

**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**

ICS 67.050

National foreword

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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

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

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

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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of an identical text or by endorsement, at the latest by May 2009, and conflicting national standards shall be withdrawn at the latest by May 2009.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 $c = 5 \text{ mol/l}$

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 $4 \text{ g} \pm 0,2 \text{ g}$ of magnesium sulfate anhydrous (3.6), $1 \text{ g} \pm 0,05 \text{ g}$ of sodium chloride, $1 \text{ g} \pm 0,05 \text{ g}$ of trisodium citrate dihydrate and $0,5 \text{ g} \pm 0,03 \text{ g}$ 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 $\text{pH} < 3$) 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 $\varphi = 5 \text{ ml formic acid}/100 \text{ ml}$

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. 12213023¹⁾.

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).

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, $\rho = 10 \mu\text{g/ml}$ 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} [$\mu\text{g/ml}$] ^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	-	-	++	-	-	-
<p>a Exemplary concentrations of the ISTD solutions to be added to the test samples in 5.2, use acetonitrile as solvent.</p> <p>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.</p> <p>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.</p>									

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 $\leq -18 \text{ }^\circ\text{C}$. 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 $C_{ISTD}^{cal\ mix} = 0,1 \times C_{ISTD}$). 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 A₁: Ammonium formate solution in water, c = 5 mmol/l

3.26 Mobile phase B₁: Ammonium formate solution in methanol, c = 5 mmol/l

3.27 Mobile phase A₂: Acetic acid solution in water, $\varphi = 0,1$ ml glacial acetic acid /l

3.28 Mobile phase B₂: Acetic acid solution in acetonitrile, $\varphi = 0,1$ ml glacial acetic acid /l

3.29 Mobile phase A₃: Methanol/water 2+8 (V/V) with 5 mmol/l ammonium formate

3.30 Mobile phase B₃: 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 μ l to 100 μ l, 200 μ l to 1 000 μ l 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 divider²⁾. 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. T550-10AT²⁾ (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 weight 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 $10 \text{ g} \pm 0,1 \text{ g}$ (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 $5 \text{ g} \pm 0,05 \text{ g}$ (m_a). For fermented products and extract-rich spices weigh $2 \text{ g} \pm 0,03 \text{ g}$ (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).
	lemon/lime	85			
	orange	85			Optionally perform freeze out step to remove waxes; see 5.4.1 (applies to all citrus fruits).
	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			

	savoy cabbage	90			
	white cabbage	90			
Leafy vegetables and herbs	lettuce varieties	95			For strongly coloured varieties use GCB in dispersive SPE as stated in 3.16 and 5.4.3 (mixture 1).
	endive	95			
	cress	90			Use GCB in dispersive SPE as stated in 3.17 and 5.4.3 (mixture 2).
	lamb's lettuce	85			
	parsley	80			
	rucola	85			
	spinach	90			
Stem vegetables	asparagus	95			
	celery	95			
	leek	85			
	rhubarb	95			
	artichokes	85			
Legumes	beans, peas, lentils (dried)	<10		10	
	beans (fresh)	75	2.5		
Other					
Cereals	cereals (grain, flour, etc.)	10		10	Optionally perform freeze out step or add 25 mg C18 sorbent per ml extract at the dispersive SPE step to remove lipids (see 5.4.1).
	coffee beans	<10		10 (use 2 g sample if fermented)	Add 75 mg PSA sorbent per ml extract at the dispersive SPE step if fermented.
	tea	<10			
	dry herbs and spices	<10		10 (use 2 g sample if extract-rich)	
	mushrooms	90			
	wine	90			

5.3 Second extraction step and partitioning

Add the prepared buffer-salt mixture (3.12) to the suspension from 5.2. Close the tube and immediately shake vigorously for 1 min and centrifuge for 5 min at > 3 000 g.

NOTE When dealing with highly acidic commodities, such as lemons, limes, currants and raspberries, use buffer-salt mixtures to which NaOH was added as stated in 3.12.

In the presence of water, magnesium sulfate tends to form lumps, which can harden rapidly. This can be avoided, if immediately after the addition of the salt mixture the centrifuge tube is shaken vigorously for few seconds. The 1 min extraction of the entire batch may be performed in parallel after the salts have been added to all the samples.

Pesticides with acidic groups (e.g. phenoxyalcanoic acids) interact with amino-sorbents such as PSA. Thus, if such pesticides are within the scope of analysis, their determinative analysis (preferably via LC-MS/MS ESI

neg.) should be performed directly from the raw extract after centrifugation but prior to clean-up (5.4). For this, fill an aliquot of the raw extract into a vial and employ for LC-MS/MS analysis. Typical LC-MS/MS conditions for acidic pesticides are given in Annex A.

5.4 Clean-up

5.4.1 Removal of co-extracted fat, wax, sugars (e.g. for cereals, citrus fruits)

Transfer an aliquot of 8 ml of the acetonitrile phase (5.3) into a centrifuge tube (4.5) and store overnight in a freezer (for flour, 2 h are sufficient), wherewith the major part of fat and waxes solidify and precipitate. Following a short centrifugation (where necessary), 6 ml of the still cold extract is taken for dispersive SPE according to 5.4.2.

NOTE Freezing out also helps to partly remove some additional sample co-extractives with limited solubility in acetonitrile such as sugars. Co-extracted fat and waxes, which may negatively affect the ruggedness of GC analysis can be also effectively removed using silica-based reversed phase sorbents (ODS-, C18-type). For this, 25 mg ODS together with 25 mg PSA and 150 mg magnesium sulfate per millilitre extract is employed in the dispersive SPE step. Pesticides and the proposed internal and QC-standards (see Table 1) are not affected by these treatments.

5.4.2 Clean-up with amino-sorbent („Dispersive SPE“ with PSA)

An aliquot of 6 ml of the acetonitrile phase from 5.3 or 5.4.1 is transferred into a PP-single use centrifuge tube (4.5) already containing 150 mg PSA (3.14) and 900 mg of magnesium sulfate (3.6). Close the tube, shake vigorously for 30 s and centrifuge (for 5 min at > 3000 g). Immediately isolate and acidify the clear extract as described in 5.4.4.

For 1 ml of extract 150 mg magnesium sulfate and 25 mg PSA are necessary.

NOTE It is helpful to load the centrifuge tubes with the dispersive SPE sorbents before beginning the extraction procedure needed for one batch of samples. The use of a sample divider (4.12) substantially facilitates this procedure.

5.4.3 Clean-up with a mixture of amino-sorbent + GCB („Dispersive SPE“ with PSA + GCB)

For samples, with a high content of carotinoides (e.g. red sweet pepper, carrots) or chlorophyll (e.g. spinach, lamb's lettuce, rucola, curly kale, vine leaves und *Lactuca* varieties except iceberg lettuce and lettuce hearts), dispersive SPE is performed using a combination of PSA and GCB.

Transfer an aliquot of 6 ml of the acetonitrile phase from 5.3 into a centrifuge tube (4.5), which already contains 150 mg PSA (3.14) and for extracts of carrots, *Lactuca* varieties 900 mg of sorption mixture 1 (3.16), and for extracts of red sweet pepper, spinach, lamb's lettuce or rucola 900 mg of sorption mixture 2 (3.17). Close the tube, shake vigorously for 2 min and centrifuge (e.g. for 5 min at > 3 000 g). Immediately isolate and acidify the clear extract as described in 5.4.4.

For 1 ml extract 25 mg PSA and, depending on the sample, 150 mg sorption mixture 1 or 2 are necessary.

NOTE It is helpful to load the centrifuge tubes with the dispersive SPE sorbents before beginning the extraction procedure needed for one batch of samples. The use of a sample divider (4.12) substantially facilitates this procedure.

5.4.4 Extract stabilisation

Transfer 5 ml aliquot of the cleaned-up extract from 5.4.2 or 5.4.3 into a screw cup storage vial (4.10), taking care to avoid sorbent particles of being carried over, and slightly acidify by adding 50 µl of a 5 % formic acid solution in acetonitrile (3.13). Fill the pH-adjusted extract into auto-sampler vials and use it for gas- and liquid chromatographic analysis. Store the residual extract in a refrigerator to be used if necessary.

For 1 ml extract 10 µl of the formic acid solution (3.13) are necessary.

5.5 Determination

Inject the sample extracts derived from 5.4.4 (in the case of acidic pesticides use the raw extracts from 5.3) and standard solutions (3.22) into the GC or LC instruments in an appropriate sequences. This may involve bracketing of the sample extracts with the calibration solutions.

The measurement may be performed using various instruments, instrument parameters and columns. Some instrument parameters and columns are listed in Annex A. These conditions have been shown to provide satisfactory results.

For suitable experimental conditions of LC-MS/MS measurements, see CEN/TR 15641 [5]. For suitable experimental conditions of GC-MS measurement, refer to [3]. Nevertheless, individual tuning of the compounds on the instrument that is used for measurement usually provides better sensitivities.

5.6 Test for interference and recovery

Prepare reagent blanks and carry out spiked recovery tests at various appropriate levels. The chromatogram of the reagent blank should not show any significantly interfering peak at the retention time of the analytes.

6 Evaluation of results

6.1 Identification and quantification

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

- 1) retention time of the analyte in question (RT) or, even better, the retention time ratio against the ISTD ($R_{t(A)}/R_{t(ISTD)}$) obtained from the same run;
- 2) peak shape of the analyte; and
- 3) in case of MS or MS/MS detection, the relative abundance of the recorded masses (in general 2 SRM transitions are required in MS/MS and 3 ions in MS applications).

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 may 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 refer to the EU-quality control guidelines described in the SANCO/2007/3131 document [1]. Table 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 (3.22.1 or 3.22.2) to check linearity and to determine the calibration functions for each active substance as described in 6.2. 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 (3.22.1) 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 MRL violations), a more precise determination using matrix-matched standards (3.22.2) or the standard addition method (6.3) should be used.

NOTE 1 Matrix effects influence the response of target analytes in sample extracts compared to the response of standard solutions in pure solvent.

NOTE 2 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 standard prescribes the use of an internal standard for quantification and identification. Nevertheless, it is still possible to quantify without ISTD. Without ISTD, the volume of the acetonitrile phase (5.3) is assumed to be identical to the volume of acetonitrile added to the sample in 5.2.3 (10 ml).

When using internal standards it is important to know that any shift in the ISTD signal will directly influence the calculated concentration of the analytes. Ideally, the ISTD signal should only shift due to volume differences and thus improve the accuracy of measurement. However, there are also other, non-desirable, factors that may also affect the signals of the ISTD thus introducing errors in the analyte quantification. Losses of the ISTD during partitioning or clean-up will result in an overestimation of analyte concentration. Such losses should thus be minimal. Experiments have shown that the recovery of the ISTD is very high so that the error introduced to the analyte results due to the ISTD-losses remains insignificant compared to other sources of errors. A specific suppression of the ISTD signal, potentially occurring in LC-MS applications due to co-eluting matrix components, will also result in analyte overestimations, while a relative enhancement of the ISTD signal, typical in GC applications due to presence of matrix co-extractives, will result in underestimated analyte concentrations. ISTDs used in GC applications should thus be compounds that are practically not affected by matrix-induced enhancement phenomena. In LC-MS applications matrix effects will depend on whether the commodity extract contains specific components that will co-elute with the ISTD and affect its ionisation process.

In any case it is always crucial to introduce quality control measures to ensure that any error introduced by the ISTD remains insignificant. Quality control measures may include the use of backup ISTDs and quality control standards that may be added at other stages of the analytical procedure (e.g. to the final extract) and that may help to identify any non-volume related shifts of the ISTD signal. Very helpful for quality control is the observation of the signal intensity of the ISTD in every sample within a sequence. Should a significant signal shift occur, quantification should be performed using a backup ISTD or without using ISTD. In the latter case exact liquid transfers and equalisation of the volumes of the standard solutions and the sample extracts is mandatory.

6.2 Calculation of residue concentration without standard addition

Variables used:

— Concentration of internal standard in ISTD solution	C_{ISTD}	µg/ml
— Concentration of pesticide in calibration mixture	$C_{pest}^{cal\ mix}$	µg/ml
— Concentration of internal standard in calibration mixture	$C_{ISTD}^{cal\ mix}$	µg/ml
— Concentration of pesticide in the final extract	C_{pest}^{sample}	µg/ml
— Concentration of internal standard in the final extract	C_{ISTD}^{sample}	µg/ml
— Mass of test portion	m_a	g
— Volume of ISTD added to the test portion	V_{ISTD}^{sample}	ml
— Peak area of pesticide obtained from calibration mixture	$A_{pest}^{cal\ mix}$	(counts)
— Peak area of ISTD obtained from calibration mixture	$A_{ISTD}^{cal\ mix}$	(counts)
— Peak area of pesticide obtained from the final extract	A_{pest}^{sample}	(counts)
— Peak area of ISTD obtained from the final extract	A_{ISTD}^{sample}	(counts)
— Peak ratio obtained from calibration mixture	$PR^{cal\ mix}$	(dimensionless)

— Peak ratio obtained from final extract	PR^{sample}	(dimensionless)
— Slope of calibration graph	a_{cal}	(dimensionless)
— Bias of calibration graph	b_{cal}	(dimensionless)
— Mass fraction of pesticide in the sample	w_R	mg/kg

Furthermore in the text:

— Mass of the internal standard(s) to be employed for preparation of standard solution	$m_{ISTD}^{cal\ mix}$
— Resulting mass of pesticide added to each vial (for standard addition)	$m_{pest}^{std\ add}$
— Volume of the standard solution (solvent based or matrix-matched standard)	$V^{cal\ mix}$
— Volume of the internal standard solution to be employed for preparation of standard solution	$V_{ISTD}^{cal\ mix}$
— Volume of the pesticide working solutions used to prepare the calibration mixtures	$V_{pest}^{cal\ mix}$
— Added volume of an appropriate dilution of pesticide stock solution (for standard addition)	$V_{pest}^{std\ add}$

Determine the calibration functions for each active substance by plotting the peak ratio $PR^{cal\ mix}$ ($A_{pest}^{cal\ mix} / A_{ISTD}^{cal\ mix}$) of each calibration level against the dimensionless concentration ratio ($C_{pest}^{cal\ mix} / C_{ISTD}^{cal\ mix}$) of the standard solution. From the corresponding calibration graph described by the following formula:

$$PR^{cal\ mix} = a_{cal} \times \frac{C_{pest}^{cal\ mix}}{C_{ISTD}^{cal\ mix}} + b_{cal} \quad (1)$$

each expected concentration ratio ($C_{pest}^{cal\ mix} / C_{ISTD}^{cal\ mix}$) can be calculated as follows:

$$\frac{C_{pest}^{cal\ mix}}{C_{ISTD}^{cal\ mix}} = \frac{PR^{cal\ mix} - b_{cal}}{a_{cal}} \quad (2)$$

The concentration ratio $C_{pest}^{sample} / C_{ISTD}^{sample}$ in the final extract depends on the mass fraction w_R of the pesticide in the test portion m_a , the concentration of the internal standard C_{ISTD} and its volume V_{ISTD}^{sample} added to the test portion.

$$\frac{C_{pest}^{sample}}{C_{ISTD}^{sample}} = \frac{w_R \times m_a}{C_{ISTD} \times V_{ISTD}^{sample}} \quad (3)$$

When the peak ratio $PR^{sample} = A_{pest}^{sample} / A_{ISTD}^{sample}$ obtained from the final extract is identical to the peak ratio $PR^{cal mix}$ obtained from the calibration mixture, the concentration ratios $C_{pest}^{sample} / C_{ISTD}^{sample}$ and $C_{pest}^{cal mix} / C_{ISTD}^{cal mix}$ are identical and the mass fraction w_R (residue concentration in the test sample) is calculated as follows:

$$w_R = \frac{(PR^{sample} - b_{cal}) \times C_{ISTD} \times V_{ISTD}^{sample}}{a_{cal} \times m_a} \left(\frac{\text{mg}}{\text{kg}} \right) \quad (4)$$

NOTE A simplified and practice-oriented formula for the calculation of the results, which does not contain the concentration of the ISTD but requires certain preconditions to be met, can be found in Annex D.

6.3 Calculation of residue concentration using the standard additions approach

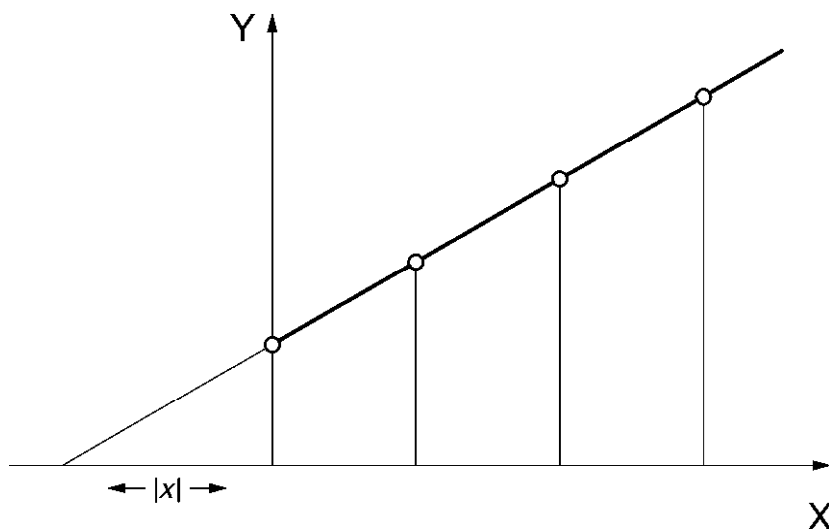
In case of suspected violative residues, or for compounds which are known to be strongly affected by matrix-induced enhancement or suppression phenomena, the procedure of standard additions is recommended provided that the function between response and concentrations at the concentration range in question is linear. In that case several aliquots of the final sample extract are fortified with increasing volumes ($V_{pest}^{std add}$) of an appropriate dilution of pesticide stock solutions (3.20) followed by a solvent adjustment as described in Table 4. This procedure requires knowledge of the approximate residue level w_R from preliminary analysis.

Assuming a sample (used sample amount 10 g) with an estimated residue level of $w_R = 0,8 \text{ mg/kg}$, the pipetting scheme as in Table 4 may be appropriate. The amount of analyte in the sample is calculated using a graphical presentation as shown in Figure 1 via linear regression.

NOTE In the case of other residue levels w_R an adjusted concentration of the analyte standard solution and/or more appropriate volumes of analyte standard solution and solvent are needed.

Table 4 — Exemplary pipetting scheme of a standard additions approach

Additions	Vial 1	Vial 2	Vial 3	Vial 4
Volume of sample extract (V_{al})	500 μl (= 0,5 g sample)	500 μl (= 0,5 g sample)	500 μl (= 0,5 g sample)	500 μl (= 0,5 g sample)
ISTD	Already contained	Already contained	Already contained	Already contained
Added volume ($V_{pest}^{std add}$) of an appropriate dilution of pesticide stock solution, (e.g. 4 $\mu\text{g/ml}$)	-	50 μl	100 μl	150 μl
Resulting mass ($m_{pest}^{std add}$) of pesticide added to each vial		0,2 μg	0,4 μg	0,6 μg
Volume of solvent	150 μl	100 μl	50 μl	-
Final volume	650 μl	650 μl	650 μl	650 μl



Key

Y Ratio of peak area of analyte divided by peak area of ISTD

X Added absolute mass of analyte $m_{pest}^{std\ add}$ in μg

$|x|$ Absolute amount of analyte in the sample extract (in μg) before standard addition ($y = 0$)

$$x = \frac{y - \text{intercept } (c)}{\text{slope of the curve } (b)} \quad (5)$$

Figure 1 — Internal calibration using the procedure of standard additions, schematically

The calculation is performed by using Equation (6) and the regression graph shown in Figure 1.

$$w_R = \frac{c}{b} \times \frac{V}{V_{al} \times m_a} \left(\frac{\text{mg}}{\text{kg}} \right) \quad (6)$$

where:

c is the Y-intercept of the calibration graph of the analyte in question;

b is the slope of the calibration graph of the analyte in question ($1/\mu\text{g}$);

V is the volume of acetonitrile added in 5.2.3 (ml);

V_{al} is the volume of the aliquots used for the standard additions approach (ml);

m_a is the initial sample weight (g).

6.4 Reliance of the method

Several recovery experiments (spiking levels 0,01 mg/kg to 0,25 mg/kg) have been performed within the frame of coordinated interlaboratory method validation studies with this method using representative commodities. The recoveries obtained were usually between 70 % and 110 % and relative standard deviations for replicate analysis below 10 %. The results of these validation studies are shown in Table B.1 in Annex B.

Table B 2 in Annex B shows a compilation of recovery study results individually performed in various laboratories within the frame of the method validation and on-going quality control.

The detection and determination limits obtained by this method strongly depend on numerous parameters including the type of the pesticide and sample in question and the sensitivity and selectivity obtained by the instrumentation at the conditions employed. Using state-of-the-art instrumentation, determinative analysis of pesticides at levels around 0,01 mg/kg (usually lowest MRL) is in most cases easily achieved.

7 Confirmatory tests

A confirmation of quantity involves analysis of a second sample portion and is to be performed if the first analysis indicates a suspected violative residue. For more information about the confirmation of identity refer to the EU-quality control guidelines described in the SANCO/2007/3131 document [1].

8 Precision

Details of the inter-laboratory test of the precision of the method according to ISO 5725-1 and ISO 5725-2 are summarised in Annex B. The values derived from the inter-laboratory test may not be applicable to pesticide concentration ranges and matrices other than given in Annex B.

9 Test report

The test report shall contain at least the following:

- all information necessary for the identification of the sample;
- reference to this European Standard;
- results and the units in which the results have been expressed;
- date and type of sampling procedure (if possible);
- date of receipt of sample in the laboratory;
- 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.

Annex A (informative)

Examples of experimental conditions

The following GC or LC-MS operating conditions have been shown to be satisfactory.

A.1 GC-MSD-System

Column	DB 5 MS crosslinked, 30 m x 0,25 mm x 0,25 µm, 5 % Ph Me Silicon
Carrier gas	Helium, constant flow 2 ml/min
GC temperature program	2 min at 40 °C then with 30 °C/min to 220 °C then with 5 °C/min to 260 °C then with 20 °C/min to 280 °C (15 min)
Transfer-line	280 °C
Injection volume	3 µl (PTV, solvent vent mode)
PTV temperature program	0,8 min at 50 °C then with 720 °C/min to 300 °C, hold 5 min then cool down to 280 °C, hold 10 min
PTV gas flow	Vent flow 20 ml/min until 0,5 min Purge flow 47,4 ml/min starting at 2 min Gas saver 20 ml/min starting at 6 min

A.2 HPLC-System 1

For most LC-amenable compounds:

Column	Zorbax XDB C18, length 150 mm, inner diameter 2,1 mm, particle size 3,5 µm
Mobile phase A ₁ (3.25)	Ammonium formate solution in water, c = 5 mmol/l
Mobile phase B ₁ (3.26)	Ammonium formate solution in methanol, c = 5 mmol/l
Column temperature	40 °C
Injection volume	5 µl

Table A.1 — Flow rate and elution gradient

Time min	Flow rate $\mu\text{l}/\text{min}$	Mobile phase A ₁ %	Mobile phase B ₁ %
0	300	50	50
20	300	0	100
25	300	0	100
26	300	50	50
30	300	50	50

A.3 HPLC-System 2

For polar compounds (e.g. with $\log K_{ow} < 0,5$) that show low retention at reversed-phased columns:

Column	Phenomenex Aqua, length 150 mm, inner diameter 2 mm, filled with 125 A C18-material, particle size 3 μm
Mobile phase A ₁ (3.25)	Ammonium formate solution in water, $c = 5 \text{ mmol/l}$
Mobile phase B ₁ (3.26)	Ammonium formate solution in methanol, $c = 5 \text{ mmol/l}$
Column temperature	40°C
Injection volume	3 μl , automatically diluted with 3 μl of mobile phase A during injection procedure

Table A.2 — Flow rate and elution gradient

Time min	Flow rate $\mu\text{l}/\text{min}$	Mobile phase A ₁ %	Mobile phase B ₁ %
0	100	100	0
3	100	30	70
6	300	15	85
9	300	10	90
20,5	300	10	90
21	300	100	0
32	300	100	0

NOTE Should the possibility for an automated dilution of the solutions in the instrument injector not exist, these should be manually diluted with mobile phase A1 (1 + 1), and 6 μl thereof should be injected.

A.4 HPLC-System 3

For acidic compounds:

Column	Zorbax XDB C18, length 150 mm, inner diameter 2,1 mm, particle size 3,5 μm
Mobile phase A ₂ (3.27)	Acetic acid solution in water, $\rho = 0,1 \text{ ml glacial acetic acid / l}$
Mobile phase B ₂ (3.28)	Acetic acid solution in acetonitrile, $\rho = 0,1 \text{ ml glacial acetic acid / l}$
Column temperature	40 °C
Injection volume	5 μl

Table A.3 — Flow rate and elution gradient

Time min	Flow rate µl/min	Mobile phase A ₂ %	Mobile phase B ₂ %
0	300	80	20
20	300	0	100
22	300	0	100
22,1	300	80	20
30	300	80	20

A.5 HPLC system 4

For most LC-amenable compounds:

HPLC pump HP1100 Binary Pump (G1312A)

Autosampler HP1100 (G1313A)

Injector programme draw 5 µl Mobile phase A₃
draw 1 µl sample
wash needle with acetonitrile
draw 2 µl Mobile phase A₃
draw 1 µl sample
wash needle with acetonitrile
draw 2 µl Mobile phase A₃
draw 1 µl sample
wash needle with acetonitrile
draw 2 µl Mobile phase A₃
draw 1 µl sample
wash needle with acetonitrile
draw 5 µl Mobile phase A₃

Column Phenomenex Aqua 5 µ C18 125Å, 50 mm × 2 mm

Mobile phase A₃ (3.29) Methanol/water 2+8 (V/V) with 5 mmol/l ammonium formate

Mobile phase B₃ (3.30) Methanol/water 9+1 (V/V) with 5 mmol/l ammonium formate

Column temperature 20 °C

Table A.4 — Flow rate and elution gradient

Time min	Flow rate µl/min	Mobile phase A ₃ %	Mobile phase B ₃ %
0	200	100	0
11	200	0	100
23	200	0	100
25	200	100	0
33	200	100	0

Annex B (informative)

Precision data

In accordance with ISO 5725-1 and ISO 5725-2, the parameters given in Table B.1 have been defined in an inter-laboratory test. The precision data listed in Table B.2 are summarized from single laboratory method validation trails. An updated version of validation data can be found on the website www.crl-pesticides-datapool.eu [7], which is run by the EU Community Reference Laboratories for Pesticides.

Table B.1 — Results of interlaboratory method validation study of the German Working Group „Unterarbeitsgruppe Analytik der Bund-Länder-Arbeitsgruppe Pflanzenschutz- und Schädlingsbekämpfungsmittel“ as well as the Pesticide Working Group of the German Chemical Society (GDCh) (n approx. 23 000)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
2,4,5-T	LC	Acidic	0,010	105	8	15	3
	LC	Acidic	0,100	101	8	15	3
	LC	Water containing	0,010	104	15	15	3
	LC	Water containing	0,100	101	5	15	3
2,4,5-TP	LC	Acidic	0,010	99	2	14	3
	LC	Acidic	0,100	100	5	14	3
	LC	Water containing	0,010	102	8	14	3
	LC	Water containing	0,100	101	4	15	3
2,4-D	LC	Acidic	0,010	103	7	15	3
	LC	Acidic	0,025	97	5	15	3
	LC	Acidic	0,100	100	5	14	3
	LC	Acidic	0,250	102	8	15	3
	LC	Water containing	0,010	113	12	15	3
	LC	Water containing	0,025	97	6	30	3
	LC	Water containing	0,100	99	8	14	3
	LC	Water containing	0,250	101	8	35	4
2,4-DB	LC	Water containing	0,010	103	10	15	3
	LC	Water containing	0,100	99	8	15	3
4-CPA	LC	Acidic	0,010	95	10	11	3
	LC	Acidic	0,100	101	7	15	3
	LC	Water containing	0,100	104	5	15	3
Acephate	LC	Acidic	0,100	89	10	15	3
	LC	Dry (Cereal)	0,010	81	11	15	3
	LC	Dry (Cereal)	0,100	81	5	15	3

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Sugar containing	0,010	82	7	15	3
	LC	Sugar containing	0,100	81	6	15	3
	LC	Water containing	0,010	92	15	19	4
	LC	Water containing	0,100	88	8	20	4
Acetamiprid	LC	Acidic	0,010	97	9	25	5
	LC	Acidic	0,025	94	11	39	8
	LC	Acidic	0,100	98	7	25	5
	LC	Acidic	0,250	99	6	40	8
	LC	Dry (Cereal)	0,010	99	7	20	4
	LC	Dry (Cereal)	0,100	101	10	20	4
	LC	Sugar containing	0,010	94	11	22	5
	LC	Sugar containing	0,100	99	7	20	4
	LC	Water containing	0,010	98	6	25	5
	LC	Water containing	0,025	98	8	79	8
	LC	Water containing	0,100	99	6	25	5
	LC	Water containing	0,250	98	6	85	9
Aldicarb	LC	Acidic	0,010	96	7	15	3
	LC	Acidic	0,100	100	6	15	3
	LC	Dry (Cereal)	0,010	100	8	15	3
	LC	Dry (Cereal)	0,100	97	6	15	3
	LC	Sugar containing	0,010	99	8	15	3
	LC	Sugar containing	0,100	99	6	15	3
	LC	Water containing	0,010	98	7	20	4
	LC	Water containing	0,100	95	11	19	4
Avermectin B1a	LC	Acidic	0,100	99	9	15	3
	LC	Dry (Cereal)	0,100	96	12	15	3
Azoxystrobin	LC	Acidic	0,010	102	7	15	3
	LC	Acidic	0,025	99	11	40	8
	LC	Acidic	0,100	96	4	15	3
	LC	Acidic	0,250	102	7	40	8
	LC	Dry (Cereal)	0,010	95	8	15	3
	LC	Dry (Cereal)	0,100	99	3	15	3
	LC	Sugar containing	0,010	100	5	15	3
	LC	Sugar containing	0,100	100	4	15	3
	LC	Water containing	0,010	97	5	20	4
	LC	Water containing	0,025	100	6	78	8
	LC	Water containing	0,100	99	6	20	4
	LC	Water containing	0,250	101	6	84	9

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
Bendiocarb	LC	Acidic	0,010	98	7	15	3
	LC	Acidic	0,100	102	4	15	3
	LC	Dry (Cereal)	0,010	102	7	15	3
	LC	Dry (Cereal)	0,100	97	6	15	3
	LC	Sugar containing	0,010	100	10	15	3
	LC	Sugar containing	0,100	101	4	15	3
	LC	Water containing	0,010	98	8	20	4
	LC	Water containing	0,100	100	5	20	4
Bentazone	LC	Acidic	0,010	101	10	15	3
	LC	Acidic	0,100	102	6	15	3
	LC	Water containing	0,100	101	5	15	3
Boscalid	LC	Acidic	0,010	101	8	25	5
	LC	Acidic	0,100	98	6	25	5
	LC	Dry (Cereal)	0,010	101	12	20	4
	LC	Dry (Cereal)	0,100	102	5	20	4
	LC	Sugar containing	0,010	98	10	25	5
	LC	Sugar containing	0,100	100	14	20	4
	LC	Water containing	0,010	101	9	25	5
	LC	Water containing	0,100	100	7	25	5
Bromoxynil	LC	Acidic	0,010	99	5	14	3
	LC	Acidic	0,025	96	6	15	3
	LC	Acidic	0,100	106	7	15	3
	LC	Acidic	0,250	100	6	15	3
	LC	Water containing	0,010	96	12	15	3
	LC	Water containing	0,025	98	3	30	3
	LC	Water containing	0,100	104	7	15	3
	LC	Water containing	0,250	102	8	35	4
Buprofezin	LC	Acidic	0,010	93	9	25	5
	LC	Acidic	0,100	95	6	25	5
	LC	Dry (Cereal)	0,010	102	14	20	4
	LC	Dry (Cereal)	0,100	102	6	19	4
	LC	Sugar containing	0,010	97	8	25	5
	LC	Sugar containing	0,100	99	8	20	4
	LC	Water containing	0,010	101	10	24	5
	LC	Water containing	0,100	101	6	25	5
Butocarboxim	LC	Acidic	0,010	97	7	15	3
	LC	Acidic	0,100	97	8	15	3

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Dry (Cereal)	0,010	97	11	13	3
	LC	Dry (Cereal)	0,100	95	6	15	3
	LC	Sugar containing	0,010	99	11	14	3
	LC	Sugar containing	0,100	98	5	15	3
	LC	Water containing	0,010	103	9	15	3
	LC	Water containing	0,100	100	8	17	4
Carbaryl	LC	Acidic	0,010	103	7	15	3
	LC	Acidic	0,100	100	8	15	3
	LC	Dry (Cereal)	0,010	97	8	15	3
	LC	Dry (Cereal)	0,100	97	5	15	3
	LC	Sugar containing	0,010	98	9	15	3
	LC	Sugar containing	0,100	97	6	15	3
	LC	Water containing	0,010	102	8	20	4
	LC	Water containing	0,100	102	4	20	4
Carbendazim	LC	Acidic	0,010	88	4	15	3
	LC	Acidic	0,025	82	9	38	8
	LC	Acidic	0,100	89	5	15	3
	LC	Acidic	0,250	89	5	39	8
	LC	Dry (Cereal)	0,010	87	6	15	3
	LC	Dry (Cereal)	0,100	86	3	15	3
	LC	Sugar containing	0,010	86	7	15	3
	LC	Sugar containing	0,100	84	8	15	3
	LC	Water containing	0,010	93	4	20	4
	LC	Water containing	0,025	90	7	79	8
	LC	Water containing	0,100	93	6	20	4
	LC	Water containing	0,250	92	6	85	9
Carbofuran	LC	Acidic	0,010	102	6	15	3
	LC	Acidic	0,100	103	15	15	3
	LC	Dry (Cereal)	0,010	98	5	15	3
	LC	Dry (Cereal)	0,100	97	3	15	3
	LC	Sugar containing	0,010	99	5	15	3
	LC	Sugar containing	0,100	98	5	15	3
	LC	Water containing	0,010	97	6	20	4
	LC	Water containing	0,100	102	4	20	4
Carboxin	LC	Acidic	0,010	95	5	25	5
	LC	Acidic	0,100	96	4	25	5
	LC	Dry (Cereal)	0,010	100	7	20	4
	LC	Dry (Cereal)	0,100	98	6	20	4

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Sugar containing	0,010	91	13	25	5
	LC	Sugar containing	0,100	95	7	20	4
	LC	Water containing	0,010	73	7	17	4
	LC	Water containing	0,100	75	8	20	4
Chloridazon	LC	Acidic	0,010	89	9	20	4
	LC	Acidic	0,100	93	8	25	5
	LC	Dry (Cereal)	0,010	98	10	20	4
	LC	Dry (Cereal)	0,100	96	6	20	4
	LC	Sugar containing	0,010	96	9	20	4
	LC	Sugar containing	0,100	96	8	20	4
	LC	Water containing	0,010	98	7	25	5
	LC	Water containing	0,100	100	6	25	5
Chlorpyrifos	GC	Acidic	0,025	105	8	40	8
	GC	Acidic	0,250	102	5	40	8
	GC	Water containing	0,025	103	8	80	8
	GC	Water containing	0,250	104	5	75	8
Cinosulfuron^b	LC	Acidic	0,010	74	9	12	3
	LC	Acidic	0,100	75	9	13	3
	LC	Water containing	0,010	57	15	11	3
Clofentezine	LC	Acidic	0,010	98	9	25	5
	LC	Acidic	0,100	97	6	25	5
	LC	Dry (Cereal)	0,010	107	8	19	4
	LC	Dry (Cereal)	0,100	95	9	20	4
	LC	Sugar containing	0,010	103	16	22	5
	LC	Sugar containing	0,100	95	11	19	4
	LC	Water containing	0,010	105	8	25	5
	LC	Water containing	0,100	102	6	25	5
Cycloxydim	LC	Acidic	0,100	104	10	15	3
Cyhalothrin, lambda-	GC	Acidic	0,025	116	16	26	6
	GC	Acidic	0,250	106	13	35	7
	GC	Water containing	0,025	113	13	57	7
	GC	Water containing	0,250	100	7	64	7
Cymoxanil	LC	Acidic	0,010	105	14	20	4
	LC	Acidic	0,100	94	8	25	5
	LC	Dry (Cereal)	0,010	109	8	20	4
	LC	Dry (Cereal)	0,100	103	9	20	4

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Sugar containing	0,010	111	12	19	4
	LC	Sugar containing	0,100	101	11	20	4
	LC	Water containing	0,010	108	12	19	4
	LC	Water containing	0,100	98	5	20	4
Cyproconazole	LC	Acidic	0,010	95	8	24	5
	LC	Acidic	0,100	98	7	25	5
	LC	Dry (Cereal)	0,010	94	10	18	4
	LC	Dry (Cereal)	0,100	99	6	20	4
	LC	Sugar containing	0,010	94	12	22	5
	LC	Sugar containing	0,100	98	8	20	4
	LC	Water containing	0,010	99	6	25	5
	LC	Water containing	0,100	101	5	25	5
Cyprodinil	GC	Acidic	0,025	94	9	25	5
	GC	Acidic	0,250	99	6	25	5
	GC	Water containing	0,025	101	7	50	5
	GC	Water containing	0,250	101	6	50	5
	LC	Acidic	0,010	97	6	15	3
	LC	Acidic	0,025	97	5	30	6
	LC	Acidic	0,100	97	4	15	3
	LC	Acidic	0,250	100	5	30	6
	LC	Dry (Cereal)	0,010	92	6	15	3
	LC	Dry (Cereal)	0,100	98	6	15	3
	LC	Sugar containing	0,010	101	8	15	3
	LC	Sugar containing	0,100	97	5	15	3
	LC	Water containing	0,010	98	10	20	4
	LC	Water containing	0,025	97	6	60	6
	LC	Water containing	0,100	99	7	20	4
	LC	Water containing	0,250	98	5	65	7
Cyromazine	LC	Water containing	0,100	48	5	12	3
Demeton-S-Methylsulfon	LC	Acidic	0,010	85	10	25	4
	LC	Acidic	0,100	90	8	30	4
	LC	Dry (Cereal)	0,010	95	7	30	4
	LC	Dry (Cereal)	0,100	99	6	25	4
	LC	Sugar containing	0,010	95	9	24	4
	LC	Sugar containing	0,100	100	8	30	4
	LC	Water containing	0,010	95	9	29	5
	LC	Water containing	0,100	99	6	35	5

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
Demeton-S-Methylsulfoxid	LC	Water containing	0,010	98	5	14	3
	LC	Water containing	0,100	92	6	15	3
Dicamba	LC	Acidic	0,100	88	11	14	3
	LC	Water containing	0,100	103	9	15	3
Dichlorprop (incl. Dichlorprop-P)	LC	Acidic	0,010	99	9	15	3
	LC	Acidic	0,100	97	8	13	3
	LC	Water containing	0,010	100	7	15	3
	LC	Water containing	0,100	102	5	14	3
Difenoconazole	LC	Acidic	0,010	93	9	25	5
	LC	Acidic	0,100	99	6	25	5
	LC	Dry (Cereal)	0,010	96	9	20	4
	LC	Dry (Cereal)	0,100	96	8	20	4
	LC	Sugar containing	0,010	98	9	25	5
	LC	Sugar containing	0,100	100	9	20	4
	LC	Water containing	0,010	101	9	25	5
	LC	Water containing	0,100	102	6	25	5
Dimethachlor	LC	Acidic	0,010	101	7	25	5
	LC	Acidic	0,100	98	5	25	5
	LC	Dry (Cereal)	0,010	103	5	20	4
	LC	Dry (Cereal)	0,100	102	6	20	4
	LC	Sugar containing	0,010	107	6	25	5
	LC	Sugar containing	0,100	101	8	20	4
	LC	Water containing	0,010	104	7	25	5
	LC	Water containing	0,100	101	6	25	5
Dimethoate	LC	Acidic	0,010	98	4	15	3
	LC	Acidic	0,025	93	8	38	8
	LC	Acidic	0,100	97	5	15	3
	LC	Acidic	0,250	99	6	40	8
	LC	Dry (Cereal)	0,010	99	4	15	3
	LC	Dry (Cereal)	0,100	97	6	15	3
	LC	Sugar containing	0,010	97	6	15	3
	LC	Sugar containing	0,100	96	5	15	3
	LC	Water containing	0,010	99	6	20	4
	LC	Water containing	0,025	98	6	75	8
	LC	Water containing	0,100	100	4	20	4
	LC	Water containing	0,250	98	8	84	9

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
Dimethomorph	LC	Acidic	0,010	94	7	25	5
	LC	Acidic	0,100	97	5	24	5
	LC	Dry (Cereal)	0,010	102	7	20	4
	LC	Dry (Cereal)	0,100	102	6	20	4
	LC	Sugar containing	0,010	99	8	25	5
	LC	Sugar containing	0,100	98	5	19	4
	LC	Water containing	0,010	96	8	25	5
	LC	Water containing	0,100	99	5	25	5
Diniconazole	LC	Acidic	0,010	91	10	24	5
	LC	Acidic	0,100	99	5	25	5
	LC	Dry (Cereal)	0,010	94	14	19	4
	LC	Dry (Cereal)	0,100	97	9	20	4
	LC	Sugar containing	0,010	97	8	20	4
	LC	Sugar containing	0,100	102	8	20	4
	LC	Water containing	0,010	99	10	25	5
	LC	Water containing	0,100	101	7	25	5
Epoxiconazole	LC	Acidic	0,010	97	9	25	5
	LC	Acidic	0,100	99	5	25	5
	LC	Dry (Cereal)	0,010	103	8	15	3
	LC	Dry (Cereal)	0,100	100	6	15	3
	LC	Sugar containing	0,010	101	7	25	5
	LC	Sugar containing	0,100	99	7	20	4
	LC	Water containing	0,010	97	6	25	5
	LC	Water containing	0,100	102	6	25	5
Ethiofencarb	LC	Acidic	0,010	96	11	14	3
	LC	Acidic	0,100	98	6	15	3
	LC	Dry (Cereal)	0,010	98	10	15	3
	LC	Dry (Cereal)	0,100	95	3	15	3
	LC	Sugar containing	0,010	93	14	15	3
	LC	Sugar containing	0,100	91	14	15	3
	LC	Water containing	0,010	77	17	15	3
Ethoprophos	LC	Acidic	0,010	98	9	25	5
	LC	Acidic	0,100	98	5	25	5
	LC	Dry (Cereal)	0,010	98	6	20	4
	LC	Dry (Cereal)	0,100	102	7	20	4
	LC	Sugar containing	0,010	97	6	25	5
	LC	Sugar containing	0,100	101	8	20	4
	LC	Water containing	0,010	100	4	25	5

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Water containing	0,100	102	5	25	5
Famoxadone	LC	Acidic	0,010	100	12	25	5
	LC	Acidic	0,100	100	6	25	5
	LC	Dry (Cereal)	0,010	103	7	20	4
	LC	Dry (Cereal)	0,100	100	6	20	4
	LC	Sugar containing	0,010	102	9	25	5
	LC	Sugar containing	0,100	99	9	20	4
	LC	Water containing	0,010	97	9	25	5
	LC	Water containing	0,100	101	7	25	5
Fenarimol	LC	Acidic	0,010	96	8	25	5
	LC	Acidic	0,100	98	6	25	5
	LC	Dry (Cereal)	0,010	89	8	20	4
	LC	Dry (Cereal)	0,100	98	6	20	4
	LC	Sugar containing	0,010	95	12	24	5
	LC	Sugar containing	0,100	96	9	20	4
	LC	Water containing	0,010	97	10	25	5
	LC	Water containing	0,100	99	7	25	5
Fenazaquin	LC	Acidic	0,010	92	8	20	4
	LC	Acidic	0,100	95	7	20	4
	LC	Dry (Cereal)	0,010	107	7	15	3
	LC	Dry (Cereal)	0,100	99	5	15	3
	LC	Sugar containing	0,010	100	10	20	4
	LC	Sugar containing	0,100	92	14	15	3
	LC	Water containing	0,010	98	8	20	4
	LC	Water containing	0,100	99	6	20	4
Fenhexamid	GC	Acidic	0,025	83	12	12	3
	GC	Acidic	0,250	87	12	15	3
	GC	Water containing	0,025	82	10	23	3
	GC	Water containing	0,250	80	10	22	3
	LC	Acidic	0,010	101	7	15	3
	LC	Acidic	0,025	83	11	39	8
	LC	Acidic	0,100	94	5	15	3
	LC	Acidic	0,250	89	9	40	8
	LC	Dry (Cereal)	0,010	78	9	15	3
	LC	Dry (Cereal)	0,100	77	15	12	3
	LC	Sugar containing	0,010	86	12	13	3
	LC	Sugar containing	0,100	84	11	15	3
	LC	Water containing	0,010	78	13	19	4

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Water containing	0,025	77	11	74	8
	LC	Water containing	0,100	80	12	14	4
	LC	Water containing	0,250	77	10	84	9
Fenoxaprop-P	LC	Acidic	0,010	103	5	15	3
	LC	Acidic	0,100	104	8	15	3
	LC	Water containing	0,010	96	10	15	3
	LC	Water containing	0,100	102	6	15	3
Fenoxycarb	LC	Acidic	0,010	96	6	15	3
	LC	Acidic	0,100	106	9	15	3
	LC	Dry (Cereal)	0,010	102	10	15	3
	LC	Dry (Cereal)	0,100	96	3	15	3
	LC	Sugar containing	0,010	101	6	15	3
	LC	Sugar containing	0,100	103	4	15	3
	LC	Water containing	0,010	99	8	20	4
	LC	Water containing	0,100	99	5	20	4
Fenpropidin	LC	Acidic	0,010	93	8	25	5
	LC	Acidic	0,100	98	5	25	5
	LC	Dry (Cereal)	0,010	99	9	20	4
	LC	Dry (Cereal)	0,100	98	6	20	4
	LC	Sugar containing	0,010	101	6	25	5
	LC	Sugar containing	0,100	101	7	20	4
	LC	Water containing	0,010	94	5	25	5
	LC	Water containing	0,100	100	6	25	5
Fenpropimorph	LC	Acidic	0,010	99	4	15	3
	LC	Acidic	0,100	101	7	15	3
	LC	Dry (Cereal)	0,010	101	8	15	3
	LC	Dry (Cereal)	0,100	97	7	15	3
	LC	Sugar containing	0,010	99	5	15	3
	LC	Sugar containing	0,100	100	6	15	3
	LC	Water containing	0,010	97	5	20	4
	LC	Water containing	0,100	98	5	20	4
Fenpyroximate	LC	Acidic	0,010	95	12	25	5
	LC	Acidic	0,100	97	8	25	5
	LC	Dry (Cereal)	0,010	105	10	20	4
	LC	Dry (Cereal)	0,100	101	5	20	4
	LC	Sugar containing	0,010	98	9	25	5
	LC	Sugar containing	0,100	95	13	20	4

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Water containing	0,010	99	7	25	5
	LC	Water containing	0,100	101	5	25	5
Fenthion	LC	Acidic	0,010	84	12	18	4
	LC	Acidic	0,100	100	7	20	4
	LC	Dry (Cereal)	0,010	90	7	15	3
	LC	Dry (Cereal)	0,100	96	11	15	3
	LC	Sugar containing	0,010	96	12	20	4
	LC	Sugar containing	0,100	98	12	20	4
	LC	Water containing	0,010	101	12	15	3
	LC	Water containing	0,100	92	11	20	4
Fluazifop	LC	Acidic	0,010	104	8	20	3
	LC	Acidic	0,100	106	7	20	3
	LC	Water containing	0,010	101	8	20	3
	LC	Water containing	0,100	103	4	20	3
Fludioxonil	LC	Acidic	0,010	98	7	15	3
	LC	Acidic	0,025	104	10	20	4
	LC	Acidic	0,100	100	7	14	3
	LC	Acidic	0,250	102	10	25	5
	LC	Water containing	0,010	96	4	15	3
	LC	Water containing	0,025	97	5	40	5
	LC	Water containing	0,100	97	6	15	3
	LC	Water containing	0,250	103	8	55	6
Flufenacet	LC	Acidic	0,010	96	11	25	5
	LC	Acidic	0,100	99	5	25	5
	LC	Dry (Cereal)	0,010	99	7	20	4
	LC	Dry (Cereal)	0,100	101	5	20	4
	LC	Sugar containing	0,010	100	7	25	5
	LC	Sugar containing	0,100	101	8	20	4
	LC	Water containing	0,010	99	4	25	5
	LC	Water containing	0,100	101	5	25	5
Flufenoxuron	LC	Acidic	0,010	106	11	15	3
	LC	Acidic	0,100	103	7	15	3
	LC	Dry (Cereal)	0,010	104	11	15	3
	LC	Dry (Cereal)	0,100	99	7	15	3
	LC	Sugar containing	0,010	101	11	14	3
	LC	Sugar containing	0,100	100	6	15	3
	LC	Water containing	0,010	108	14	20	4

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Water containing	0,100	101	6	20	4
Fluroxypyr	LC	Acidic	0,100	94	10	14	3
Flurtamone	LC	Acidic	0,010	97	7	25	5
	LC	Acidic	0,100	98	6	25	5
	LC	Dry (Cereal)	0,010	102	7	20	4
	LC	Dry (Cereal)	0,100	103	6	20	4
	LC	Sugar containing	0,010	99	8	25	5
	LC	Sugar containing	0,100	103	9	20	4
	LC	Water containing	0,010	100	5	25	5
	LC	Water containing	0,100	101	7	25	5
Flusilazole	LC	Acidic	0,010	97	8	25	5
	LC	Acidic	0,100	100	5	25	5
	LC	Dry (Cereal)	0,010	102	7	20	4
	LC	Dry (Cereal)	0,100	101	4	20	4
	LC	Sugar containing	0,010	99	6	25	5
	LC	Sugar containing	0,100	99	8	20	4
	LC	Water containing	0,010	97	5	25	5
	LC	Water containing	0,100	102	6	25	5
Fomesafen	LC	Acidic	0,100	106	6	15	3
	LC	Water containing	0,010	101	9	15	3
	LC	Water containing	0,100	102	3	14	3
Haloxyfop	LC	Acidic	0,010	113	9	15	3
	LC	Acidic	0,100	106	7	15	3
	LC	Water containing	0,010	99	10	15	3
	LC	Water containing	0,100	102	7	15	3
Hexaconazole	LC	Acidic	0,010	95	10	24	5
	LC	Acidic	0,100	97	5	25	5
	LC	Dry (Cereal)	0,010	94	10	20	4
	LC	Dry (Cereal)	0,100	97	7	20	4
	LC	Sugar containing	0,010	101	8	25	5
	LC	Sugar containing	0,100	94	9	20	4
	LC	Water containing	0,010	95	8	25	5
	LC	Water containing	0,100	100	6	25	5
Hexythiazox	LC	Acidic	0,010	95	10	25	5
	LC	Acidic	0,100	97	7	25	5

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Dry (Cereal)	0,010	104	16	20	4
	LC	Dry (Cereal)	0,100	103	5	20	4
	LC	Sugar containing	0,010	101	8	25	5
	LC	Sugar containing	0,100	95	12	20	4
	LC	Water containing	0,010	98	10	24	5
	LC	Water containing	0,100	102	7	25	5
Imazalil	GC	Acidic	0,025	97	8	15	3
	GC	Acidic	0,250	102	9	15	3
	GC	Water containing	0,025	116	29	25	3
	GC	Water containing	0,250	102	9	30	3
	LC	Acidic	0,010	96	5	15	3
	LC	Acidic	0,025	93	10	40	8
	LC	Acidic	0,100	96	4	15	3
	LC	Acidic	0,250	98	7	40	8
	LC	Dry (Cereal)	0,010	94	7	15	3
	LC	Dry (Cereal)	0,100	94	4	15	3
	LC	Sugar containing	0,010	93	13	15	3
	LC	Sugar containing	0,100	89	10	15	3
	LC	Water containing	0,010	88	15	17	4
	LC	Water containing	0,025	96	10	79	8
	LC	Water containing	0,100	87	13	16	4
	LC	Water containing	0,250	96	6	85	9
Imazapyr	LC	Acidic	0,100	93	7	14	3
	LC	Water containing	0,010	92	6	15	3
	LC	Water containing	0,100	96	5	15	3
Imazaquin	LC	Acidic	0,010	99	9	20	3
	LC	Acidic	0,100	102	7	20	3
	LC	Water containing	0,010	97	6	20	3
	LC	Water containing	0,100	102	5	20	3
Imazethapyr	LC	Acidic	0,010	94	13	19	3
	LC	Acidic	0,100	96	6	20	3
	LC	Water containing	0,010	98	9	20	3
	LC	Water containing	0,100	100	4	20	3
Imazosulfuron^b	LC	Acidic	0,010	100	11	15	3
	LC	Acidic	0,100	96	10	15	3
	LC	Water containing	0,010	96	12	14	3
	LC	Water containing	0,100	95	10	15	3

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
Imidacloprid	LC	Acidic	0,010	96	5	15	3
	LC	Acidic	0,100	99	8	15	3
	LC	Dry (Cereal)	0,010	98	7	14	3
	LC	Dry (Cereal)	0,100	95	4	15	3
	LC	Sugar containing	0,010	95	10	13	3
	LC	Sugar containing	0,100	96	5	15	3
	LC	Water containing	0,010	98	8	19	4
	LC	Water containing	0,100	97	4	20	4
Indoxacarb	LC	Acidic	0,010	101	9	14	3
	LC	Acidic	0,100	103	6	15	3
	LC	Dry (Cereal)	0,010	114	10	15	3
	LC	Dry (Cereal)	0,100	98	5	15	3
	LC	Sugar containing	0,010	92	8	15	3
	LC	Sugar containing	0,100	102	7	15	3
	LC	Water containing	0,010	100	8	20	4
	LC	Water containing	0,100	97	6	20	4
loxynil	LC	Acidic	0,010	96	4	15	3
	LC	Acidic	0,100	97	4	14	3
	LC	Water containing	0,010	98	9	15	3
	LC	Water containing	0,100	102	4	15	3
lprovalicarb	LC	Acidic	0,010	102	6	15	3
	LC	Acidic	0,100	101	8	15	3
	LC	Dry (Cereal)	0,010	97	10	15	3
	LC	Dry (Cereal)	0,100	97	4	15	3
	LC	Sugar containing	0,010	99	7	15	3
	LC	Sugar containing	0,100	99	5	15	3
	LC	Water containing	0,010	100	7	20	4
	LC	Water containing	0,100	100	4	20	4
Isoproturon	LC	Acidic	0,010	103	7	15	3
	LC	Acidic	0,100	98	7	15	3
	LC	Dry (Cereal)	0,010	95	5	15	3
	LC	Dry (Cereal)	0,100	96	4	15	3
	LC	Sugar containing	0,010	97	8	15	3
	LC	Sugar containing	0,100	100	4	15	3
	LC	Water containing	0,010	102	11	17	4
	LC	Water containing	0,100	97	6	20	4

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
Kresoxim-Methyl	GC	Acidic	0,025	104	10	30	6
	GC	Acidic	0,250	103	10	29	6
	GC	Water containing	0,025	104	9	60	6
	GC	Water containing	0,250	103	7	60	6
	LC	Acidic	0,025	103	4	15	3
	LC	Acidic	0,250	104	5	15	3
	LC	Water containing	0,025	102	6	25	3
	LC	Water containing	0,250	101	9	34	4
Linuron	LC	Acidic	0,010	104	5	15	3
	LC	Acidic	0,100	101	4	15	3
	LC	Dry (Cereal)	0,010	98	8	15	3
	LC	Dry (Cereal)	0,100	102	4	15	3
	LC	Sugar containing	0,010	100	10	15	3
	LC	Sugar containing	0,100	98	7	15	3
	LC	Water containing	0,010	94	7	20	4
	LC	Water containing	0,100	101	7	20	4
Lufenuron	LC	Acidic	0,025	106	11	20	4
	LC	Acidic	0,250	101	6	25	5
	LC	Water containing	0,025	106	16	38	4
	LC	Water containing	0,250	101	10	55	6
MCPA	LC	Acidic	0,010	100	7	14	3
	LC	Acidic	0,100	99	3	15	3
	LC	Water containing	0,010	103	10	14	3
	LC	Water containing	0,100	101	4	15	3
MCPB	LC	Acidic	0,010	101	16	15	3
	LC	Acidic	0,100	106	11	15	3
	LC	Water containing	0,010	98	11	15	3
	LC	Water containing	0,100	103	7	15	3
MCPP	LC	Acidic	0,010	102	6	15	3
	LC	Acidic	0,100	98	9	15	3
	LC	Water containing	0,010	101	10	15	3
	LC	Water containing	0,100	101	2	14	3
Mepanipyrim	LC	Acidic	0,010	92	10	25	5
	LC	Acidic	0,100	100	7	25	5
	LC	Dry (Cereal)	0,010	101	9	20	4
	LC	Dry (Cereal)	0,100	99	8	20	4

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Sugar containing	0,010	101	7	20	4
	LC	Sugar containing	0,100	99	7	20	4
	LC	Water containing	0,010	96	7	25	5
	LC	Water containing	0,100	100	4	25	5
Metalaxyl	GC	Acidic	0,025	100	8	25	5
	GC	Acidic	0,250	98	7	25	5
	GC	Water containing	0,025	104	10	47	5
	GC	Water containing	0,250	104	5	50	5
	LC	Acidic	0,010	101	10	15	3
	LC	Acidic	0,025	95	10	35	7
	LC	Acidic	0,100	101	8	15	3
	LC	Acidic	0,250	101	6	35	7
	LC	Dry (Cereal)	0,010	98	10	15	3
	LC	Dry (Cereal)	0,100	99	6	15	3
	LC	Sugar containing	0,010	98	6	15	3
	LC	Sugar containing	0,100	100	7	15	3
	LC	Water containing	0,010	102	10	12	3
	LC	Water containing	0,025	100	7	68	7
	LC	Water containing	0,100	102	6	20	4
	LC	Water containing	0,250	100	6	74	8
Methamidophos	LC	Acidic	0,025	75	8	39	8
	LC	Acidic	0,100	83	13	14	3
	LC	Acidic	0,250	78	6	39	8
	LC	Dry (Cereal)	0,010	84	18	15	3
	LC	Dry (Cereal)	0,100	77	19	15	3
	LC	Sugar containing	0,100	85	16	14	3
	LC	Water containing	0,010	92	11	20	4
	LC	Water containing	0,025	81	10	77	8
	LC	Water containing	0,100	92	8	19	4
	LC	Water containing	0,250	81	7	84	9
Methiocarb	LC	Acidic	0,010	94	8	15	3
	LC	Acidic	0,025	99	11	38	8
	LC	Acidic	0,100	96	12	15	3
	LC	Acidic	0,250	101	5	40	8
	LC	Dry (Cereal)	0,010	90	9	15	3
	LC	Dry (Cereal)	0,100	89	8	15	3
	LC	Sugar containing	0,010	94	10	14	3
	LC	Sugar containing	0,100	89	11	12	3
	LC	Water containing	0,010	92	9	20	4

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Water containing	0,025	103	7	73	8
	LC	Water containing	0,100	97	7	20	4
	LC	Water containing	0,250	100	7	76	9
Methomyl	LC	Acidic	0,010	104	10	15	3
	LC	Acidic	0,100	97	8	15	3
	LC	Dry (Cereal)	0,010	98	8	15	3
	LC	Dry (Cereal)	0,100	90	6	15	3
	LC	Sugar containing	0,010	97	9	15	3
	LC	Sugar containing	0,100	94	6	15	3
	LC	Water containing	0,010	103	14	20	4
	LC	Water containing	0,100	99	6	18	4
Methoxyfenozide	LC	Acidic	0,010	101	5	15	3
	LC	Acidic	0,100	100	6	15	3
	LC	Dry (Cereal)	0,010	97	7	15	3
	LC	Dry (Cereal)	0,100	100	4	15	3
	LC	Sugar containing	0,010	104	6	15	3
	LC	Sugar containing	0,100	101	5	15	3
	LC	Water containing	0,010	95	10	20	4
	LC	Water containing	0,100	98	7	20	4
Metobromuron	LC	Acidic	0,010	93	13	23	5
	LC	Acidic	0,100	98	7	25	5
	LC	Dry (Cereal)	0,010	103	9	20	4
	LC	Dry (Cereal)	0,100	102	6	20	4
	LC	Sugar containing	0,010	102	10	25	5
	LC	Sugar containing	0,100	105	9	20	4
	LC	Water containing	0,010	96	7	25	5
	LC	Water containing	0,100	100	6	25	5
Metolachlor	LC	Acidic	0,010	104	7	15	3
	LC	Acidic	0,100	102	6	15	3
	LC	Dry (Cereal)	0,010	104	10	15	3
	LC	Dry (Cereal)	0,100	99	4	15	3
	LC	Sugar containing	0,010	101	8	15	3
	LC	Sugar containing	0,100	102	5	15	3
	LC	Water containing	0,010	97	6	20	4
	LC	Water containing	0,100	100	4	20	4
Metosulam	LC	Acidic	0,010	94	20	19	3
	LC	Acidic	0,100	96	22	23	3

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Water containing	0,010	104	3	15	3
	LC	Water containing	0,100	80	36	18	3
Metsulfuron-Methyl^b	LC	Acidic	0,010	88	19	27	4
	LC	Acidic	0,100	87	25	25	4
	LC	Sugar containing	0,010	64	36	19	3
	LC	Sugar containing	0,100	69	35	20	3
	LC	Water containing	0,010	84	22	27	4
	LC	Water containing	0,100	84	21	25	4
Monocrotophos	LC	Acidic	0,010	95	10	15	3
	LC	Acidic	0,100	96	8	15	3
	LC	Dry (Cereal)	0,010	93	7	15	3
	LC	Dry (Cereal)	0,100	90	3	15	3
	LC	Sugar containing	0,010	92	12	15	3
	LC	Sugar containing	0,100	94	7	15	3
	LC	Water containing	0,010	95	6	19	4
	LC	Water containing	0,100	97	5	19	4
Myclobutanil	GC	Acidic	0,025	101	6	30	6
	GC	Acidic	0,250	103	7	30	6
	GC	Water containing	0,025	104	7	60	6
	GC	Water containing	0,250	103	5	60	6
	LC	Acidic	0,025	101	7	20	4
	LC	Acidic	0,250	103	6	20	4
	LC	Water containing	0,025	98	8	40	4
	LC	Water containing	0,250	98	8	45	5
Naphthoxyacetic acid, 2-	LC	Acidic	0,100	103	8	15	3
	LC	Water containing	0,100	98	7	14	3
Omethoate	LC	Acidic	0,010	84	10	20	4
	LC	Acidic	0,100	83	8	20	4
	LC	Dry (Cereal)	0,010	92	9	15	3
	LC	Dry (Cereal)	0,100	88	15	20	4
	LC	Sugar containing	0,010	81	15	19	4
	LC	Sugar containing	0,100	89	4	20	4
	LC	Water containing	0,010	92	9	19	4
	LC	Water containing	0,100	89	7	25	5
Oxamyl	LC	Acidic	0,010	103	11	12	3
	LC	Acidic	0,100	96	8	15	3

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Dry (Cereal)	0,010	99	18	11	3
	LC	Dry (Cereal)	0,100	98	10	15	3
	LC	Sugar containing	0,100	98	7	15	3
	LC	Water containing	0,010	96	13	15	4
	LC	Water containing	0,100	101	7	20	4
Penconazole	GC	Acidic	0,025	101	5	30	6
	GC	Acidic	0,250	102	7	30	6
	GC	Water containing	0,025	103	6	60	6
	GC	Water containing	0,250	102	4	60	6
	LC	Acidic	0,025	97	6	25	5
	LC	Acidic	0,250	101	7	25	5
	LC	Water containing	0,025	102	8	49	5
	LC	Water containing	0,250	99	6	55	6
Picoxystrobin	LC	Acidic	0,010	102	5	15	3
	LC	Acidic	0,100	102	8	15	3
	LC	Dry (Cereal)	0,010	103	8	15	3
	LC	Dry (Cereal)	0,100	99	5	15	3
	LC	Sugar containing	0,010	103	6	15	3
	LC	Sugar containing	0,100	99	6	15	3
	LC	Water containing	0,010	98	7	20	4
	LC	Water containing	0,100	99	4	20	4
Pirimicarb	GC	Acidic	0,025	97	6	20	4
	GC	Acidic	0,250	96	6	20	4
	GC	Water containing	0,025	101	7	40	4
	GC	Water containing	0,250	101	5	40	4
	LC	Acidic	0,010	92	5	14	3
	LC	Acidic	0,025	95	4	30	6
	LC	Acidic	0,100	96	7	15	3
	LC	Acidic	0,250	99	4	30	6
	LC	Dry (Cereal)	0,010	98	4	15	3
	LC	Dry (Cereal)	0,100	98	4	15	3
	LC	Sugar containing	0,010	95	7	15	3
	LC	Sugar containing	0,100	94	4	15	3
	LC	Water containing	0,010	94	8	20	4
	LC	Water containing	0,025	98	8	59	6
	LC	Water containing	0,100	95	8	20	4
	LC	Water containing	0,250	96	6	65	7
Pirimiphos-Ethyl	LC	Acidic	0,010	93	9	15	3

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Acidic	0,100	96	6	15	3
	LC	Sugar containing	0,010	98	13	15	3
	LC	Sugar containing	0,100	99	10	15	3
	LC	Water containing	0,010	100	5	15	3
	LC	Water containing	0,100	100	6	15	3
Procymidone	GC	Acidic	0,025	103	6	35	7
	GC	Acidic	0,250	102	5	35	7
	GC	Water containing	0,025	104	5	70	7
	GC	Water containing	0,250	104	4	70	7
Profenofos	LC	Acidic	0,010	94	9	25	5
	LC	Acidic	0,100	96	5	25	5
	LC	Dry (Cereal)	0,010	113	22	14	4
	LC	Dry (Cereal)	0,100	103	5	20	4
	LC	Sugar containing	0,010	98	9	25	5
	LC	Sugar containing	0,100	99	10	20	4
	LC	Water containing	0,010	112	24	20	4
	LC	Water containing	0,100	100	5	25	5
Promecarb	LC	Acidic	0,010	101	3	15	3
	LC	Acidic	0,100	98	7	15	3
	LC	Dry (Cereal)	0,010	98	6	15	3
	LC	Dry (Cereal)	0,100	99	3	15	3
	LC	Sugar containing	0,010	102	6	15	3
	LC	Sugar containing	0,100	100	3	15	3
	LC	Water containing	0,010	97	5	20	4
	LC	Water containing	0,100	99	6	20	4
Prometryn	LC	Acidic	0,010	94	6	25	5
	LC	Acidic	0,100	97	5	25	5
	LC	Dry (Cereal)	0,010	98	7	20	4
	LC	Dry (Cereal)	0,100	102	4	20	4
	LC	Sugar containing	0,010	97	6	25	5
	LC	Sugar containing	0,100	100	6	20	4
	LC	Water containing	0,010	98	5	25	5
	LC	Water containing	0,100	100	5	25	5
Propamocarb	LC	Acidic	0,010	85	11	13	3
	LC	Acidic	0,025	78	7	40	8
	LC	Acidic	0,100	88	8	15	3
	LC	Acidic	0,250	80	6	40	8

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Dry (Cereal)	0,010	92	6	15	3
	LC	Dry (Cereal)	0,100	86	5	15	3
	LC	Sugar containing	0,010	81	7	15	3
	LC	Sugar containing	0,100	74	8	15	3
	LC	Water containing	0,010	97	11	15	4
	LC	Water containing	0,025	85	8	78	8
	LC	Water containing	0,100	93	10	20	4
	LC	Water containing	0,250	85	8	84	9
Propargite	LC	Acidic	0,010	96	8	25	5
	LC	Acidic	0,100	101	7	25	5
	LC	Dry (Cereal)	0,010	102	9	20	4
	LC	Dry (Cereal)	0,100	103	5	20	4
	LC	Sugar containing	0,010	100	8	25	5
	LC	Sugar containing	0,100	105	10	17	4
	LC	Water containing	0,010	102	6	24	5
	LC	Water containing	0,100	102	6	25	5
Propiconazole	LC	Acidic	0,010	94	7	20	4
	LC	Acidic	0,100	99	6	25	5
	LC	Dry (Cereal)	0,010	102	9	20	4
	LC	Dry (Cereal)	0,100	99	6	20	4
	LC	Sugar containing	0,010	95	8	20	4
	LC	Sugar containing	0,100	99	9	20	4
	LC	Water containing	0,010	95	7	25	5
	LC	Water containing	0,100	101	5	25	5
Propoxur	LC	Acidic	0,010	105	6	15	3
	LC	Acidic	0,100	98	4	15	3
	LC	Dry (Cereal)	0,010	96	7	15	3
	LC	Dry (Cereal)	0,100	98	4	15	3
	LC	Sugar containing	0,010	98	7	15	3
	LC	Sugar containing	0,100	99	5	15	3
	LC	Water containing	0,010	103	5	20	4
	LC	Water containing	0,100	98	5	20	4
Propyzamide	GC	Acidic	0,025	103	6	30	6
	GC	Acidic	0,250	101	7	30	6
	GC	Water containing	0,025	105	6	60	6
	GC	Water containing	0,250	105	5	60	6
	LC	Acidic	0,010	95	9	24	5
	LC	Acidic	0,025	96	8	20	4

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Acidic	0,100	101	6	25	5
	LC	Acidic	0,250	101	4	20	4
	LC	Dry (Cereal)	0,010	101	7	20	4
	LC	Dry (Cereal)	0,100	99	6	20	4
	LC	Sugar containing	0,010	100	8	25	5
	LC	Sugar containing	0,100	104	9	20	4
	LC	Water containing	0,010	98	6	25	5
	LC	Water containing	0,025	98	5	40	4
	LC	Water containing	0,100	100	5	25	5
	LC	Water containing	0,250	101	8	50	5
Prosulfuron^b	LC	Acidic	0,010	78	13	14	3
	LC	Acidic	0,100	77	9	15	3
	LC	Water containing	0,010	65	12	16	4
Pymetrozine	LC	Acidic	0,010	42	17	11	3
	LC	Acidic	0,100	46	12	15	3
	LC	Dry (Cereal)	0,010	60	7	15	3
	LC	Dry (Cereal)	0,100	55	5	15	3
	LC	Sugar containing	0,010	41	16	14	3
	LC	Sugar containing	0,100	39	18	15	3
	LC	Water containing	0,010	66	5	15	3
	LC	Water containing	0,100	63	6	20	4
Pyraclostrobin	LC	Acidic	0,010	101	9	15	3
	LC	Acidic	0,100	98	5	15	3
	LC	Dry (Cereal)	0,010	98	10	15	3
	LC	Dry (Cereal)	0,100	98	4	15	3
	LC	Sugar containing	0,010	100	8	15	3
	LC	Sugar containing	0,100	100	6	15	3
	LC	Water containing	0,010	101	7	20	4
	LC	Water containing	0,100	98	5	20	4
Pyridaben	GC	Acidic	0,025	103	9	30	6
	GC	Acidic	0,250	104	7	30	6
	GC	Water containing	0,025	101	6	60	6
	GC	Water containing	0,250	102	6	60	6
	LC	Acidic	0,025	108	8	15	3
	LC	Acidic	0,250	106	11	15	3
	LC	Water containing	0,025	102	10	29	3
	LC	Water containing	0,250	100	7	33	4

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
Pyrifenox	LC	Acidic	0,010	94	6	25	5
	LC	Acidic	0,100	97	5	25	5
	LC	Dry (Cereal)	0,100	99	5	20	4
	LC	Sugar containing	0,010	99	8	20	4
	LC	Sugar containing	0,100	98	7	20	4
	LC	Water containing	0,010	96	7	25	5
	LC	Water containing	0,100	101	6	25	5
Pyrimethanil	GC	Acidic	0,025	100	5	30	6
	GC	Acidic	0,250	98	5	30	6
	GC	Water containing	0,025	101	5	60	6
	GC	Water containing	0,250	101	3	60	6
	LC	Acidic	0,010	98	5	15	3
	LC	Acidic	0,025	98	8	30	6
	LC	Acidic	0,100	96	5	15	3
	LC	Acidic	0,250	101	6	30	6
	LC	Dry (Cereal)	0,010	92	6	15	3
	LC	Dry (Cereal)	0,100	94	4	15	3
	LC	Sugar containing	0,010	96	5	15	3
	LC	Sugar containing	0,100	98	5	15	3
	LC	Water containing	0,010	95	6	20	4
	LC	Water containing	0,025	100	9	59	6
	LC	Water containing	0,100	98	5	20	4
	LC	Water containing	0,250	97	5	65	7
Pyriproxyfen	LC	Acidic	0,010	97	7	25	5
	LC	Acidic	0,100	97	6	25	5
	LC	Dry (Cereal)	0,010	106	13	20	4
	LC	Dry (Cereal)	0,100	100	6	19	4
	LC	Sugar containing	0,010	97	8	25	5
	LC	Sugar containing	0,100	99	11	19	4
	LC	Water containing	0,010	105	10	24	5
	LC	Water containing	0,100	102	6	25	5
Quinmerac^c	LC	Acidic	0,100	64	38	24	3
	LC	Sugar containing	0,100	56	30	15	3
	LC	Water containing	0,010	79	33	20	4
	LC	Water containing	0,100	63	40	24	3
Quinoxifen	GC	Acidic	0,025	102	4	35	7
	GC	Acidic	0,250	101	4	35	7
	GC	Water containing	0,025	100	6	70	7

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	GC	Water containing	0,250	100	4	70	7
	LC	Acidic	0,010	94	10	25	5
	LC	Acidic	0,100	94	7	25	5
	LC	Dry (Cereal)	0,010	107	7	15	3
	LC	Dry (Cereal)	0,100	99	6	20	4
	LC	Sugar containing	0,010	93	9	25	5
	LC	Sugar containing	0,100	95	13	19	4
	LC	Water containing	0,010	100	9	23	5
	LC	Water containing	0,100	100	7	25	5
Spinosyn A	LC	Acidic	0,010	100	13	24	5
	LC	Acidic	0,100	99	6	25	5
	LC	Dry (Cereal)	0,010	111	11	20	4
	LC	Dry (Cereal)	0,100	105	7	20	4
	LC	Sugar containing	0,010	97	11	23	5
	LC	Sugar containing	0,100	100	7	20	4
	LC	Water containing	0,010	99	9	25	5
	LC	Water containing	0,100	101	9	25	5
Spinosyn D	LC	Acidic	0,010	95	10	18	4
	LC	Acidic	0,100	98	7	25	5
	LC	Dry (Cereal)	0,010	107	7	11	3
	LC	Dry (Cereal)	0,100	104	8	20	4
	LC	Sugar containing	0,010	103	21	20	5
	LC	Sugar containing	0,100	102	7	20	4
	LC	Water containing	0,010	91	15	25	5
	LC	Water containing	0,100	97	9	25	5
Spiroxamine	LC	Acidic	0,010	98	4	15	3
	LC	Acidic	0,100	98	5	15	3
	LC	Dry (Cereal)	0,010	96	5	15	3
	LC	Dry (Cereal)	0,100	95	6	15	3
	LC	Sugar containing	0,010	99	5	15	3
	LC	Sugar containing	0,100	99	4	15	3
	LC	Water containing	0,010	96	7	20	4
	LC	Water containing	0,100	97	6	20	4
Tebuconazole	LC	Acidic	0,010	104	11	15	3
	LC	Acidic	0,100	101	7	15	3
	LC	Dry (Cereal)	0,010	105	18	15	3
	LC	Dry (Cereal)	0,100	94	6	15	3
	LC	Sugar containing	0,010	102	8	15	3

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Sugar containing	0,100	100	6	15	3
	LC	Water containing	0,010	100	9	20	4
	LC	Water containing	0,100	99	5	20	4
Tebufenozide	LC	Acidic	0,010	103	5	15	3
	LC	Acidic	0,025	98	11	40	8
	LC	Acidic	0,100	99	4	15	3
	LC	Acidic	0,250	102	6	40	8
	LC	Dry (Cereal)	0,010	104	8	15	3
	LC	Dry (Cereal)	0,100	96	5	15	3
	LC	Sugar containing	0,010	105	5	15	3
	LC	Sugar containing	0,100	102	4	15	3
	LC	Water containing	0,010	100	8	20	4
	LC	Water containing	0,025	99	6	78	8
	LC	Water containing	0,100	104	8	20	4
	LC	Water containing	0,250	102	7	84	9
Tebufenpyrad	LC	Acidic	0,010	99	12	24	5
	LC	Acidic	0,100	96	10	25	5
	LC	Dry (Cereal)	0,010	119	30	14	3
	LC	Dry (Cereal)	0,100	100	5	20	4
	LC	Sugar containing	0,010	99	10	22	5
	LC	Sugar containing	0,100	100	8	20	4
	LC	Water containing	0,010	99	10	20	4
	LC	Water containing	0,100	100	7	25	5
Tetraconazole	LC	Acidic	0,010	95	9	25	5
	LC	Acidic	0,100	100	5	25	5
	LC	Dry (Cereal)	0,010	99	9	20	4
	LC	Dry (Cereal)	0,100	102	6	20	4
	LC	Sugar containing	0,010	100	8	25	5
	LC	Sugar containing	0,100	101	7	20	4
	LC	Water containing	0,010	94	8	25	5
	LC	Water containing	0,100	100	5	25	5
Tetradifon	GC	Acidic	0,025	102	7	34	7
	GC	Acidic	0,250	102	6	35	7
	GC	Water containing	0,025	102	9	64	7
	GC	Water containing	0,250	102	5	70	7
Thiabendazole	LC	Acidic	0,010	92	12	15	3
	LC	Acidic	0,025	85	8	38	8

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
	LC	Acidic	0,100	89	6	15	3
	LC	Acidic	0,250	91	7	40	8
	LC	Dry (Cereal)	0,010	89	7	15	3
	LC	Dry (Cereal)	0,100	86	4	15	3
	LC	Sugar containing	0,010	85	6	15	3
	LC	Sugar containing	0,100	86	14	15	3
	LC	Water containing	0,010	98	7	20	4
	LC	Water containing	0,025	92	9	79	8
	LC	Water containing	0,100	94	7	20	4
	LC	Water containing	0,250	91	7	85	9
Thiacloprid	LC	Acidic	0,010	98	9	15	3
	LC	Acidic	0,100	103	12	15	3
	LC	Dry (Cereal)	0,010	96	6	15	3
	LC	Dry (Cereal)	0,100	97	6	15	3
	LC	Sugar containing	0,010	101	6	15	3
	LC	Sugar containing	0,100	95	5	15	3
	LC	Water containing	0,010	95	7	20	4
	LC	Water containing	0,100	101	7	20	4
Thiamethoxam	LC	Acidic	0,010	89	9	20	4
	LC	Acidic	0,100	93	9	22	5
	LC	Dry (Cereal)	0,010	96	8	20	4
	LC	Dry (Cereal)	0,100	99	7	20	4
	LC	Sugar containing	0,010	92	11	19	4
	LC	Sugar containing	0,100	99	7	20	4
	LC	Water containing	0,010	96	8	20	4
	LC	Water containing	0,100	101	6	25	5
Thifensulfuron-Methyl^b	LC	Acidic	0,010	85	26	29	4
	LC	Acidic	0,100	88	24	25	4
	LC	Dry (Cereal)	0,010	66	39	23	3
	LC	Sugar containing	0,010	62	42	20	3
	LC	Sugar containing	0,100	61	37	20	3
	LC	Water containing	0,010	83	24	26	4
	LC	Water containing	0,100	85	21	25	4
Thiodicarb	LC	Acidic	0,100	96	7	25	5
	LC	Dry (Cereal)	0,100	81	11	15	3
	LC	Sugar containing	0,100	99	9	20	4
	LC	Water containing	0,100	99	5	25	5

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
Thiofanox	LC	Acidic	0,010	107	13	13	3
	LC	Acidic	0,100	98	8	15	3
	LC	Dry (Cereal)	0,100	98	5	15	3
	LC	Sugar containing	0,100	88	9	15	3
	LC	Water containing	0,010	92	14	20	4
	LC	Water containing	0,100	91	12	16	4
Thiophanate-Methyl	LC	Acidic	0,100	97	6	20	4
	LC	Dry (Cereal)	0,100	97	6	20	4
	LC	Sugar containing	0,100	97	7	20	4
	LC	Water containing	0,100	91	5	15	3
Triadimefon	LC	Acidic	0,010	99	10	25	5
	LC	Acidic	0,100	99	4	25	5
	LC	Dry (Cereal)	0,010	102	7	20	4
	LC	Dry (Cereal)	0,100	101	7	20	4
	LC	Sugar containing	0,010	95	7	25	5
	LC	Sugar containing	0,100	101	7	20	4
	LC	Water containing	0,010	102	8	25	5
	LC	Water containing	0,100	101	6	25	5
Triclopyr	LC	Acidic	0,100	100	8	15	3
	LC	Water containing	0,010	112	12	14	3
	LC	Water containing	0,100	104	5	14	3
Trifloxystrobin	LC	Acidic	0,010	96	8	25	5
	LC	Acidic	0,100	98	6	25	5
	LC	Dry (Cereal)	0,010	108	11	20	4
	LC	Dry (Cereal)	0,100	105	11	19	4
	LC	Sugar containing	0,010	102	6	25	5
	LC	Sugar containing	0,100	102	8	20	4
	LC	Water containing	0,010	105	15	24	5
	LC	Water containing	0,100	101	5	25	5
Trimethacarb	LC	Acidic	0,010	103	7	15	3
	LC	Acidic	0,100	95	6	15	3
	LC	Dry (Cereal)	0,010	97	6	15	3
	LC	Dry (Cereal)	0,100	97	3	15	3
	LC	Sugar containing	0,010	100	6	15	3
	LC	Sugar containing	0,100	99	5	15	3
	LC	Water containing	0,010	97	8	20	4
	LC	Water containing	0,100	99	5	20	4

Table B.1 (continued)

Pesticide	GC/ LC	Matrix type	Spiking Level mg/kg	Recoveries ^a			N° of Labs
				X %	V %	n	
Vamidothion	LC	Acidic	0,010	96	7	15	3
	LC	Acidic	0,100	98	12	15	3
	LC	Dry (Cereal)	0,010	96	7	15	3
	LC	Dry (Cereal)	0,100	97	5	15	3
	LC	Sugar containing	0,010	94	6	15	3
	LC	Sugar containing	0,100	96	7	15	3
	LC	Water containing	0,010	97	8	20	4
	LC	Water containing	0,100	99	7	20	4

a X = recovery, V = relative standard deviation; n = number of results
b Not analysed as described from the non-acidified extract.
c Not analysed as described from the raw extract.

Table B.2 — Results of single laboratory validations (n approximately 25 000)

Pesticide	LC/ GC	Matrix type	Spiking Level mg/kg		Recoveries ^a			N° of labs
			min	max	X %	V %	n	
Acephate		Summary	0,010	0,750	83	15	98	3
	GC	Acidic	0,050	0,050	66		1	1
	GC	Water containing	0,050	0,050	71	26	6	1
	LC	Sugar containing	0,020	0,020	72		1	1
	LC	Acidic	0,010	0,100	81	16	14	2
	LC	Dry (Cereal)	0,020	0,020	71	15	2	1
	LC	Water containing	0,010	0,750	85	14	73	3
Acetamiprid		Summary	0,010	0,100	97	13	102	3
	LC	Water containing	0,010	0,100	97	13	83	3
	LC	Sugar containing	0,020	0,020	63	3	2	1
	LC	Dry (Cereal)	0,020	0,020	102	7	2	1
	LC	Acidic	0,010	0,100	98	6	14	2
Aclonifen		Summary	0,010	0,200	90	20	95	5
	GC	Water containing	0,050	0,100	100	19	28	4
	GC	Acidic	0,100	0,100	95	15	6	2
	GC	Sugar containing	0,100	0,100	106		1	1
	LC	Sugar containing	0,010	0,100	74	9	12	1
	LC	Acidic	0,010	0,100	94	18	12	1
	LC	Water containing	0,010	0,100	86	16	24	1
	LC	Dry (Cereal)	0,020	0,200	87	26	12	1
Acrinathrin		Summary	0,010	1,000	92	21	150	6
	GC	Dry	0,100	0,100	96		1	1
	GC	Sugar containing	0,100	0,100	105	5	2	1
	GC	Acidic	0,010	0,100	92	7	10	1
	GC	Water containing	0,010	1,000	99	18	83	5
	LC	Sugar containing	0,100	0,100	92	2	6	1
	LC	Dry (Cereal)	0,020	0,200	83	17	12	1
	LC	Water containing	0,010	0,100	80	27	24	1
	LC	Acidic	0,010	0,100	70	17	12	1
Aldicarb		Summary	0,010	0,200	89	27	150	4
	LC	Water containing	0,010	0,100	85	23	95	4
	LC	Sugar containing	0,010	0,100	93	13	14	2
	LC	Dry (Cereal)	0,020	0,200	118	43	14	2
	LC	Acidic	0,010	0,100	86	24	26	3
Aldicarb-Sulfon		Summary	0,010	0,200	85	23	97	3
	LC	Sugar containing	0,010	0,100	94	11	12	1
	LC	Acidic	0,010	0,100	95	11	23	2
	LC	Dry (Cereal)	0,020	0,200	49	29	12	1
	LC	Water containing	0,010	0,100	88	20	50	3
Aldicarb-Sulfoxid		Summary	0,010	0,200	81	15	97	3
	LC	Dry (Cereal)	0,020	0,200	73	7	12	1
	LC	Sugar containing	0,010	0,100	83	10	12	1
	LC	Water containing	0,010	0,100	85	16	50	3
	LC	Acidic	0,010	0,100	73	13	23	2
Aldrin		Summary	0,032	1,000	98	13	53	4
	GC	Water containing	0,032	1,000	98	13	53	4
Azinphos-Ethyl		Summary	0,010	0,200	96	11	69	4
	GC	Acidic	0,050	0,050	98		1	1
	GC	Water containing	0,050	0,111	104	16	20	3
	LC	Water containing	0,010	0,100	92	6	24	1
	LC	Dry (Cereal)	0,020	0,200	94	8	12	1
	LC	Sugar containing	0,010	0,100	93	8	12	1
Azinphos-Methyl		Summary	0,010	0,200	95	18	92	4
	GC	Dry	0,100	0,100	74		1	1
	GC	Water containing	0,010	0,200	101	26	30	3
	GC	Acidic	0,010	0,200	92	15	13	2
	LC	Water containing	0,010	0,100	87	6	24	1
	LC	Sugar containing	0,010	0,100	94	7	12	1
	LC	Dry (Cereal)	0,020	0,200	102	4	12	1

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
Azoxystrobin		Summary	0,010	2,500	93	13	203	6
	GC	Acidic	0,010	0,100	100	10	10	1
	GC	Water containing	0,010	1,000	96	11	50	4
	LC	Sugar containing	0,020	0,020	78	4	2	1
	LC	Dry (Cereal)	0,020	0,020	103	13	2	1
	LC	Other	0,010	2,500	82	8	24	1
	LC	Acidic	0,010	0,100	99	8	14	2
	LC	Water containing	0,010	0,100	95	14	76	2
	LC	Dry	0,010	2,500	80	9	24	1
Benalaxyl		Summary	0,010	2,500	94	10	197	6
	GC	Water containing	0,050	1,000	96	9	51	4
	GC	Acidic	0,100	0,500	99	11	7	2
	LC	Dry (Cereal)	0,020	0,200	103	5	12	1
	LC	Sugar containing	0,010	0,100	96	4	12	1
	LC	Other	0,010	2,500	86	7	24	1
	LC	Water containing	0,010	0,100	95	6	44	2
	LC	Dry	0,010	2,500	81	8	24	1
	LC	Acidic	0,010	0,100	102	6	23	2
Bifenox		Summary	0,010	0,200	85	24	85	5
	GC	Water containing	0,051	0,200	96	19	26	4
	GC	Sugar containing	0,200	0,200	87		1	1
	LC	Water containing	0,010	0,100	78	32	24	1
	LC	Acidic	0,010	0,100	88	19	12	1
	LC	Dry (Cereal)	0,020	0,200	80	15	12	1
	LC	Sugar containing	0,010	0,100	76	25	9	1
Bifenthrin		Summary	0,010	1,000	102	11	117	6
	GC	Sugar containing	0,100	0,100	93	17	2	1
	GC	Dry	0,100	0,100	98	3	2	1
	GC	Acidic	0,010	0,100	105	18	17	3
	GC	Water containing	0,010	1,000	102	10	96	6
Binapacryl		Summary	0,025	0,168	91	6	14	3
	GC	Water containing	0,100	0,168	91	7	12	2
	LC	Water containing	0,025	0,025	89	1	2	1
Bitertanol		Summary	0,010	2,500	94	15	243	6
	GC	Water containing	0,010	1,000	100	16	69	4
	GC	Dry	0,100	0,100	90	5	2	1
	GC	Acidic	0,010	0,100	102	13	10	1
	LC	Acidic	0,010	0,100	86	9	15	2
	LC	Dry	0,010	2,500	86	10	24	1
	LC	Sugar containing	0,010	0,100	87	8	14	2
	LC	Other	0,010	2,500	79	9	24	1
	LC	Dry (Cereal)	0,020	0,200	94	10	14	2
	LC	Water containing	0,010	0,100	95	15	70	2
Boscalid		Summary	0,010	0,200	93	14	156	4
	GC	Water containing	0,101	0,101	66	12	7	1
	LC	Dry (Cereal)	0,020	0,200	96	8	14	2
	LC	Water containing	0,010	0,100	93	14	95	3
	LC	Sugar containing	0,010	0,100	96	8	14	2
	LC	Acidic	0,010	0,100	94	10	26	3
Bromophos		Summary	0,010	1,000	102	14	108	6
	GC	Water containing	0,010	1,000	102	12	87	6
	GC	Sugar containing	0,100	0,400	103	19	3	2
	GC	Dry	0,100	0,100	137	57	2	1
	GC	Acidic	0,010	0,400	95	7	16	3
Bromophos-Ethyl		Summary	0,010	0,100	101	10	62	5
	GC	Dry	0,100	0,100	90	25	2	1
	GC	Acidic	0,010	0,100	101	8	15	2
	GC	Water containing	0,010	0,100	101	10	45	5
Bromopropylate		Summary	0,010	1,000	98	26	94	6
	GC	Dry	0,100	0,100	90	11	2	1
	GC	Water containing	0,010	1,000	103	11	77	6
	GC	Acidic	0,010	0,100	72	53	15	2
Bromuconazole		Summary	0,010	0,500	96	10	101	5
	GC	Acidic	0,500	0,500	91		1	1

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	GC	Water containing	0,100	0,500	99	16	20	3
	GC	Sugar containing	0,500	0,500	112		1	1
	LC	Acidic	0,010	0,100	98	8	23	2
	LC	Sugar containing	0,010	0,100	95	5	12	1
	LC	Water containing	0,010	0,100	94	8	44	2
Bupirimate		Summary	0,010	1,000	97	10	162	7
	GC	Sugar containing	0,100	0,500	112	5	2	2
	GC	Water containing	0,010	1,000	100	12	77	6
	GC	Acidic	0,010	0,500	96	11	16	3
	GC	Dry	0,100	0,100	94		1	1
	LC	Water containing	0,010	0,100	90	6	30	2
	LC	Dry (Cereal)	0,020	0,200	98	5	12	1
	LC	Acidic	0,010	0,100	95	10	12	1
	LC	Sugar containing	0,010	0,100	96	5	12	1
Buprofezin		Summary	0,010	5,000	100	8	149	7
	GC	Acidic	0,100	5,000	102	14	6	2
	GC	Water containing	0,050	5,000	105	8	64	5
	LC	Sugar containing	0,010	0,100	94	1	12	1
	LC	Water containing	0,010	0,100	95	6	44	2
	LC	Acidic	0,010	0,100	100	6	11	1
	LC	Dry (Cereal)	0,020	0,200	99	5	12	1
Captafol		Summary	0,050	1,000	100	31	47	4
	GC	Water containing	0,050	1,000	101	31	45	4
	GC	Sugar containing	0,500	0,500	50		1	1
Captan		Summary	0,050	1,000	90	14	51	5
	GC	Water containing	0,050	1,000	90	14	51	5
Carbendazim		Summary	0,010	2,500	85	14	132	4
	LC	Water containing	0,010	0,100	91	12	76	3
	LC	Acidic	0,010	0,100	84	8	15	2
	LC	Sugar containing	0,020	0,020	69	13	2	1
	LC	Other	0,050	2,500	79	10	18	1
	LC	Dry (Cereal)	0,020	0,020	90	6	2	1
	LC	Dry	0,010	2,500	69	11	18	1
Carbofuran		Summary	0,010	0,100	100	11	94	3
	LC	Sugar containing	0,020	0,020	82	1	2	1
	LC	Water containing	0,010	0,100	99	11	75	3
	LC	Acidic	0,010	0,100	102	7	14	2
	LC	Dry (Cereal)	0,020	0,020	108	11	2	1
Carboxin		Summary	0,025	1,000	91	9	49	3
	GC	Acidic	0,100	0,100	91	5	5	1
	GC	Water containing	0,050	1,000	92	8	37	2
	LC	Water containing	0,025	0,025	89	12	7	1
Chinomethionat		Summary	0,050	1,000	75	21	58	4
	GC	Sugar containing	0,100	0,100	58		1	1
	GC	Acidic	0,100	0,100	90	9	6	2
	GC	Water containing	0,050	1,000	74	22	51	4
Chlorfenapyr		Summary	0,010	0,200	91	18	83	4
	GC	Water containing	0,018	0,050	97	11	23	3
	LC	Acidic	0,010	0,100	76	15	12	1
	LC	Sugar containing	0,010	0,100	99	22	12	1
	LC	Dry (Cereal)	0,020	0,200	90	14	12	1
	LC	Water containing	0,010	0,100	89	21	24	1
Chlorfenson		Summary	0,050	1,000	100	9	54	3
	GC	Water containing	0,050	1,000	100	9	54	3
Chlorfenvinphos		Summary	0,010	1,000	100	11	144	6
	GC	Water containing	0,010	1,000	102	10	77	5
	GC	Dry	0,100	0,100	80		1	1
	GC	Sugar containing	0,050	0,050	148		1	1
	GC	Acidic	0,010	0,400	104	11	17	3
	LC	Sugar containing	0,010	0,100	93	6	12	1
	LC	Dry (Cereal)	0,020	0,200	100	5	12	1
	LC	Water containing	0,010	0,100	92	8	24	1
Chlorothalonil		Summary	0,010	1,000	76	39	71	5
	GC	Acidic	0,100	0,100	96	3	5	1

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	GC	Sugar containing	0,100	0,100	29	4	2	1
	GC	Water containing	0,010	1,000	76	40	63	5
	GC	Dry	0,100	0,100	50		1	1
Chlorpropham		Summary	0,010	1,000	96	13	149	7
	GC	Dry	0,100	0,100	91	4	2	1
	GC	Acidic	0,010	0,100	97	8	17	3
	GC	Water containing	0,010	1,000	100	14	70	5
	LC	Dry (Cereal)	0,020	0,200	107	9	12	1
	LC	Acidic	0,010	0,100	90	8	12	1
	LC	Sugar containing	0,100	0,100	82	4	6	1
	LC	Water containing	0,010	0,100	87	9	30	2
Chlorpyrifos		Summary	0,010	1,000	102	10	104	6
	GC	Sugar containing	0,050	0,050	123		1	1
	GC	Dry	0,100	0,100	106	31	2	1
	GC	Water containing	0,010	1,000	101	10	85	6
	GC	Acidic	0,010	0,100	102	7	16	3
Chlorpyrifos-Methyl		Summary	0,010	1,000	102	12	102	6
	GC	Acidic	0,010	0,100	102	8	15	2
	GC	Water containing	0,010	1,000	102	11	85	6
	GC	Dry	0,100	0,100	122	53	2	1
Chlozolinate		Summary	0,010	0,100	101	15	78	5
	GC	Water containing	0,010	0,100	100	9	57	5
	GC	Acidic	0,010	0,100	98	12	17	3
	GC	Sugar containing	0,100	0,100	91	24	2	1
	GC	Dry	0,100	0,100	149	72	2	1
Clothianidin		Summary	0,020	0,050	96	21	60	3
	LC	Acidic	0,020	0,020	91	12	3	1
	LC	Water containing	0,020	0,050	97	21	52	3
	LC	Sugar containing	0,020	0,020	78	37	2	1
	LC	Dry (Cereal)	0,020	0,020	102	6	2	1
Cyanofenphos		Summary	0,050	1,000	102	6	45	3
	GC	Water containing	0,050	1,000	102	6	45	3
Cyazofamid		Summary	0,010	0,200	92	13	77	3
	LC	Water containing	0,010	0,100	90	17	36	3
	LC	Sugar containing	0,010	0,100	90	3	12	1
	LC	Acidic	0,010	0,100	98	10	17	2
	LC	Dry (Cereal)	0,020	0,200	93	3	12	1
Cycloxydim		Summary	0,010	0,200	73	30	111	3
	LC	Dry (Cereal)	0,020	0,200	80	17	14	2
	LC	Sugar containing	0,010	0,100	29	22	14	2
	LC	Water containing	0,010	0,100	79	27	78	3
	LC	Acidic	0,020	0,020	89	22	4	1
Cyfluthrin (incl. beta-)		Summary	0,010	0,500	97	13	62	4
	GC	Water containing	0,010	0,500	97	11	47	4
	GC	Sugar containing	0,050	0,500	118	13	3	2
	GC	Dry	0,100	0,100	62		1	1
	GC	Acidic	0,010	0,500	96	15	11	2
Cyhalothrin, lambda-		Summary	0,010	0,200	106	12	61	5
	GC	Water containing	0,010	0,200	106	11	47	5
	GC	Dry	0,100	0,100	126		1	1
	GC	Acidic	0,010	0,100	105	14	10	1
	GC	Sugar containing	0,050	0,200	111	27	2	2
Cymoxanil		Summary	0,010	2,500	81	26	129	4
	GC	Water containing	0,500	0,500	75	14	7	1
	LC	Sugar containing	0,020	0,020	66	13	2	1
	LC	Acidic	0,010	0,100	95	11	15	2
	LC	Water containing	0,010	0,100	95	18	56	2
	LC	Dry (Cereal)	0,020	0,020	100		1	1
	LC	Other	0,010	2,500	58	27	24	1
	LC	Dry	0,010	2,500	66	22	24	1
Cypermethrin		Summary	0,010	1,000	101	16	79	4
	GC	Dry	0,100	0,100	130		1	1
	GC	Acidic	0,010	1,000	100	10	11	2
	GC	Water containing	0,010	1,000	100	16	65	4

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
Cyproconazole	GC	Sugar containing	0,050	1,000	112	29	2	2
		Summary	0,010	1,229	95	11	137	4
	GC	Water containing	1,229	1,229	76	9	6	1
	LC	Acidic	0,010	0,100	98	12	14	2
	LC	Sugar containing	0,010	0,100	96	7	14	2
Cyprodinil	LC	Dry (Cereal)	0,020	0,200	99	7	14	2
	LC	Water containing	0,010	0,100	95	11	88	3
		Summary	0,010	1,000	97	12	187	5
	GC	Water containing	0,010	1,000	101	8	71	4
	GC	Acidic	0,010	0,100	100	8	15	2
	GC	Dry	0,100	0,100	84		1	1
	GC	Sugar containing	0,050	0,050	112		1	1
	LC	Dry (Cereal)	0,020	0,020	110	11	2	1
	LC	Acidic	0,010	0,100	99	10	15	2
	LC	Water containing	0,010	0,100	93	14	78	2
DDD, o,p-	LC	Sugar containing	0,020	0,050	87	5	3	2
		Summary	0,050	1,000	101	9	57	4
	GC	Water containing	0,050	1,000	101	10	52	4
	GC	Acidic	0,100	0,100	100	2	5	1
		Summary	0,050	1,000	97	11	47	3
DDE, o,p-	GC	Water containing	0,050	1,000	97	11	47	3
		Summary	0,050	1,000	95	16	57	4
DDE, p,p-	GC	Water containing	0,050	1,000	95	17	52	4
	GC	Acidic	0,100	0,100	99	3	5	1
		Summary	0,010	1,000	98	15	72	3
DDT, o,p-	GC	Water containing	0,010	1,000	97	16	60	3
	GC	Acidic	0,010	0,100	103	12	10	1
	GC	Dry	0,100	0,100	78		1	1
	GC	Sugar containing	0,100	0,100	108		1	1
		Summary	0,050	1,000	98	11	47	3
DDT, p,p-	GC	Water containing	0,050	1,000	98	11	47	3
		Summary	0,050	1,000	98	13	57	4
Deltamethrin	GC	Acidic	0,100	0,100	98	4	5	1
	GC	Water containing	0,050	1,000	98	14	52	4
		Summary	0,010	0,500	105	17	52	4
	GC	Sugar containing	0,050	0,050	98		1	1
Demeton-S-Methyl	GC	Acidic	0,010	0,100	110	28	10	1
	GC	Water containing	0,010	0,500	104	14	41	4
		Summary	0,010	0,176	91	15	92	3
	GC	Water containing	0,176	0,176	69	12	7	1
	LC	Dry (Cereal)	0,020	0,020	114	18	2	1
Diazinon	LC	Sugar containing	0,020	0,020	69	4	2	1
	LC	Water containing	0,010	0,100	92	13	66	2
	LC	Acidic	0,010	0,100	100	10	14	2
		Summary	0,010	1,000	98	9	161	7
	GC	Acidic	0,010	1,000	97	11	17	3
	GC	Sugar containing	0,100	0,100	94	16	2	1
	GC	Dry	0,100	0,100	110	14	2	1
	GC	Water containing	0,010	1,000	101	9	92	6
Dichlobenil	LC	Water containing	0,010	0,100	94	4	24	1
	LC	Sugar containing	0,010	0,100	89	3	12	1
	LC	Dry (Cereal)	0,020	0,200	99	3	12	1
		Summary	0,050	1,000	97	11	46	3
	GC	Water containing	0,050	1,000	97	11	46	3
		Summary	0,046	1,000	98	10	46	3
	GC	Water containing	0,046	1,000	98	10	46	3
Dichlofluanid		Summary	0,010	1,000	70	35	121	6
	GC	Water containing	0,050	1,000	90	23	67	5
	GC	Sugar containing	0,050	0,100	106	20	2	2
	LC	Sugar containing	0,010	0,100	11	9	12	1
	LC	Water containing	0,010	0,100	54	24	26	2
	LC	Acidic	0,025	0,025	109		1	1
	LC	Dry (Cereal)	0,020	0,200	32	6	12	1
Dichlorvos		Summary	0,050	1,000	103	12	57	4

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	GC	Acidic	0,050	0,100	111	3	3	2
	GC	Water containing	0,050	1,000	103	12	53	4
	GC	Sugar containing	0,050	0,050	86		1	1
Dicloran		Summary	0,010	0,250	102	17	67	4
	GC	Dry	0,100	0,100	80		1	1
	GC	Water containing	0,010	0,250	107	17	43	4
	GC	Sugar containing	0,050	0,050	96		1	1
	GC	Acidic	0,010	0,250	97	11	16	3
	LC	Acidic	0,025	0,100	82	11	6	1
Dicofol		Summary	0,010	0,127	95	21	45	3
	GC	Sugar containing	0,050	0,100	102	19	2	1
	GC	Dry	0,100	0,100	88		1	1
	GC	Acidic	0,010	0,100	82	13	10	1
	GC	Water containing	0,010	0,127	99	22	32	3
Dicrotophos		Summary	0,010	0,129	89	10	91	3
	GC	Water containing	0,129	0,129	86	6	6	1
	LC	Sugar containing	0,020	0,020	70	4	2	1
	LC	Dry (Cereal)	0,020	0,020	94	0	2	1
	LC	Acidic	0,010	0,100	91	6	13	2
	LC	Water containing	0,010	0,100	90	11	68	2
Dieldrin		Summary	0,010	1,000	104	16	85	3
	GC	Acidic	0,010	0,100	112	27	15	2
	GC	Sugar containing	0,100	0,100	108	3	2	1
	GC	Dry	0,100	0,100	88		1	1
	GC	Water containing	0,010	1,000	102	12	67	3
Diethofencarb		Summary	0,010	1,000	97	8	131	5
	GC	Acidic	0,100	0,500	106	8	7	2
	GC	Water containing	0,050	1,000	100	7	45	3
	LC	Water containing	0,010	0,100	94	8	44	2
	LC	Acidic	0,010	0,100	100	4	11	1
	LC	Dry (Cereal)	0,020	0,200	95	3	12	1
	LC	Sugar containing	0,010	0,100	93	4	12	1
Difenoconazole		Summary	0,010	1,000	100	11	125	4
	GC	Water containing	0,050	1,000	103	10	38	2
	LC	Sugar containing	0,020	0,020	81	4	2	1
	LC	Dry (Cereal)	0,020	0,020	97	21	2	1
	LC	Acidic	0,010	0,100	103	6	13	2
	LC	Water containing	0,010	0,100	97	12	69	2
Diflubenzuron		Summary	0,010	2,500	91	15	181	3
	LC	Dry (Cereal)	0,020	0,200	89	8	14	2
	LC	Other	0,010	2,500	73	8	24	1
	LC	Acidic	0,010	0,100	98	7	27	3
	LC	Dry	0,050	2,500	77	10	18	1
	LC	Sugar containing	0,020	0,100	92	6	8	2
	LC	Water containing	0,010	0,100	97	14	89	3
Diflufenican		Summary	0,010	0,100	95	16	62	3
	GC	Water containing	0,100	0,100	111	3	7	1
	LC	Acidic	0,010	0,100	113	13	11	1
	LC	Dry	0,010	0,100	82	6	12	1
	LC	Water containing	0,010	0,100	98	11	20	1
	LC	Other	0,010	0,100	80	7	12	1
Dimethoate		Summary	0,010	1,000	94	11	133	5
	GC	Water containing	0,064	1,000	90	11	37	3
	LC	Dry (Cereal)	0,020	0,020	89	1	2	1
	LC	Acidic	0,010	0,100	98	11	14	2
	LC	Water containing	0,010	0,100	96	11	77	2
	LC	Sugar containing	0,020	0,020	78	8	2	1
Dimethomorph		Summary	0,010	1,000	96	13	173	5
	GC	Water containing	0,010	1,000	95	10	67	4
	GC	Dry	0,100	0,100	128		1	1
	GC	Acidic	0,010	0,100	85	5	10	1
	GC	Sugar containing	0,100	0,100	105		1	1
	LC	Dry (Cereal)	0,020	0,020	103	7	2	1
	LC	Sugar containing	0,020	0,020	76	21	2	1

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	LC	Acidic	0,010	0,100	95	8	13	2
	LC	Water containing	0,010	0,100	97	14	76	2
Diniconazole		Summary	0,010	0,250	96	9	114	4
	GC	Acidic	0,250	0,250	100		1	1
	GC	Sugar containing	0,250	0,250	114		1	1
	GC	Water containing	0,025	0,250	96	17	14	3
	LC	Acidic	0,010	0,100	96	7	22	2
	LC	Water containing	0,010	0,100	95	9	52	2
	LC	Sugar containing	0,010	0,100	94	6	12	1
	LC	Dry (Cereal)	0,020	0,200	102	3	12	1
Dioxathion		Summary	0,010	0,400	102	16	66	4
	GC	Dry	0,100	0,100	105	16	2	1
	GC	Acidic	0,050	0,400	93	12	7	2
	GC	Water containing	0,010	0,400	104	17	53	4
	GC	Sugar containing	0,050	0,400	93	11	4	2
Diphenylamine		Summary	0,010	0,500	95	13	85	4
	GC	Acidic	0,500	0,500	116	1	2	1
	GC	Water containing	0,025	0,500	98	21	23	3
	GC	Sugar containing	0,050	0,050	84		1	1
	LC	Water containing	0,010	0,100	91	10	33	2
	LC	Sugar containing	0,010	0,100	98	4	13	2
	LC	Dry (Cereal)	0,020	0,200	97	5	12	1
	LC	Acidic	0,050	0,050	85		1	1
Disulfoton		Summary	0,050	1,000	99	9	51	3
	GC	Acidic	0,050	0,100	99	11	3	2
	GC	Water containing	0,050	1,000	99	9	48	3
Ditalimfos		Summary	0,025	1,000	83	19	55	4
	GC	Sugar containing	0,050	0,050	150		1	1
	GC	Water containing	0,050	1,000	86	10	47	3
	LC	Water containing	0,025	0,025	44	12	6	1
Endosulfan, alpha-		Summary	0,010	1,000	96	16	105	6
	GC	Sugar containing	0,100	0,100	96	17	2	1
	GC	Dry	0,100	0,100	96		1	1
	GC	Acidic	0,010	0,100	96	10	10	1
	GC	Water containing	0,010	1,000	96	17	92	6
Endosulfan, beta-		Summary	0,010	1,000	99	15	105	5
	GC	Dry	0,100	0,100	98		1	1
	GC	Acidic	0,010	0,100	96	10	10	1
	GC	Sugar containing	0,100	0,100	106	5	2	1
	GC	Water containing	0,010	1,000	100	16	92	5
Endosulfansulfate		Summary	0,010	1,000	95	16	97	5
	GC	Sugar containing	0,100	0,100	111	4	2	1
	GC	Acidic	0,010	0,100	94	10	11	2
	GC	Dry	0,100	0,100	98		1	1
	GC	Water containing	0,010	1,000	95	16	83	5
EPN		Summary	0,010	0,200	83	23	79	3
	GC	Acidic	0,050	0,100	102	5	6	2
	GC	Water containing	0,050	0,100	106	14	13	2
	LC	Dry (Cereal)	0,020	0,200	86	9	12	1
	LC	Water containing	0,010	0,100	74	21	24	1
	LC	Sugar containing	0,010	0,100	68	17	12	1
	LC	Acidic	0,010	0,100	82	30	12	1
Epoxiconazole		Summary	0,010	0,400	93	11	88	4
	GC	Acidic	0,400	0,400	118		1	1
	GC	Water containing	0,025	0,400	90	24	12	3
	LC	Sugar containing	0,010	0,100	91	3	12	1
	LC	Acidic	0,010	0,100	98	6	10	1
	LC	Dry (Cereal)	0,020	0,200	94	4	12	1
	LC	Water containing	0,010	0,100	93	7	41	2
Ethiofencarb		Summary	0,010	0,200	88	18	146	3
	GC	Water containing	0,025	0,025	70		1	1
	LC	Acidic	0,010	0,100	84	24	25	3
	LC	Water containing	0,010	0,100	88	19	91	3
	LC	Dry (Cereal)	0,020	0,200	93	4	14	2

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	LC	Sugar containing	0,010	0,100	91	7	14	2
Ethion		Summary	0,010	1,000	101	9	123	6
	GC	Sugar containing	0,100	0,100	111		1	1
	GC	Water containing	0,010	1,000	102	9	76	6
	GC	Dry	0,100	0,100	94		1	1
	GC	Acidic	0,010	0,500	100	12	16	3
	LC	Acidic	0,010	0,100	99	4	10	1
	LC	Water containing	0,010	0,100	102	8	19	1
Ethofumesate		Summary	0,010	1,000	94	12	122	3
	GC	Water containing	0,025	1,000	97	7	33	2
	LC	Water containing	0,010	0,100	92	13	43	2
	LC	Sugar containing	0,010	0,100	90	7	12	1
	LC	Dry (Cereal)	0,020	0,200	98	13	12	1
	LC	Acidic	0,010	0,100	94	16	22	2
Ethoprophos		Summary	0,010	1,000	99	10	126	6
	GC	Acidic	0,010	0,100	96	10	16	3
	GC	Dry	0,100	0,100	100		1	1
	GC	Sugar containing	0,050	0,100	97	7	2	2
	GC	Water containing	0,010	1,000	100	10	78	6
	LC	Water containing	0,010	0,100	99	8	19	1
	LC	Acidic	0,010	0,100	92	6	10	1
Etofenprox		Summary	0,010	1,000	97	11	164	6
	GC	Dry	0,100	0,100	98		1	1
	GC	Acidic	0,010	0,100	96	10	16	3
	GC	Water containing	0,010	1,000	97	11	78	5
	GC	Sugar containing	0,050	0,100	105	5	2	2
	LC	Water containing	0,010	0,100	94	12	30	2
	LC	Sugar containing	0,010	0,100	102	7	12	1
	LC	Dry (Cereal)	0,020	0,200	99	14	12	1
	LC	Acidic	0,010	0,100	102	13	13	2
Etridiazole		Summary	0,010	0,100	107	26	64	3
	GC	Dry	0,100	0,100	163	91	2	1
	GC	Water containing	0,010	0,100	108	24	43	3
	GC	Acidic	0,010	0,100	101	8	17	3
	GC	Sugar containing	0,100	0,100	81	28	2	1
Etrimfos		Summary	0,033	1,000	101	15	57	4
	GC	Acidic	0,050	0,050	110	11	2	2
	GC	Water containing	0,033	1,000	101	15	53	4
	GC	Sugar containing	0,050	0,050	95	13	2	2
Famoxadone		Summary	0,010	0,750	98	15	52	3
	GC	Acidic	0,750	0,750	137		1	1
	GC	Water containing	0,025	0,750	101	21	13	3
	LC	Water containing	0,010	0,100	94	11	28	1
	LC	Acidic	0,010	0,100	99	6	10	1
Fenamiphos		Summary	0,010	1,000	97	12	91	5
	GC	Acidic	0,050	0,050	106		1	1
	GC	Sugar containing	0,050	0,050	103		1	1
	GC	Water containing	0,025	1,000	97	14	61	5
	LC	Acidic	0,010	0,100	96	4	10	1
	LC	Water containing	0,010	0,100	98	11	18	1
Fenarimol		Summary	0,010	1,000	95	11	172	6
	GC	Sugar containing	0,100	0,100	105		1	1
	GC	Acidic	0,010	0,250	96	10	11	2
	GC	Dry	0,100	0,100	98		1	1
	GC	Water containing	0,010	1,000	96	15	73	5
	LC	Sugar containing	0,010	0,100	93	5	12	1
	LC	Dry (Cereal)	0,020	0,200	97	4	12	1
	LC	Acidic	0,010	0,100	97	6	10	1
	LC	Water containing	0,010	0,100	94	9	52	2
Fenazaquin		Summary	0,010	2,500	90	16	207	6
	GC	Dry	0,100	0,100	91	4	2	1
	GC	Water containing	0,010	1,000	102	11	63	4
	GC	Acidic	0,010	0,500	92	18	17	3
	LC	Dry (Cereal)	0,020	0,200	92	3	12	1

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	LC	Sugar containing	0,010	0,100	88	4	12	1
	LC	Other	0,050	2,500	73	14	18	1
	LC	Water containing	0,010	0,150	87	14	52	3
	LC	Acidic	0,010	0,100	97	15	13	1
	LC	Dry	0,050	2,500	70	12	18	1
Fenbuconazole		Summary	0,010	0,200	93	10	100	3
	GC	Water containing	0,025	0,200	102	10	9	2
	GC	Acidic	0,200	0,200	120	18	2	1
	LC	Sugar containing	0,010	0,100	87	6	12	1
	LC	Water containing	0,010	0,100	93	11	43	2
	LC	Dry (Cereal)	0,020	0,200	87	4	12	1
	LC	Acidic	0,010	0,100	94	7	22	2
Fenchlorphos		Summary	0,050	1,000	101	11	55	3
	GC	Acidic	0,050	0,050	114		1	1
	GC	Water containing	0,050	1,000	101	10	53	3
	GC	Sugar containing	0,050	0,050	90		1	1
Fenhexamid		Summary	0,010	1,000	91	17	137	5
	GC	Water containing	0,025	1,000	91	13	46	4
	LC	Water containing	0,010	0,100	93	19	73	2
	LC	Acidic	0,010	0,100	90	13	13	2
	LC	Dry (Cereal)	0,020	0,020	68	32	2	1
	LC	Sugar containing	0,020	0,020	81	9	2	1
Fenitrothion		Summary	0,010	1,000	98	18	172	7
	GC	Water containing	0,010	1,000	99	15	76	6
	GC	Sugar containing	0,050	0,500	105	26	2	2
	GC	Acidic	0,010	0,500	103	11	17	3
	GC	Dry	0,100	0,100	98		1	1
	LC	Dry (Cereal)	0,020	0,200	89	27	12	1
	LC	Acidic	0,010	0,100	92	22	18	2
	LC	Water containing	0,010	0,100	92	18	34	2
	LC	Sugar containing	0,010	0,100	110	22	12	1
Fenoxycarb		Summary	0,010	2,500	93	15	235	5
	GC	Dry	0,100	0,100	90		1	1
	GC	Water containing	0,010	1,000	98	14	51	3
	GC	Acidic	0,100	0,100	90	2	5	1
	LC	Sugar containing	0,010	0,100	94	7	14	2
	LC	Other	0,010	2,500	71	12	24	1
	LC	Acidic	0,010	0,100	98	10	13	2
	LC	Dry	0,010	2,500	78	12	21	1
	LC	Water containing	0,010	0,100	98	10	91	3
	LC	Dry (Cereal)	0,020	0,200	99	6	14	2
Fenpropathrin		Summary	0,010	1,000	95	14	156	7
	GC	Acidic	0,100	0,400	81	6	6	2
	GC	Water containing	0,010	1,000	100	12	86	6
	GC	Dry	0,100	0,100	115	24	2	1
	GC	Sugar containing	0,100	0,100	93	21	2	1
	LC	Dry (Cereal)	0,020	0,200	90	10	12	1
	LC	Acidic	0,010	0,100	91	19	12	1
	LC	Sugar containing	0,010	0,100	93	5	12	1
	LC	Water containing	0,010	0,100	84	9	24	1
Fenpropimorph		Summary	0,010	2,500	95	13	197	4
	GC	Water containing	0,100	0,100	102	11	7	1
	LC	Water containing	0,010	0,100	100	11	86	3
	LC	Other	0,010	2,500	88	10	24	1
	LC	Acidic	0,010	0,100	100	15	27	3
	LC	Dry (Cereal)	0,020	0,200	100	9	14	2
	LC	Sugar containing	0,010	0,100	93	9	14	2
	LC	Dry	0,010	2,500	76	5	24	1
Fenpyroximate		Summary	0,010	0,200	93	14	149	4
	GC	Water containing	0,025	0,100	82	9	7	2
	LC	Acidic	0,010	0,100	95	23	24	3
	LC	Sugar containing	0,010	0,100	85	9	14	2
	LC	Water containing	0,010	0,100	95	12	89	3
	LC	Dry (Cereal)	0,020	0,200	88	13	14	2

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
Fenson		Summary	0,010	0,100	105	18	47	3
	GC	Acidic	0,010	0,100	110	20	15	2
	GC	Water containing	0,010	0,100	103	17	30	3
	GC	Dry	0,100	0,100	99	13	2	1
Fenthion		Summary	0,010	1,000	96	13	157	6
	GC	Dry	0,100	0,100	88		1	1
	GC	Acidic	0,010	0,500	102	9	12	2
	GC	Water containing	0,010	1,000	103	9	66	5
	LC	Acidic	0,010	0,100	92	7	10	1
	LC	Sugar containing	0,010	0,100	91	4	12	1
	LC	Water containing	0,010	0,100	87	17	44	2
	LC	Dry (Cereal)	0,020	0,200	98	4	12	1
Fenthion-Sulfon		Summary	0,010	0,200	99	11	68	3
	GC	Sugar containing	0,200	0,200	103		1	1
	GC	Acidic	0,010	0,200	95	8	11	2
	GC	Water containing	0,010	0,200	98	13	27	3
	LC	Water containing	0,010	0,100	102	12	19	1
	LC	Acidic	0,010	0,100	98	4	10	1
Fenthion-Sulfoxide		Summary	0,010	0,100	96	16	74	3
	GC	Acidic	0,010	0,100	94	10	12	2
	GC	Dry	0,100	0,100	100		1	1
	GC	Sugar containing	0,100	0,100	125		1	1
	GC	Water containing	0,010	0,100	91	21	31	3
	LC	Water containing	0,010	0,100	101	11	19	1
	LC	Acidic	0,010	0,100	100	5	10	1
Fenvalerate/Esfenvalerate (sum)		Summary	0,010	1,000	102	9	82	4
	GC	Sugar containing	0,050	0,050	98		1	1
	GC	Dry	0,100	0,100	86		1	1
	GC	Acidic	0,010	0,100	101	11	10	1
	GC	Water containing	0,010	1,000	102	9	70	4
Flucythrinate		Summary	0,010	0,250	100	14	61	4
	GC	Water containing	0,010	0,250	100	14	46	4
	GC	Dry	0,100	0,100	130		1	1
	GC	Acidic	0,010	0,100	98	11	10	1
	GC	Sugar containing	0,050	0,250	106	13	3	2
Fludioxonil		Summary	0,010	1,000	99	12	159	5
	GC	Sugar containing	0,050	0,050	104		1	1
	GC	Acidic	0,010	0,100	101	12	10	1
	GC	Water containing	0,010	1,000	98	11	70	4
	GC	Dry	0,100	0,100	86		1	1
	LC	Sugar containing	0,020	0,020	80	4	2	1
	LC	Acidic	0,010	0,100	103	15	15	2
	LC	Dry (Cereal)	0,020	0,020	88	14	2	1
	LC	Water containing	0,010	0,100	99	13	57	2
Flufenoxuron		Summary	0,010	0,200	94	15	159	4
	GC	Water containing	0,025	0,025	94		1	1
	LC	Sugar containing	0,010	0,100	87	9	14	2
	LC	Dry (Cereal)	0,020	0,200	87	8	14	2
	LC	Water containing	0,010	0,100	96	17	103	4
	LC	Acidic	0,010	0,100	95	10	26	3
Fluquinconazole		Summary	0,010	0,250	92	13	105	5
	GC	Acidic	0,250	0,250	112		1	1
	GC	Water containing	0,051	0,250	96	19	19	3
	LC	Sugar containing	0,010	0,100	95	7	12	1
	LC	Acidic	0,010	0,100	89	13	12	1
	LC	Water containing	0,010	0,100	90	13	49	2
	LC	Dry (Cereal)	0,020	0,200	92	3	12	1
Flusilazole		Summary	0,010	1,260	98	11	249	6
	GC	Dry	0,100	0,100	101	6	2	1
	GC	Acidic	0,010	0,100	96	10	10	1
	GC	Water containing	0,010	1,260	101	9	77	4
	GC	Sugar containing	0,100	0,100	94	17	2	1
	LC	Water containing	0,010	0,100	97	13	103	3
	LC	Acidic	0,010	0,100	99	7	26	3

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	LC	Dry (Cereal)	0,020	0,200	98	5	14	2
	LC	Sugar containing	0,010	0,100	96	11	14	2
Fluvalinate		Summary	0,050	1,000	106	9	46	3
	GC	Water containing	0,050	1,000	105	8	45	3
	GC	Acidic	0,500	0,500	136		1	1
Folpet		Summary	0,050	0,500	86	29	28	3
	GC	Water containing	0,050	0,500	86	18	20	3
	GC	Dry	0,100	0,100	147	78	2	1
	GC	Acidic	0,100	0,500	67	14	6	2
Fonofos		Summary	0,010	1,000	97	11	115	5
	GC	Water containing	0,032	1,000	101	10	53	4
	GC	Acidic	0,500	0,500	115	9	2	1
	LC	Dry (Cereal)	0,020	0,200	98	6	12	1
	LC	Acidic	0,010	0,100	100	8	12	1
	LC	Water containing	0,010	0,100	85	6	24	1
	LC	Sugar containing	0,010	0,100	94	6	12	1
HCH, alpha-		Summary	0,031	1,000	94	14	54	4
	GC	Water containing	0,031	1,000	94	14	54	4
HCH, beta-		Summary	0,029	1,000	92	12	54	4
	GC	Water containing	0,029	1,000	92	12	54	4
HCH, gamma-		Summary	0,010	0,100	98	16	47	3
	GC	Acidic	0,010	0,100	101	10	10	1
	GC	Dry	0,100	0,100	73	7	2	1
	GC	Water containing	0,010	0,100	99	16	35	3
Heptachlor		Summary	0,031	0,100	94	15	21	3
	GC	Water containing	0,031	0,100	94	15	21	3
Heptenophos		Summary	0,010	1,000	94	10	168	6
	GC	Sugar containing	0,050	0,050	98		1	1
	GC	Water containing	0,049	1,000	96	11	51	4
	GC	Acidic	0,050	0,050	114		1	1
	LC	Water containing	0,010	0,100	95	9	71	2
	LC	Sugar containing	0,010	0,100	86	6	14	2
	LC	Dry (Cereal)	0,020	0,200	91	4	14	2
	LC	Acidic	0,010	0,100	89	15	15	2
Hexachlorobenzene		Summary	0,028	1,000	91	15	54	4
	GC	Water containing	0,028	1,000	91	15	54	4
Hexaconazole		Summary	0,010	1,000	93	10	154	6
	GC	Water containing	0,050	1,000	97	13	53	4
	GC	Sugar containing	0,250	0,250	115		1	1
	GC	Acidic	0,250	0,250	92		1	1
	LC	Dry (Cereal)	0,020	0,200	90	4	12	1
	LC	Acidic	0,010	0,100	89	7	23	2
	LC	Sugar containing	0,010	0,100	89	7	14	2
	LC	Water containing	0,010	0,100	93	8	50	2
Hexaflumuron		Summary	0,010	0,200	94	19	140	3
	LC	Sugar containing	0,020	0,100	95	11	10	3
	LC	Acidic	0,010	0,100	94	19	27	3
	LC	Dry (Cereal)	0,020	0,200	88	7	14	2
	LC	Water containing	0,010	0,100	95	21	88	3
Hexythiazox		Summary	0,010	1,110	93	15	100	3
	GC	Water containing	1,110	1,110	70	10	7	1
	LC	Dry (Cereal)	0,020	0,020	104	11	2	1
	LC	Acidic	0,010	0,100	98	14	14	2
	LC	Sugar containing	0,020	0,025	86	22	4	2
	LC	Water containing	0,010	0,100	95	13	72	2
Imazalil		Summary	0,010	2,500	95	20	190	7
	GC	Water containing	0,100	1,000	99	15	42	4
	GC	Acidic	0,100	0,100	88	10	5	1
	LC	Acidic	0,010	0,100	89	14	13	2
	LC	Sugar containing	0,020	0,025	87	11	4	2
	LC	Dry (Cereal)	0,020	0,020	91	9	2	1
	LC	Other	0,010	2,500	86	18	24	1
	LC	Dry	0,010	2,500	79	11	24	1
	LC	Water containing	0,010	0,100	102	22	75	2

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
Imidacloprid		Summary	0,010	0,100	99	18	99	3
	LC	Dry (Cereal)	0,020	0,020	81	25	2	1
	LC	Sugar containing	0,020	0,025	90	19	4	2
	LC	Water containing	0,010	0,100	102	18	78	3
	LC	Acidic	0,010	0,100	90	13	14	2
Indoxacarb		Summary	0,010	0,140	99	15	78	3
	GC	Water containing	0,140	0,140	102	11	6	1
	LC	Sugar containing	0,020	0,020	102	26	2	1
	LC	Dry (Cereal)	0,020	0,020	88	1	2	1
	LC	Acidic	0,010	0,020	99	16	8	2
	LC	Water containing	0,010	0,025	98	15	59	2
Iprodione		Summary	0,010	0,500	96	17	135	6
	GC	Acidic	0,100	0,500	104	13	7	2
	GC	Sugar containing	0,100	0,200	101	1	3	2
	GC	Water containing	0,010	0,500	99	18	64	5
	GC	Dry	0,100	0,100	98		1	1
	LC	Acidic	0,010	0,100	103	20	12	1
	LC	Sugar containing	0,010	0,100	81	15	12	1
	LC	Dry (Cereal)	0,020	0,200	80	10	12	1
	LC	Water containing	0,010	0,100	97	14	24	1
Iprovalicarb		Summary	0,010	0,200	96	11	143	3
	LC	Acidic	0,010	0,100	92	9	14	2
	LC	Sugar containing	0,010	0,100	99	9	16	3
	LC	Water containing	0,010	0,100	95	12	98	3
	LC	Dry (Cereal)	0,020	0,200	104	8	14	2
Isofenphos		Summary	0,050	1,000	103	16	47	3
	GC	Sugar containing	0,050	0,050	150		1	1
	GC	Acidic	0,050	0,050	120		1	1
	GC	Water containing	0,050	1,000	102	14	45	3
Kresoxim-Methyl		Summary	0,010	1,000	99	9	139	5
	GC	Dry	0,100	0,100	88	2	2	1
	GC	Water containing	0,010	1,000	100	9	77	5
	GC	Acidic	0,010	0,500	105	11	17	3
	LC	Water containing	0,010	0,100	96	6	29	1
	LC	Sugar containing	0,025	0,025	90	4	2	1
	LC	Acidic	0,010	0,100	92	9	12	1
Linuron		Summary	0,010	2,500	89	17	130	3
	LC	Dry (Cereal)	0,020	0,020	103	3	2	1
	LC	Other	0,010	2,500	82	13	24	1
	LC	Acidic	0,010	0,100	92	10	14	2
	LC	Dry	0,010	2,500	76	12	24	1
	LC	Sugar containing	0,020	0,025	90	16	4	2
	LC	Water containing	0,010	0,100	97	17	61	2
Lufenuron		Summary	0,010	0,200	98	17	156	4
	LC	Acidic	0,010	0,100	102	11	27	3
	LC	Dry (Cereal)	0,020	0,200	88	12	14	2
	LC	Sugar containing	0,020	0,100	98	12	10	3
	LC	Water containing	0,010	0,100	99	19	104	4
Malaoxon		Summary	0,010	1,000	99	18	129	4
	GC	Acidic	0,010	0,100	104	17	11	1
	GC	Water containing	0,010	1,000	102	21	61	3
	GC	Sugar containing	0,050	0,050	40		1	1
	GC	Dry	0,100	0,100	80		1	1
	LC	Acidic	0,020	0,020	100	16	3	1
	LC	Water containing	0,020	0,020	96	12	47	1
	LC	Dry (Cereal)	0,020	0,020	104	16	2	1
	LC	Sugar containing	0,020	0,020	85	16	2	1
Malathion		Summary	0,010	1,000	98	12	213	7
	GC	Sugar containing	0,050	0,100	120	12	2	1
	GC	Acidic	0,010	0,100	91	7	16	2
	GC	Water containing	0,010	1,000	101	13	93	5
	GC	Dry	0,100	0,100	92		1	1
	LC	Acidic	0,020	0,020	108	6	3	1
	LC	Dry (Cereal)	0,020	0,200	98	5	14	2

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	LC	Water containing	0,010	0,100	97	12	69	2
	LC	Sugar containing	0,010	0,100	89	8	14	2
Mecarbam		Summary	0,010	1,000	101	12	125	6
	GC	Sugar containing	0,100	1,000	117	2	2	2
	GC	Water containing	0,010	1,000	102	13	76	6
	GC	Dry	0,100	0,100	98		1	1
	GC	Acidic	0,010	1,000	100	9	16	3
	LC	Water containing	0,010	0,100	97	9	17	1
	LC	Acidic	0,010	0,100	93	10	11	1
	LC	Sugar containing	0,025	0,025	101	8	2	1
Mepanipyrim		Summary	0,010	0,200	96	8	118	4
	GC	Acidic	0,100	0,100	101	8	7	2
	GC	Water containing	0,050	0,100	100	6	21	2
	LC	Acidic	0,010	0,100	94	10	23	2
	LC	Water containing	0,010	0,100	95	8	41	2
	LC	Dry (Cereal)	0,020	0,200	104	2	12	1
	LC	Sugar containing	0,010	0,100	91	6	14	2
Mepronil		Summary	0,010	0,500	91	13	57	4
	GC	Acidic	0,100	0,500	88	22	7	2
	GC	Water containing	0,100	0,500	90	14	20	3
	LC	Acidic	0,010	0,100	90	6	11	1
	LC	Sugar containing	0,025	0,025	97	4	2	1
	LC	Water containing	0,010	0,100	94	10	17	1
Metalaxyl		Summary	0,010	1,000	101	17	144	4
	GC	Acidic	0,100	1,000	100	4	5	1
	GC	Water containing	0,050	1,000	101	9	45	2
	LC	Dry (Cereal)	0,020	0,020	102	1	2	1
	LC	Acidic	0,010	0,100	90	10	14	2
	LC	Sugar containing	0,020	0,025	88	10	4	2
	LC	Water containing	0,010	0,100	103	22	73	2
Metamitron		Summary	0,010	0,200	88	15	140	3
	LC	Dry (Cereal)	0,020	0,200	89	9	14	2
	LC	Acidic	0,010	0,100	80	19	26	3
	LC	Sugar containing	0,010	0,100	89	12	16	3
	LC	Water containing	0,010	0,100	90	15	83	3
Metazachlor		Summary	0,020	1,000	97	11	110	5
	GC	Water containing	0,050	1,000	100	9	39	2
	LC	Acidic	0,020	0,020	100	5	3	1
	LC	Sugar containing	0,020	0,020	81	5	2	1
	LC	Dry (Cereal)	0,020	0,020	109	4	2	1
	LC	Water containing	0,020	0,050	95	12	63	3
Methamidophos		Summary	0,010	0,100	84	14	111	3
	GC	Acidic	0,050	0,050	62		1	1
	GC	Water containing	0,050	0,075	76	23	7	1
	LC	Water containing	0,010	0,100	86	13	82	3
	LC	Acidic	0,010	0,100	78	11	14	2
	LC	Sugar containing	0,020	0,025	76	10	4	2
	LC	Dry (Cereal)	0,020	0,020	75	1	2	1
Methidathion		Summary	0,010	1,000	95	14	151	6
	GC	Water containing	0,050	1,000	98	16	58	5
	GC	Sugar containing	0,050	0,050	161		1	1
	LC	Acidic	0,010	0,100	90	10	23	2
	LC	Water containing	0,010	0,100	93	11	43	2
	LC	Sugar containing	0,010	0,100	94	5	14	2
	LC	Dry (Cereal)	0,020	0,200	99	1	12	1
Methomyl		Summary	0,010	2,500	95	16	142	4
	LC	Dry (Cereal)	0,020	0,020	97	9	2	1
	LC	Water containing	0,010	0,100	99	18	76	3
	LC	Acidic	0,010	0,100	91	13	13	2
	LC	Sugar containing	0,020	0,020	87	5	2	1
	LC	Dry	0,010	2,500	90	11	24	1
	LC	Other	0,010	2,500	87	10	24	1
Methoxychlor		Summary	0,050	1,000	100	15	57	4
	GC	Sugar containing	0,050	0,050	180		1	1

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	GC	Water containing	0,050	1,000	98	11	51	4
	GC	Acidic	0,100	0,100	105	4	5	1
Metobromuron		Summary	0,010	0,200	95	14	125	4
	LC	Sugar containing	0,010	0,100	96	13	14	2
	LC	Acidic	0,010	0,100	91	13	15	2
	LC	Dry (Cereal)	0,020	0,200	105	9	14	2
	LC	Water containing	0,010	0,100	94	14	81	4
Metolachlor		Summary	0,020	1,000	101	8	95	3
	GC	Water containing	0,050	1,000	103	6	39	2
	LC	Dry (Cereal)	0,020	0,020	104	7	2	1
	LC	Acidic	0,020	0,020	97	5	3	1
	LC	Sugar containing	0,020	0,020	92	8	2	1
	LC	Water containing	0,020	0,020	100	10	48	1
Metribuzin		Summary	0,010	1,000	97	13	131	3
	GC	Water containing	0,050	1,000	97	6	32	1
	LC	Sugar containing	0,010	0,100	90	9	14	2
	LC	Water containing	0,010	0,100	99	16	69	2
	LC	Acidic	0,020	0,020	111	7	2	1
	LC	Dry (Cereal)	0,020	0,200	90	11	14	2
Mevinphos		Summary	0,010	0,100	96	16	99	4
	GC	Dry	0,100	0,100	81	6	2	1
	GC	Water containing	0,010	0,100	97	20	33	3
	GC	Acidic	0,010	0,100	98	11	10	1
	LC	Acidic	0,020	0,020	97	13	3	1
	LC	Dry (Cereal)	0,020	0,020	102	6	2	1
	LC	Water containing	0,020	0,020	97	14	46	1
	LC	Sugar containing	0,020	0,020	77	7	2	1
Monocrotophos		Summary	0,010	0,200	89	12	163	5
	GC	Water containing	0,100	0,147	69	11	14	2
	LC	Dry (Cereal)	0,020	0,200	90	5	14	2
	LC	Water containing	0,010	0,100	92	11	94	3
	LC	Acidic	0,010	0,100	86	8	25	3
	LC	Sugar containing	0,010	0,100	90	7	15	3
Myclobutanil		Summary	0,010	1,000	95	12	101	6
	GC	Water containing	0,050	1,000	95	15	62	5
	LC	Water containing	0,010	0,100	96	8	26	1
	LC	Sugar containing	0,025	0,025	97	4	2	1
	LC	Acidic	0,010	0,100	95	6	11	1
Nuarimol		Summary	0,010	0,200	97	14	112	5
	GC	Water containing	0,010	0,200	102	20	38	4
	GC	Acidic	0,010	0,200	99	10	11	2
	GC	Dry	0,100	0,100	91	6	2	1
	GC	Sugar containing	0,200	0,200	115		1	1
	LC	Water containing	0,010	0,100	90	6	24	1
	LC	Acidic	0,010	0,100	99	7	12	1
	LC	Dry (Cereal)	0,020	0,200	100	8	12	1
	LC	Sugar containing	0,010	0,100	92	4	12	1
Omethoate		Summary	0,010	0,200	88	15	169	5
	GC	Water containing	0,100	0,100	91	9	7	1
	LC	Water containing	0,010	0,100	90	15	105	4
	LC	Sugar containing	0,010	0,100	89	15	16	3
	LC	Acidic	0,010	0,100	77	14	26	3
	LC	Dry (Cereal)	0,020	0,200	89	8	14	2
Orthophenylphenol		Summary	0,010	0,100	103	17	69	3
	GC	Acidic	0,010	0,100	96	9	15	2
	GC	Water containing	0,010	0,100	106	18	50	3
	GC	Dry	0,100	0,100	103	16	2	1
	GC	Sugar containing	0,100	0,100	91	13	2	1
Oxadixyl		Summary	0,010	1,000	97	11	160	5
	GC	Water containing	0,010	1,000	99	14	65	4
	GC	Acidic	0,010	0,100	100	13	10	1
	GC	Sugar containing	0,500	0,500	75		1	1
	GC	Dry	0,100	0,100	88	11	2	1
	LC	Acidic	0,010	0,100	90	10	13	2

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	LC	Water containing	0,010	0,100	93	9	42	2
	LC	Sugar containing	0,010	0,100	101	4	14	2
	LC	Dry (Cereal)	0,020	0,200	98	2	12	1
Oxamyl		Summary	0,010	0,100	95	20	76	3
	LC	Sugar containing	0,025	0,025	92	1	2	1
	LC	Water containing	0,020	0,100	98	21	57	3
	LC	Acidic	0,010	0,100	87	12	14	2
	LC	Dry (Cereal)	0,020	0,020	85	15	2	1
Paclobutrazol		Summary	0,010	1,000	95	10	147	3
	GC	Water containing	0,050	1,000	98	8	32	1
	LC	Sugar containing	0,010	0,100	88	7	14	2
	LC	Dry (Cereal)	0,020	0,200	98	9	14	2
	LC	Acidic	0,010	0,100	94	7	15	2
	LC	Water containing	0,010	0,100	95	11	71	2
Paraoxon		Summary	0,010	0,200	95	15	110	5
	GC	Water containing	0,010	0,200	102	17	38	4
	GC	Acidic	0,050	0,100	96	11	11	2
	GC	Dry	0,100	0,100	92		1	1
	LC	Acidic	0,010	0,100	79	18	12	1
	LC	Dry (Cereal)	0,020	0,200	102	3	12	1
	LC	Sugar containing	0,010	0,100	95	3	12	1
	LC	Water containing	0,010	0,100	89	7	24	1
Paraoxon-Methyl		Summary	0,010	0,200	90	15	99	4
	GC	Acidic	0,050	0,100	86	11	6	1
	GC	Water containing	0,050	0,200	95	19	31	3
	GC	Dry	0,100	0,100	78		1	1
	GC	Sugar containing	0,050	0,050	50		1	1
	LC	Sugar containing	0,010	0,100	92	4	12	1
	LC	Water containing	0,010	0,100	83	11	24	1
	LC	Dry (Cereal)	0,020	0,200	98	2	12	1
	LC	Acidic	0,010	0,100	84	9	12	1
Parathion		Summary	0,010	1,000	95	16	164	6
	GC	Dry	0,100	0,100	100		1	1
	GC	Sugar containing	0,050	0,100	102	6	3	1
	GC	Acidic	0,010	0,100	94	10	11	1
	GC	Water containing	0,010	1,000	102	10	89	5
	LC	Dry (Cereal)	0,020	0,200	75	10	12	1
	LC	Sugar containing	0,010	0,100	80	12	12	1
	LC	Water containing	0,010	0,100	90	21	24	1
	LC	Acidic	0,010	0,100	90	20	12	1
Parathion-Methyl		Summary	0,010	1,000	97	20	159	6
	GC	Acidic	0,010	0,100	91	7	11	1
	GC	Water containing	0,010	1,000	99	13	85	5
	GC	Dry	0,100	0,100	100		1	1
	GC	Sugar containing	0,050	0,100	118	3	2	1
	LC	Sugar containing	0,010	0,100	91	12	12	1
	LC	Acidic	0,010	0,100	94	36	12	1
	LC	Water containing	0,010	0,100	89	23	24	1
	LC	Dry (Cereal)	0,020	0,200	116	36	12	1
Penconazole		Summary	0,010	1,000	98	11	83	5
	GC	Water containing	0,050	1,000	98	12	53	4
	LC	Acidic	0,010	0,100	106	4	10	1
	LC	Water containing	0,010	0,025	96	8	20	1
Pencycuron		Summary	0,010	2,500	94	13	225	4
	GC	Water containing	0,050	1,000	98	9	32	1
	LC	Other	0,010	2,500	82	9	24	1
	LC	Sugar containing	0,010	0,100	94	7	14	2
	LC	Dry (Cereal)	0,020	0,200	92	6	14	2
	LC	Water containing	0,010	0,100	99	10	89	3
	LC	Acidic	0,010	0,100	99	8	27	3
	LC	Dry	0,010	2,500	74	14	24	1
Pendimethalin		Summary	0,010	1,000	96	14	218	7
	GC	Acidic	0,010	0,100	96	8	15	2
	GC	Sugar containing	0,100	0,100	90	19	2	1

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	GC	Water containing	0,010	1,000	103	11	88	5
	GC	Dry	0,100	0,100	129	40	2	1
	LC	Sugar containing	0,010	0,100	90	7	14	2
	LC	Dry (Cereal)	0,020	0,200	91	7	14	2
	LC	Water containing	0,010	0,100	90	15	69	2
	LC	Acidic	0,010	0,100	92	20	14	2
Permethrin		Summary	0,010	1,000	98	18	103	5
	GC	Sugar containing	0,100	0,200	97	16	3	2
	GC	Acidic	0,010	0,200	93	11	16	3
	GC	Dry	0,100	0,100	119	10	2	1
	GC	Water containing	0,010	1,000	98	20	82	5
Phorate		Summary	0,010	1,000	96	23	99	5
	GC	Acidic	0,010	0,500	103	11	12	2
	GC	Dry	0,100	0,100	90		1	1
	GC	Water containing	0,010	1,000	91	26	65	5
	LC	Water containing	0,010	0,025	100	14	11	1
	LC	Acidic	0,010	0,100	114	12	10	1
Phosalone		Summary	0,010	1,000	95	21	158	6
	GC	Acidic	0,010	0,400	95	10	11	2
	GC	Dry	0,100	0,100	148	74	2	1
	GC	Sugar containing	0,100	0,400	99	26	3	2
	GC	Water containing	0,010	1,000	100	23	82	5
	LC	Sugar containing	0,010	0,100	86	8	12	1
	LC	Water containing	0,010	0,100	80	8	24	1
	LC	Acidic	0,010	0,100	87	15	12	1
	LC	Dry (Cereal)	0,020	0,200	92	3	12	1
Phosmet		Summary	0,010	1,000	89	17	151	6
	GC	Dry	0,100	0,100	102		1	1
	GC	Acidic	0,010	0,100	91	9	11	1
	GC	Water containing	0,010	1,000	96	16	75	5
	GC	Sugar containing	0,050	0,200	104	43	3	2
	LC	Water containing	0,010	0,100	77	12	24	1
	LC	Dry (Cereal)	0,020	0,200	91	3	12	1
	LC	Sugar containing	0,010	0,100	73	4	12	1
	LC	Acidic	0,010	0,100	74	16	12	1
Phosphamidon		Summary	0,010	0,100	96	20	64	5
	GC	Dry	0,100	0,100	81		1	1
	GC	Water containing	0,010	0,100	97	20	44	5
	GC	Acidic	0,010	0,100	99	11	17	3
	GC	Sugar containing	0,050	0,100	62	52	2	2
Picoxystrobin		Summary	0,010	1,000	100	10	118	4
	GC	Acidic	0,100	0,100	100	9	5	1
	GC	Water containing	0,050	1,000	99	5	37	2
	LC	Dry (Cereal)	0,020	0,020	96	18	2	1
	LC	Acidic	0,010	0,100	108	9	13	2
	LC	Water containing	0,010	0,025	99	12	58	2
	LC	Sugar containing	0,020	0,020	96	18	2	1
Piperonyl butoxide		Summary	0,010	0,200	96	13	116	5
	GC	Water containing	0,010	0,100	98	19	38	4
	GC	Acidic	0,010	0,100	106	8	16	3
	GC	Sugar containing	0,050	0,050	103		1	1
	GC	Dry	0,100	0,100	88		1	1
	LC	Water containing	0,010	0,100	92	8	24	1
	LC	Acidic	0,010	0,100	93	6	12	1
	LC	Sugar containing	0,010	0,100	92	4	12	1
	LC	Dry (Cereal)	0,020	0,200	92	3	12	1
Pirimicarb		Summary	0,010	1,000	94	9	139	6
	GC	Acidic	0,100	0,100	97	2	5	1
	GC	Water containing	0,050	1,000	92	8	59	4
	LC	Water containing	0,010	0,025	95	11	57	2
	LC	Acidic	0,010	0,100	98	7	13	2
	LC	Dry (Cereal)	0,020	0,020	98	8	2	1
	LC	Sugar containing	0,020	0,020	92	19	2	1
Pirimiphos-Methyl		Summary	0,010	1,000	99	10	177	7

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	GC	Sugar containing	0,100	0,100	94	18	2	1
	GC	Dry	0,100	0,100	116	31	2	1
	GC	Water containing	0,010	1,000	103	10	96	6
	GC	Acidic	0,010	0,500	101	11	17	3
	LC	Water containing	0,010	0,100	89	4	24	1
	LC	Acidic	0,010	0,100	94	5	12	1
	LC	Sugar containing	0,010	0,100	91	6	12	1
	LC	Dry (Cereal)	0,020	0,200	97	2	12	1
Prochloraz		Summary	0,010	1,000	101	12	115	4
	GC	Acidic	0,010	0,100	104	14	10	1
	GC	Water containing	0,010	1,000	101	10	42	3
	GC	Dry	0,100	0,100	84		1	1
	LC	Water containing	0,020	0,025	100	14	54	2
	LC	Acidic	0,020	0,020	106	14	3	1
	LC	Sugar containing	0,020	0,020	82	1	2	1
	LC	Dry (Cereal)	0,020	0,020	105	18	2	1
Procymidone		Summary	0,010	1,000	100	11	118	6
	GC	Water containing	0,010	1,000	101	11	97	6
	GC	Sugar containing	0,050	0,100	96	9	3	2
	GC	Dry	0,100	0,100	107	16	2	1
	GC	Acidic	0,010	0,100	99	12	16	3
Profenofos		Summary	0,010	1,000	94	9	122	5
	GC	Water containing	0,050	1,000	93	10	46	3
	LC	Sugar containing	0,010	0,100	94	5	12	1
	LC	Acidic	0,010	0,100	98	8	12	1
	LC	Dry (Cereal)	0,020	0,200	89	8	12	1
	LC	Water containing	0,010	0,100	95	9	40	2
Profluralin		Summary	0,050	1,000	98	14	47	3
	GC	Water containing	0,050	1,000	98	14	47	3
Prometryn		Summary	0,010	1,000	95	9	151	5
	GC	Water containing	0,050	1,000	99	6	32	1
	LC	Dry (Cereal)	0,020	0,200	99	4	14	2
	LC	Water containing	0,010	0,100	93	10	87	4
	LC	Acidic	0,020	0,020	99	11	3	1
	LC	Sugar containing	0,010	0,100	94	2	14	2
Propamocarb		Summary	0,010	0,100	87	12	82	3
	LC	Acidic	0,010	0,100	83	9	13	2
	LC	Water containing	0,010	0,050	89	12	64	3
	LC	Sugar containing	0,020	0,020	73	11	2	1
	LC	Dry (Cereal)	0,020	0,020	86	5	2	1
Propargite		Summary	0,010	1,000	96	12	185	5
	GC	Water containing	0,050	1,000	101	6	40	2
	LC	Sugar containing	0,010	0,100	89	6	14	2
	LC	Dry (Cereal)	0,020	0,200	96	11	14	2
	LC	Water containing	0,010	0,100	93	13	93	3
	LC	Acidic	0,010	0,100	103	16	23	3
Propham		Summary	0,010	2,500	84	18	152	5
	GC	Acidic	0,100	0,100	103	2	5	1
	GC	Water containing	0,050	1,000	97	10	44	3
	LC	Water containing	0,010	0,100	84	10	31	2
	LC	Dry	0,010	2,500	72	23	24	1
	LC	Other	0,010	2,500	65	15	24	1
	LC	Dry (Cereal)	0,020	0,200	92	4	12	1
	LC	Sugar containing	0,010	0,100	87	3	12	1
Propiconazole		Summary	0,010	1,000	99	9	121	6
	GC	Water containing	0,050	1,000	102	9	52	4
	LC	Sugar containing	0,010	0,100	95	3	12	1
	LC	Water containing	0,010	0,100	94	9	35	2
	LC	Dry (Cereal)	0,020	0,200	100	4	12	1
	LC	Acidic	0,010	0,100	104	3	10	1
Propoxur		Summary	0,010	1,140	94	11	143	4
	GC	Water containing	1,140	1,140	98	12	6	1
	LC	Sugar containing	0,010	0,100	91	5	14	2
	LC	Dry (Cereal)	0,020	0,200	97	4	14	2

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	LC	Water containing	0,010	0,100	95	10	83	3
	LC	Acidic	0,010	0,100	91	17	25	3
Propyzamide		Summary	0,010	1,000	102	13	170	5
	GC	Water containing	0,010	1,000	101	13	75	4
	GC	Dry	0,100	0,100	106	11	2	1
	GC	Sugar containing	0,100	0,100	97	16	2	1
	GC	Acidic	0,010	0,100	93	9	10	1
	LC	Acidic	0,010	0,100	103	5	14	2
	LC	Sugar containing	0,020	0,020	95	34	2	1
	LC	Water containing	0,010	0,100	104	15	62	2
	LC	Dry (Cereal)	0,020	0,020	107	6	2	1
Prothiofos		Summary	0,010	1,000	98	12	163	7
	GC	Dry	0,100	0,100	91	6	2	1
	GC	Acidic	0,010	0,100	100	8	15	2
	GC	Water containing	0,010	1,000	102	9	77	6
	LC	Sugar containing	0,010	0,100	97	9	12	1
	LC	Acidic	0,010	0,100	99	11	12	1
	LC	Water containing	0,010	0,100	88	14	33	2
	LC	Dry (Cereal)	0,020	0,200	95	7	12	1
Pyraclostrobin		Summary	0,010	0,200	93	11	133	3
	LC	Sugar containing	0,010	0,100	86	10	14	2
	LC	Water containing	0,010	0,100	94	12	80	3
	LC	Acidic	0,010	0,100	93	7	24	3
	LC	Dry (Cereal)	0,020	0,200	94	6	14	2
Pyrazophos		Summary	0,010	1,000	97	10	122	6
	GC	Water containing	0,050	1,000	101	10	53	4
	LC	Sugar containing	0,010	0,100	91	5	12	1
	LC	Water containing	0,010	0,100	93	11	35	2
	LC	Acidic	0,010	0,100	105	3	10	1
	LC	Dry (Cereal)	0,020	0,200	95	3	12	1
Pyridaben		Summary	0,010	1,000	102	10	93	6
	GC	Water containing	0,050	1,000	102	10	66	5
	LC	Water containing	0,010	0,100	97	11	16	1
	LC	Acidic	0,010	0,100	108	3	11	1
Pyridaphenthion		Summary	0,010	0,100	101	13	49	5
	GC	Acidic	0,100	0,100	119	18	2	1
	GC	Water containing	0,052	0,100	96	12	26	4
	LC	Water containing	0,010	0,025	100	8	11	1
	LC	Acidic	0,010	0,100	110	14	10	1
Pyrifenox		Summary	0,010	0,501	93	10	102	5
	GC	Water containing	0,100	0,501	93	15	21	3
	LC	Dry (Cereal)	0,020	0,200	96	8	12	1
	LC	Sugar containing	0,010	0,100	93	5	12	1
	LC	Acidic	0,010	0,100	95	8	22	2
	LC	Water containing	0,010	0,100	91	8	35	2
Pyrimethanil		Summary	0,010	1,000	97	11	143	5
	GC	Acidic	0,100	0,100	99	3	5	1
	GC	Water containing	0,050	1,000	98	6	52	3
	LC	Water containing	0,010	0,025	96	15	68	2
	LC	Sugar containing	0,020	0,020	86	9	2	1
	LC	Acidic	0,010	0,100	101	8	13	2
	LC	Dry (Cereal)	0,020	0,020	93	23	2	1
Pyriproxyfen		Summary	0,010	1,000	96	10	129	5
	GC	Sugar containing	0,025	0,025	102	1	1	1
	GC	Acidic	0,025	0,100	102	7	6	2
	GC	Water containing	0,025	1,000	98	11	53	3
	LC	Sugar containing	0,010	0,100	93	6	12	1
	LC	Acidic	0,010	0,100	104	2	10	1
	LC	Water containing	0,010	0,100	91	9	35	2
	LC	Dry (Cereal)	0,020	0,200	91	8	12	1
Quinalphos		Summary	0,010	0,200	98	11	87	6
	GC	Sugar containing	0,050	0,050	95		1	1
	GC	Acidic	0,050	0,050	115		1	1
	GC	Water containing	0,050	0,100	104	12	30	4

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	LC	Water containing	0,010	0,100	93	10	31	2
	LC	Dry (Cereal)	0,020	0,200	97	4	12	1
	LC	Sugar containing	0,010	0,100	96	9	12	1
Quinoxifen		Summary	0,010	1,000	98	14	98	4
	GC	Water containing	0,010	1,000	98	18	44	4
	GC	Dry	0,100	0,100	91	7	2	1
	GC	Sugar containing	1,000	1,000	115		1	1
	GC	Acidic	0,010	1,000	102	9	16	3
	LC	Acidic	0,010	0,100	102	5	11	1
	LC	Water containing	0,010	0,100	95	13	24	1
Quintozene		Summary	0,010	0,100	100	14	71	5
	GC	Dry	0,100	0,100	90	47	2	1
	GC	Water containing	0,010	0,100	99	13	51	5
	GC	Acidic	0,010	0,100	103	12	17	3
	GC	Sugar containing	0,050	0,050	98		1	1
Simazine		Summary	0,010	0,250	94	12	77	3
	GC	Acidic	0,250	0,250	101		1	1
	GC	Sugar containing	0,250	0,250	99		1	1
	GC	Water containing	0,100	0,250	102	13	15	2
	LC	Acidic	0,010	0,100	98	18	12	1
	LC	Water containing	0,010	0,100	86	5	24	1
	LC	Sugar containing	0,010	0,100	86	4	12	1
	LC	Dry (Cereal)	0,020	0,200	100	5	12	1
Spiroxamine		Summary	0,010	2,500	90	16	221	5
	GC	Water containing	0,050	1,000	96	8	39	2
	LC	Water containing	0,010	0,100	90	17	92	3
	LC	Acidic	0,010	0,100	103	5	13	2
	LC	Dry	0,010	2,500	74	4	24	1
	LC	Sugar containing	0,010	0,100	95	11	14	2
	LC	Other	0,010	2,500	78	8	24	1
	LC	Dry (Cereal)	0,020	0,200	106	12	14	2
Sulfotep		Summary	0,032	1,000	102	16	73	6
	GC	Acidic	0,050	0,100	100	10	7	3
	GC	Sugar containing	0,050	0,050	107	18	2	2
	GC	Water containing	0,032	1,000	103	16	64	6
Tebuconazole		Summary	0,010	1,000	99	11	127	5
	GC	Water containing	0,050	1,000	99	11	46	3
	LC	Sugar containing	0,020	0,020	93	4	2	1
	LC	Acidic	0,010	0,100	99	8	13	2
	LC	Water containing	0,010	0,025	99	12	63	2
	LC	Dry (Cereal)	0,020	0,020	94	7	2	1
Tebufenpyrad		Summary	0,010	0,100	95	13	74	5
	GC	Acidic	0,100	0,100	98	14	6	2
	GC	Water containing	0,050	0,100	92	14	37	4
	GC	Sugar containing	0,100	0,100	100		1	1
	LC	Acidic	0,010	0,100	102	6	10	1
	LC	Water containing	0,010	0,025	95	11	20	1
Tecnazene		Summary	0,032	1,000	97	14	57	4
	GC	Water containing	0,032	1,000	96	15	52	4
	GC	Acidic	0,100	0,100	105	3	5	1
Teflubenzuron		Summary	0,010	0,200	100	26	147	3
	LC	Acidic	0,010	0,100	91	14	28	3
	LC	Water containing	0,010	0,100	104	30	96	3
	LC	Dry (Cereal)	0,020	0,200	89	18	14	2
	LC	Sugar containing	0,020	0,100	93	3	8	2
Terbacil		Summary	0,010	1,000	87	15	96	3
	GC	Water containing	0,050	1,000	98	10	37	2
	GC	Acidic	0,100	0,100	96	6	5	1
	LC	Sugar containing	0,100	0,100	89	2	6	1
	LC	Dry (Cereal)	0,020	0,200	88	4	12	1
	LC	Acidic	0,010	0,100	69	9	12	1
	LC	Water containing	0,010	0,100	77	13	24	1
Terbufos		Summary	0,050	1,000	100	14	67	5
	GC	Water containing	0,050	1,000	99	14	58	5

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	GC	Sugar containing	0,050	0,050	94		1	1
	GC	Acidic	0,050	0,500	106	7	8	3
Terbutylazine		Summary	0,010	0,200	93	14	153	4
	GC	Acidic	0,050	0,050	62		1	1
	GC	Sugar containing	0,050	0,050	85		1	1
	GC	Water containing	0,050	0,100	69	20	15	2
	LC	Dry (Cereal)	0,020	0,200	96	5	14	2
	LC	Sugar containing	0,010	0,100	92	9	14	2
	LC	Water containing	0,010	0,100	95	11	83	3
	LC	Acidic	0,010	0,100	103	10	25	3
Terbutryn		Summary	0,010	1,000	96	9	135	3
	GC	Water containing	0,050	1,000	98	9	32	1
	LC	Acidic	0,020	0,020	99	18	3	1
	LC	Water containing	0,010	0,100	96	9	71	2
	LC	Dry (Cereal)	0,020	0,200	97	6	14	2
	LC	Sugar containing	0,010	0,100	93	3	14	2
Tetrachlorvinphos		Summary	0,050	1,000	99	18	46	3
	GC	Water containing	0,050	1,000	99	18	46	3
Tetraconazole		Summary	0,010	1,000	98	12	200	6
	GC	Water containing	0,010	1,000	101	9	57	4
	GC	Dry	0,100	0,100	86		1	1
	GC	Acidic	0,010	0,100	102	9	10	1
	LC	Water containing	0,010	0,100	96	14	92	3
	LC	Sugar containing	0,010	0,100	91	11	14	2
	LC	Acidic	0,010	0,100	105	4	12	2
	LC	Dry (Cereal)	0,020	0,200	95	5	14	2
Tetradifon		Summary	0,010	1,000	100	14	106	6
	GC	Sugar containing	0,100	0,100	110	3	2	1
	GC	Acidic	0,010	0,250	94	12	16	3
	GC	Dry	0,100	0,100	96		1	1
	GC	Water containing	0,010	1,000	101	14	87	6
Tetramethrin		Summary	0,010	1,000	102	14	75	4
	GC	Water containing	0,010	1,000	103	14	63	4
	GC	Acidic	0,100	0,100	94	5	10	2
	GC	Dry	0,100	0,100	100	16	2	1
Thiabendazole		Summary	0,010	2,500	83	24	125	3
	LC	Dry (Cereal)	0,020	0,020	110	1	2	1
	LC	Dry	0,010	2,500	53	5	24	1
	LC	Acidic	0,010	0,100	97	14	12	2
	LC	Other	0,010	2,500	60	5	24	1
	LC	Sugar containing	0,020	0,020	97	21	2	1
	LC	Water containing	0,010	0,025	101	12	60	2
Thiacloprid		Summary	0,010	0,050	100	14	73	3
	LC	Dry (Cereal)	0,020	0,020	89	14	2	1
	LC	Acidic	0,010	0,020	102	13	8	2
	LC	Sugar containing	0,020	0,020	79	12	2	1
	LC	Water containing	0,010	0,050	100	14	60	3
Thiamethoxam		Summary	0,010	0,050	96	19	69	3
	LC	Dry (Cereal)	0,020	0,020	96	10	2	1
	LC	Sugar containing	0,020	0,020	67	16	2	1
	LC	Water containing	0,010	0,050	97	20	56	3
	LC	Acidic	0,010	0,020	96	9	8	2
Thiodicarb		Summary	0,010	2,500	86	14	126	3
	LC	Water containing	0,010	0,100	91	15	59	2
	LC	Dry	0,010	2,500	77	6	24	1
	LC	Acidic	0,010	0,100	91	15	15	2
	LC	Other	0,010	2,500	77	6	24	1
	LC	Dry (Cereal)	0,020	0,020	94	2	2	1
	LC	Sugar containing	0,020	0,020	81	16	2	1
Thiometon		Summary	0,050	0,100	99	20	28	3
	GC	Sugar containing	0,050	0,050	105		1	1
	GC	Acidic	0,050	0,100	100	11	7	3
	GC	Water containing	0,050	0,100	98	23	20	3
Tolclofos-Methyl		Summary	0,010	1,000	99	13	175	7

Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	GC	Dry	0,100	0,100	108	11	2	1
	GC	Water containing	0,010	1,000	103	11	94	6
	GC	Sugar containing	0,100	0,200	112	31	3	2
	GC	Acidic	0,010	0,100	98	9	15	2
	LC	Dry (Cereal)	0,020	0,200	100	6	12	1
	LC	Acidic	0,010	0,100	83	14	12	1
	LC	Water containing	0,010	0,100	89	11	24	1
	LC	Sugar containing	0,010	0,100	96	7	12	1
Tolyfluanid		Summary	0,010	1,000	90	27	78	4
	GC	Water containing	0,010	1,000	90	22	70	4
	GC	Acidic	0,100	0,100	58	8	5	1
	GC	Dry	0,100	0,100	102		1	1
	LC	Water containing	0,025	0,050	75	13	2	1
Triadimefon		Summary	0,010	1,000	97	11	168	6
	GC	Water containing	0,010	1,000	99	11	84	5
	GC	Dry	0,100	0,100	108	11	2	1
	GC	Acidic	0,100	0,500	91	17	7	2
	GC	Sugar containing	0,100	0,100	94	16	2	1
	LC	Acidic	0,010	0,100	107	3	11	1
	LC	Sugar containing	0,010	0,100	92	4	12	1
	LC	Dry (Cereal)	0,020	0,200	97	6	12	1
	LC	Water containing	0,010	0,100	94	8	38	2
Triadimenol		Summary	0,010	1,000	96	11	145	7
	GC	Acidic	0,010	0,100	99	16	17	3
	GC	Water containing	0,010	1,000	97	13	78	6
	GC	Sugar containing	0,050	0,050	108		1	1
	GC	Dry	0,100	0,100	84		1	1
	LC	Dry (Cereal)	0,020	0,200	102	4	12	1
	LC	Water containing	0,010	0,100	92	5	24	1
	LC	Sugar containing	0,010	0,100	93	3	12	1
Tri-Allate		Summary	0,010	1,000	91	17	98	3
	GC	Water containing	0,043	1,000	100	7	38	2
	LC	Sugar containing	0,010	0,100	91	5	12	1
	LC	Acidic	0,010	0,100	60	33	12	1
	LC	Water containing	0,010	0,100	92	5	24	1
	LC	Dry (Cereal)	0,020	0,200	91	5	12	1
Triazophos		Summary	0,010	1,000	97	12	129	5
	GC	Sugar containing	0,050	0,050	99		1	1
	GC	Acidic	0,050	0,050	140		1	1
	GC	Water containing	0,050	1,000	99	12	46	3
	LC	Dry (Cereal)	0,020	0,200	98	8	12	1
	LC	Acidic	0,010	0,100	102	15	22	2
	LC	Water containing	0,010	0,100	92	8	35	2
	LC	Sugar containing	0,010	0,100	91	7	12	1
Trifloxystrobin		Summary	0,010	1,000	96	13	193	7
	GC	Acidic	0,010	0,100	95	8	15	2
	GC	Water containing	0,010	1,000	96	12	86	6
	GC	Dry	0,100	0,100	100		1	1
	GC	Sugar containing	0,100	0,100	110		1	1
	LC	Water containing	0,010	0,100	100	16	44	2
	LC	Dry (Cereal)	0,020	0,200	88	6	12	1
	LC	Sugar containing	0,010	0,100	84	4	12	1
	LC	Acidic	0,010	0,100	95	14	22	2
Triflumizole		Summary	0,010	1,000	93	12	124	5
	GC	Water containing	0,050	1,000	95	11	39	2
	GC	Sugar containing	0,250	0,250	108		1	1
	GC	Acidic	0,250	0,250	107		1	1
	LC	Water containing	0,010	0,100	90	15	47	3
	LC	Sugar containing	0,010	0,100	92	2	12	1
	LC	Acidic	0,010	0,100	97	5	12	1
	LC	Dry (Cereal)	0,020	0,200	97	3	12	1
Triflumuron		Summary	0,010	0,200	96	14	154	3
	LC	Water containing	0,010	0,100	98	14	81	3
	LC	Other	0,010	0,100	77	9	12	1

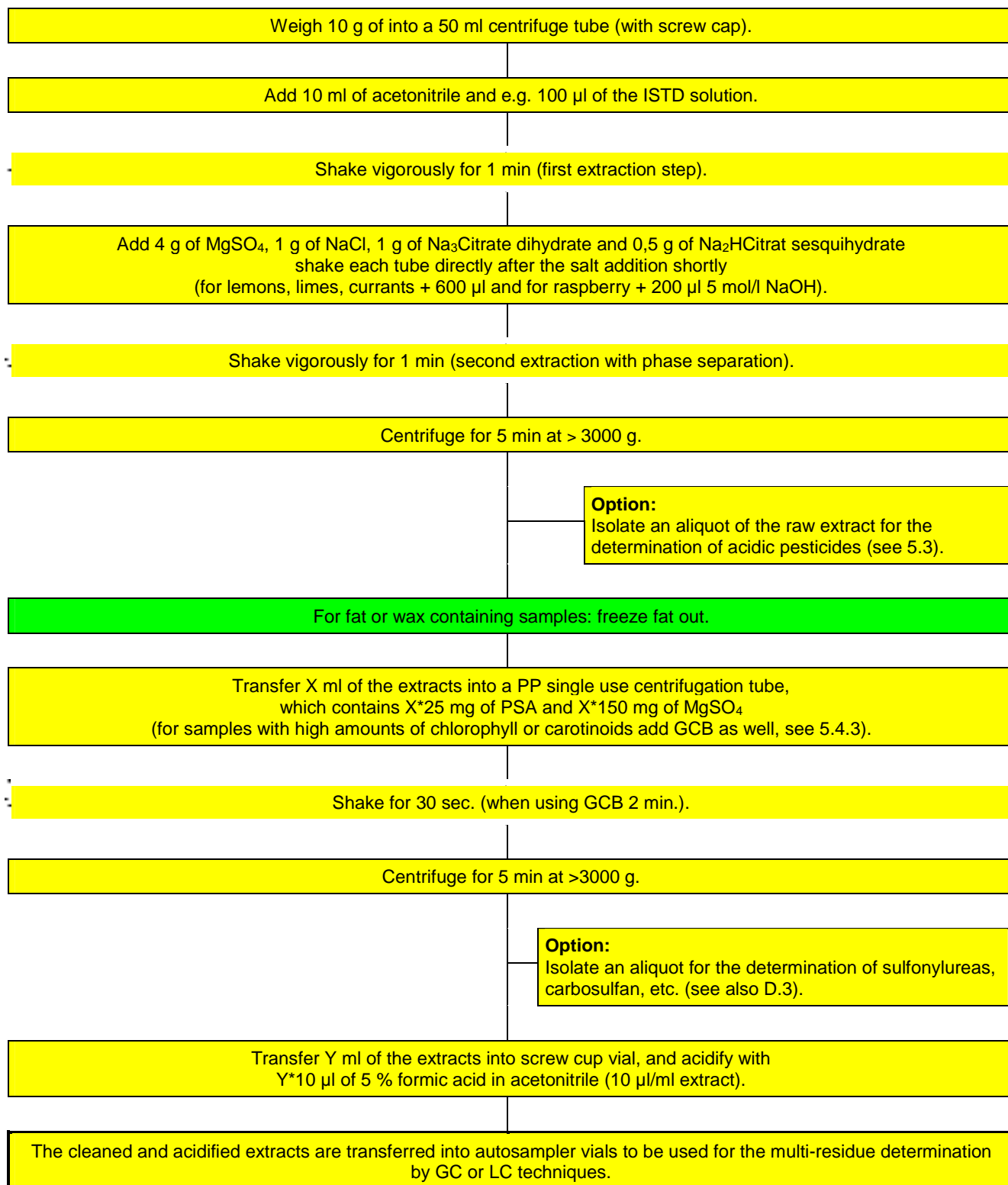
Table B.2 (continued)

Pesticide	LC/ GC	Matrix Type	Spiking Level mg/kg		Recoveries ^a			N° of Labs
			min	max	X %	V %	n	
	LC	Dry (Cereal)	0,020	0,200	93	10	14	2
	LC	Sugar containing	0,020	0,100	105	4	8	2
	LC	Dry	0,010	0,100	85	9	12	1
	LC	Acidic	0,010	0,100	101	10	26	3
Trifluralin		Summary	0,010	1,000	99	17	145	7
	GC	Water containing	0,010	1,000	103	13	88	6
	GC	Dry	0,100	0,100	127	41	2	1
	GC	Sugar containing	0,100	0,200	105	31	3	2
	GC	Acidic	0,010	0,200	103	11	16	3
	LC	Acidic	0,100	0,100	69	14	3	1
	LC	Water containing	0,010	0,100	75	11	15	1
	LC	Dry (Cereal)	0,020	0,200	98	16	12	1
	LC	Sugar containing	0,100	0,100	95	18	6	1
Triforine		Summary	0,010	0,100	97	23	75	3
	LC	Acidic	0,010	0,100	114	25	13	2
	LC	Sugar containing	0,020	0,020	107	11	2	1
	LC	Dry (Cereal)	0,020	0,020	127	9	2	1
	LC	Water containing	0,010	0,050	91	19	57	3
Vinclozolin		Summary	0,010	1,000	100	18	133	6
	GC	Acidic	0,010	0,100	89	9	15	2
	GC	Dry	0,100	0,100	108	13	2	1
	GC	Water containing	0,010	1,000	101	18	113	6
	GC	Sugar containing	0,100	0,100	116	35	3	1

a X = recovery, V = relative standard deviation; n = number of results

Annex C (informative)

Procedure schematically (for 10 g sample)



Annex D (informative)

Complementary information

D.1 General

This method (QuEChERS) was first published by M. Anastassiades *et al.* [4] in 2003 and later amended to the present procedure in order to broaden the analyte and matrix spectrum.

D.2 Scaling

The described method refers to 10 g sample for extraction (except for materials with water content < 30 %, (see Table 1 and Table 3)) and to 6 ml extract for clean-up. The described extraction and clean-up steps are scalable as desired, as long as the amounts of reagents used remain in the same proportion. It should be kept in mind, however, that the smaller the amount of sample employed the higher the sub-sampling variability will be. During validation, each laboratory should thus investigate the typical sub-sampling variability achieved when employing the available comminution devices, using representative samples containing incurred residues.

D.3 Adjustment of pH-value

By adding the citrate buffering salts (3.12) in 5.3 most samples obtain pH-values between 5 and 5,5. This pH range is a compromise, at which both, the quantitative extraction of acidic herbicides and the protection of alkali labile (e.g. captan, folpet, tolylfluanid) and acid labile (e.g. pymetrozine, dioxacarb) compounds is sufficiently achieved.

Following contact with PSA (5.4) the pH of the extracts increases, reaching measured values exceeding the value of 8. This may compromise the stability of base sensitive pesticides (e.g. captan, folpet, dichlofluanid, tolylfluanid, pyridate, methiocarb sulfon, chlorothalonil). If the extracts are acidified quickly to pH 5 the degradation of such compounds is reduced significantly so that storage over several days is possible. At this pH acid-labile pesticides (e.g. pymetrozine, dioxacarb, thiodicarb) are also sufficiently stable over several days. Only some very sensitive sulfonyl urea herbicides, carbosulfan and benfuracarb have been shown not to be sufficiently protected at pH 5. However, these compounds have been shown to be stable at the pH of the non-acidified extract (after dispersive SPE) over several days. If these compounds are within the scope of analysis an aliquot of the non-acidified extract should be employed for measurement. If the measurement can be performed quickly, the extract at pH 5 can be used as well. It should be noted, however, that the most acidic sulfonylureas may experience losses during PSA-clean-up. These may be analysed together with the acidic pesticides directly from the raw extract (5.3 and A.4). Carbosulfan and benfuracarb (both having individual MRLs) are degraded to carbofuran within the samples as well as in the extracts at pH 5. Thus, merely if carbofuran is present in the acidified extract an additional run of the alkaline aliquot is needed.

D.4 Recovery studies

For recovery studies e.g. 10 g sample is fortified using 100 µl of a pesticide solution in acetonitrile or acetone. A short vibration using a Vortex mixer (4.13) can help to disperse solvent and pesticides well throughout the sample. Fortification using larger volumes of standard solution (e. g. > 200 µl) should be avoided. If this is not possible, a volume compensation should be performed in the blank samples used to prepare matrix matched calibration solutions, to avoid differences in the matrix concentration of the final extract.

D.5 Clean-up with GCB (5.4.3)

It has to be taken into account, that some planar pesticides and ISTDs have a great affinity towards the planar structure of GCB. But recovery studies showed that no noteworthy losses occur if the extract, after dispersive SPE with GCB, still maintains some visible amount of chlorophyll or carotinoides. Anthracene (or d10-Anthracen) may be used as QC standards (see Table 1). If the recovery of anthracene is above 70 %, this will also be the case for planar pesticides having the highest affinity towards carbon.

D.6 Concentration of the end extracts and solvent exchange

If large volume injection (3 µl or more) cannot be performed and the desired detection limits of the compounds of interest cannot be achieved, the concentration of the end extracts and, if necessary, a solvent exchange may be considered. If GC/MSD is employed a simple evaporative concentration of the extracts by a factor of four should be sufficient. To achieve this e.g. 4 ml of the acidified extract (pH 5) are transferred into a test tube and reduced to approximately 1 ml at 40 °C using a slight nitrogen flow. Solvent exchange is an option if GC performance using acetonitrile is not satisfactory or if NPD is employed (without PTV-injector). For this, an extract aliquot is evaporated to almost dryness at 40 °C using a slight nitrogen flow and resolved in 1 ml of an appropriate solvent (some droplets of a keeper e.g. dodecane can help to reduce losses of the most volatile compounds). The blank extract (needed for the preparation of calibration solutions) should be treated the same way.

D.7 Alternative calibration and calculation

In the following text, the following variables are used:

— Mass of pesticide in calibration mixture	$m_{pest}^{cal\ mix}$	µg
— Mass of internal standard in calibration mixture	$m_{ISTD}^{cal\ mix}$	µg
— Mass of internal standard added to test portion	m_{ISTD}^{sample}	µg
— Mass of pesticide in final extract	m_{pest}^{sample}	µg
— Concentration of pesticide in calibration mixture	$C_{pest}^{cal\ mix}$	µg/ml
— Concentration of the ISTD in ISTD solution adding to test portion	C_{ISTD}	µg/ml
— Concentration of the ISTD in diluted ISTD solution used for calibration mixture	$C_{ISTD}^{cal\ mix}$	µg/ml
— Volume of pesticide working solution used for preparation of calibration mixture	$V_{pest}^{cal\ mix}$	ml
— Volume of ISTD used for preparation of calibration mixture	$V_{ISTD}^{cal\ mix}$	ml
— Volume of ISTD added to the test portion	V_{ISTD}^{sample}	ml
— Mass of test portion	m_a	g
— Mass fraction of pesticide in the sample	w_R	mg/kg

— Peak area of pesticide obtained from calibration mixture	$A_{pest}^{cal\ mix}$	(counts)
— Peak area of ISTD obtained from calibration mixture	$A_{ISTD}^{cal\ mix}$	(counts)
— Peak area of pesticide obtained from the final extract	A_{pest}^{sample}	(counts)
— Peak area of ISTD obtained from the final extract	A_{ISTD}^{sample}	(counts)
— Peak ratio obtained from calibration mixture	$PR^{cal\ mix}$	(dimensionless)
— Peak ratio obtained from final extract	PR^{sample}	(dimensionless)
— Slope of calibration graph	a_{cal}	(dimensionless)
— Slope of calibration graph using the simplified approach	a_{cal}^{simpl}	1/μg
— Bias of calibration graph	b_{cal}	(dimensionless)

This alternative and simplified approach of calibration and calculation requires to maintain a known and constant ratio of the ISTD-masses in the sample and the standard solutions ($m_{ISTD}^{sample} / m_{ISTD}^{cal\ mix}$), see Table 2 in 3.22. Hereby m_{ISTD}^{sample} should correspond to the entire mass of the test portion (m_a) and $m_{ISTD}^{cal\ mix}$ to the entire mass of pesticide in the standard solution (calibration mixture) ($m_{pest}^{cal\ mix}$). This approach relies on determining the mass of the pesticides in the entire sample extract and thus in the test portion. The abovementioned ISTD-mass ratio is considered in the calculation as a correction factor. The absolute concentration of the ISTD-solution used is thus irrelevant and does not appear in the formula.

Calibration: Determine the calibration functions for each active substance by plotting the peak ratio $PR^{cal\ mix}$ ($= A_{pest}^{cal\ mix} / A_{ISTD}^{cal\ mix}$) of each calibration level against the mass of active substance in the standard solution $m_{pest}^{cal\ mix}$. The corresponding calibration graph is:

$$PR^{cal\ mix} = a_{cal}^{simpl} \times m_{pest}^{cal\ mix} + b_{cal} \quad (D.1)$$

The mass fraction w_R of the pesticide in the sample is calculated using the peak ratio of pesticide and internal standard PR^{sample} ($= A_{pest}^{sample} / A_{ISTD}^{sample}$) obtained from final extract as:

$$w_R = \frac{(PR^{sample} - b_{cal})}{a_{cal}^{simpl}} \times \frac{1}{m_a} \times \frac{m_{ISTD}^{sample}}{m_{ISTD}^{cal\ mix}} \left(\frac{mg}{kg} \right) \quad (D.2)$$

This approach derives from the following calibration approach using peak ratios and mass ratios:

Determine the calibration functions for each active substance by plotting the peak ratio $PR^{cal\ mix}$ ($= A_{pest}^{cal\ mix} / A_{ISTD}^{cal\ mix}$) of each calibration level against the dimensionless mass ratio ($m_{pest}^{cal\ mix} / m_{ISTD}^{cal\ mix}$) of the standard solution. From the corresponding calibration graph:

$$PR^{cal\ mix} = a_{cal}^{simpl} \times \frac{m_{pest}^{cal\ mix}}{m_{ISTD}^{cal\ mix}} + b_{cal} \quad (D.3)$$

each expected mass ratio $m_{pest}^{cal\ mix} / m_{ISTD}^{cal\ mix}$ can be calculated as follows:

$$\frac{m_{pest}^{cal\ mix}}{m_{ISTD}^{cal\ mix}} = \frac{PR^{cal\ mix} - b_{cal}}{a_{cal}^{simpl}} \quad (D.4)$$

The slope can be calculated as follows:

$$a_{cal}^{simpl} = \frac{PR^{cal\ mix} - b_{cal}}{\frac{m_{pest}^{cal\ mix}}{m_{ISTD}^{cal\ mix}}} \quad (D.5)$$

The mass ratio $m_{pest}^{sample} / m_{ISTD}^{sample}$ in the final extract depends on the mass fraction w_R of the pesticide in the test portion m_a and the mass of the internal standard m_{ISTD}^{sample} ($= C_{ISTD} \times V_{ISTD}^{sample}$) added to the test portion.

$$\frac{m_{pest}^{sample}}{m_{ISTD}^{sample}} = \frac{w_R \times m_a}{C_{ISTD} \times V_{ISTD}^{sample}} \quad (D.6)$$

When the peak ratio PR^{sample} ($= A_{pest}^{sample} / A_{ISTD}^{sample}$) obtained from final extract is identical to the peak ratio $PR^{cal\ mix}$ obtained from calibration mixture, the mass ratios $m_{pest}^{sample} / m_{ISTD}^{sample}$ and $m_{pest}^{cal\ mix} / m_{ISTD}^{cal\ mix}$ are identical. From Equations (D.4) and (D.6) follows

$$\frac{m_{pest}^{sample}}{m_{ISTD}^{sample}} = \frac{w_R \times m_a}{m_{ISTD}^{sample}} = \frac{PR^{sample} - b_{cal}}{a_{cal}^{simpl}} = \frac{PR^{cal\ mix} - b_{cal}}{a_{cal}^{simpl}} = \frac{m_{pest}^{cal\ mix}}{m_{ISTD}^{cal\ mix}} \quad (D.7)$$

and the mass fraction w_R is calculated as follows:

$$w_R = \frac{PR^{sample} - b_{cal}}{a_{cal}^{simpl}} \times \frac{m_{ISTD}^{sample}}{m_a} \left(\frac{\text{mg}}{\text{kg}} \right) \quad (D.8)$$

or under consideration of Equation (D.3):

$$w_R = \frac{PR^{sample} - b_{cal}}{PR^{cal\ mix} - b_{cal}} \times \frac{m_{ISTD}^{sample}}{m_a} \left(\frac{\text{mg}}{\text{kg}} \right) \quad (D.9)$$

$$\frac{m_{pest}^{cal\ mix}}{m_{ISTD}^{cal\ mix}}$$

and thus

$$w_R = \frac{(PR^{sample} - b_{cal})}{\frac{PR^{cal mix} - b_{cal}}{m_{pest}^{cal mix}}} \times \frac{1}{m_a} \times \frac{m_{ISTD}^{sample}}{m_{ISTD}^{cal mix}} \left(\frac{\text{mg}}{\text{kg}} \right) \quad (\text{D.10})$$

Therefore the mass fraction w_R is a function of the peak ratios, the mass of the pesticide in calibration mixture, the mass of test portion and the ratio of the mass of the internal standard in the final extract and the calibration mixture.

Equation (D.10) can be simplified to Equation (D.2) using Equation (D.1) for the calibration graph of the simplified approach.

Bibliography

- [1] DG-SANCO, *Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed*, Document N° SANCO/2007/3131, 31 October 2007
- [2] Arbeitsgruppe „Pestizide“: 5. Empfehlung: Kriterien zur Vorbereitung und Reduzierung von Proben pflanzlicher Lebensmittel für die Rückstandsanalyse von Pflanzenschutz- und Schädlingsbekämpfungsmitteln, *Lebensmittelchemie* 49, 40–42 (1995)
- [3] L. Alder, K. Greulich, G. Kempe and B. Vieth (2006), 'Residue Analysis of 500 High Priority Pesticides – better by GC-MS or LC-MS/MS', *Mass Spectrometry Reviews*, vol. 25 n° 6, pp., 838-865
- [4] M. Anastassiades, S. J. Lehotay, D. Stajnbaher and F. J. Schenck (2003), 'Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and “Dispersive Solid-Phase Extraction” for the Determination of Pesticide Residues in Produce', *Journal of AOAC International*, vol. 86, n° 2, pp. 412-431
- [5] CEN/TR 15641 *Food analysis – Determination of pesticide residues by LC-MS/MS – Tandem mass spectrometric parameters*
- [6] ISO 5725 (all parts), *Accuracy (trueness and precision) of measurement methods and results*
- [7] Data Pool of the Community Reference Laboratories for Residues of Pesticides, online resources: <http://www.crl-pesticides-datapool.eu>

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