portion (m_a) of the comminuted homogenous sample into a 50 ml centrifuge tube (4.4). In the case of fruits and vegetables weigh 10 g ± 0,1 g (m_a) into the centrifuge tube. For dried fruit homogenates as described in 5.1.6 weigh 13,5 g corresponding to 5 g (m_a) sample. For dry sample materials like cereal products and honey weigh a homogenised portion of 5 g ± 0,05 g (m_a). For fermented products and extract-rich spices weigh 2 g ± 0,03 g (m_a).

5.2.2 Water addition

For samples having water content below 80 % add sufficient cold water (3.23), leading to a total water content in the tube of approximately 10 g. See Table 3 for typical water content and the amount of water to be added to the corresponding test portions.

NOTE The homogenates derived from 5.1.6 do not need additional water.

5.2.3 Solvent and ISTD addition

Add 10 ml of acetonitrile and a defined small volume of the ISTD solution (V_{ISTD}^{sample} e.g. 100 µl) containing one or several of the compounds listed in Table 1 at the concentrations exemplary given (C_{ISTD}).

5.2.4 Extraction

Close the tube and shake vigorously for 1 min. If the sample's degree of comminution is insufficient or the residues do not readily extract from the matrix, the extraction time may be prolonged (e.g. to 20 min using a mechanical shaker) or assisted by a high-speed disperser (e.g. Ultra-Turrax). The dispersing element is immersed into the sample/acetonitrile mixture and comminution is performed for about 2 min to 5 min at high speed. In either case ensure that no significant degradation of the target pesticides occurs. As the ISTD solution has already been added, no rinsing of the dispersing element is necessary. Nevertheless, it still has to be cleaned thoroughly before being used for the next sample to avoid cross-contamination.

Make sure to employ dispersing elements that can pass through the opening of the centrifuge tubes (4.4).

Samples should be extracted frozen or while in the process of thawing (except dry samples with water content < 20 %). If samples are employed for extraction at ambient temperature, it shall be ensured that no significant degradation of the target pesticides occurs.

Table 3 — Water content of selected foods and amount of water, which has to be added

Commodity group	Commodity	Typical water content g/100 g	Amount of water added to 10 g of test portion g	Amount of water added to 5 g of test portion g	Remarks
Fruits					
Citrus fruits	citrus juices	90			
	grapefruit	90			Add 600 µl 5 mol/l NaOH-solution to buffer salts as stated in 3.12 (applies only to lemon/lime). Optionally perform freeze out step to remove waxes; see 5.4.1 (applies to all citrus fruits).
	lemon/lime	85			
	orange	85			
	orange peel	75	2,5		
	tangerine	90			
Pome fruit	apple	85			
	apple, dried	30		8,5 (see 5.1.6)	
	apple sauce	80			
	apple juice	90			
	pear	85			
	quince	85			
Stone fruit	apricot	85			
	apricot, dried	30		8,5 (see 5.1.6)	
	apricot nectar	85			
	cherry	85			
	mirabelle	80			
	nectarine	85			
	peach	90			
	peach, dried	20		8,5 (see 5.1.6)	
	plum	85			
	plum, dried	20		8,5 (see 5.1.6)	
Soft and small fruits	blackberry	85			
	blueberry	85			
	currant	85			Add 600 µl 5 mol/l NaOH-solution to buffer salts as stated in 3.12.
	elderberry	80			
	gooseberry	90			
	grapes	80			
	raspberry	85			Add 200 µl 5 mol/l NaOH-solution to buffer salts as stated in 3.12.
	raisin	20		8,5 (see 5.1.6)	
	strawberry	90			

Other fruits	pineapple	85			
	banana	75	2,5		
	fig, dried	20		8,5 (see 5.1.6)	
	kiwi	85			
	mango	80			Use GCB in dispersive SPE as stated in 3.16 and 5.4.3 (mixture 1).
	papaya	90			
Vegetables					
Root and tuber vegetables	beetroot	90			
	carrot	90			Use GCB in dispersive SPE as stated in 3.16 and 5.4.3 (mixture 1).
	celeriac	90			
	horseradish	75	2,5		
	parsley root	90			
	radish	95			
	black salsify	80			
	potato	80			
Leek plants	garlic	60		7,0	
	onion	90			
	leek	85			
	shallot	80			
	chive	85			Use GCB in dispersive SPE as stated in 3.17 and 5.4.3 (mixture 2).
Fruiting vegetables	aubergine	90			
	cucumber	95			
	melon	90			
	pepper, sweet	90			For red sweet pepper use GCB in dispersive SPE as stated in 3.17 and 5.4.3 (mixture 2).
	pumpkin	95			For strongly coloured varieties use GCB in dispersive SPE as stated in 3.16 and 5.4.3 (mixture 1).
	tomato	95			
	zucchini (courgette)	95			
Cabbage	broccoli	90			
	brussels sprouts	85			
	cauliflower	90			
	chinese cabbage	95			
	kale	90			
	kohlrabi	90			
	red cabbage	90			