BRITISH STANDARD

BS EN 15637:2008

Foods of plant origin — Determination of pesticide residues using LC-MS/MS following methanol extraction and clean-up using diatomaceous earth

ICS 67.050

BSI British Standards

National foreword

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Pflanzliche Lebensmittel - LC-MS/MS-Verfahren zur Bestimmung von Pestizidrückständen mit Methanolextraktion und Reinigung an Diatomeenerde

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Foreword

This document (EN 15637:2008) 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of an identical text or by endorsement, at the latest by May 2009, and conflicting national standards shall be withdrawn at the latest by May 2009.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, vegetables, cereals, nuts as well as processed products including dried fruits. The method has been collaboratively studied on a large number of commodity/pesticide combinations.

2 Principle

The sample is extracted with methanol after addition of some water. After partition into dichloromethane the organic phase is evaporated and the residue is reconstituted with methanol. Quantification of pesticide residues is performed by liquid chromatography with tandem mass spectrometric detection, using electrospray ionisation. To achieve the required selectivity the mass spectrometer is operated in the selected reaction monitoring mode (SRM).

3 Reagents

3.1 General and safety considerations

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

3.2 Ammonium formate

3.3 Sodium chloride

3.4 Water, HPLC quality

3.5 Dichloromethane, for residue analysis

3.6 Methanol, HPLC quality

3.7 Internal Standard (ISTD) solutions in methanol, ρ = 10 µg/ml to 50 µg/ml[1)]

Table 1 shows a list of potential internal standards that may be used in this method. The concentrations listed refer to the ISTD solutions that should be added at the first extraction step (5.2) and to standard solutions.

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

Name of the compound	Log P (octanol-water partion coefficient)	Chlorine atoms	Concentration C_{ISTD} µg/ml
Triphenyl phosphate	4,59	-	20
Tris-(1,3-dichlorisopropyl)-phosphate	3,65	6	50
Bis-nitrophenyl urea (nicarbazin)	3,76	-	10

1) ρ = mass concentration

3.8 Pesticide stock solutions

Prepare individual stock solutions of analytical standards at concentrations that are sufficiently high to allow the preparation of complex pesticide mixtures. The solvent used should not negatively influence the stability of the pesticides employed.

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

3.9 Pesticide mixtures

Because of the broad applicability of this method and due to the partly divergent pH-stability of pesticides, analyte mixtures of different composition can be needed. These are prepared by mixing together defined volumes of the required analyte stock solutions (3.8) and appropriately diluting them with methanol. The analyte concentrations in this mixture should be sufficient to allow the preparation of the required matrix matched standards (see 3.10.3) with moderate dilution of the blank sample extract (e.g. less than 20 %).

Usually, store pesticide mixtures at ≤ -18 °C. Since the stability of the pesticides in the mixture may be lower than in stock solutions, stability has to be checked regularly. In some cases the addition of acids or bases can be helpful to enhance stability and extend acceptable storage times.

3.10 Standard solutions

3.10.1 Standard solutions prepared in pure solvent (solvent-based standards)

Solvent-based standards are prepared by mixing a certain volume of methanol with known amounts of pesticide mixtures (3.9). The preparation of multiple standards of different pesticide concentration is useful to cover a broad concentration range.

NOTE An analyte concentration of 1 µg/ml correlates to a residue level of 0,4 mg/kg when a 10 g sample is employed (e.g. samples with water content > 30 %) or 0,8 mg/kg when a 5 g sample is employed (e.g. cereals).

3.10.2 Standard solutions with internal standard prepared in pure solvent

Solvent-based standards with ISTD are prepared by mixing a certain volume of methanol with known amounts of pesticide mixtures (3.9) and a fixed volume of internal standard solution (3.7). The volume used shall result in that concentration of ISTD which is obtained in the final extracts after sample extraction and clean-up (see 5.2 and 5.3). The concentration of internal standard in the final extract (C_{ISTD}^{sample}) can be calculated using Equation (1). The preparation of multiple standards of different pesticide concentration but with constant ISTD concentration is useful to cover a broad concentration range.

$$C_{ISTD}^{sample} = \frac{V_{ISTD} \times C_{ISTD} \times (V_2 - V_1) \times V_3}{V_2 \times V_{ex} \times V_{end}} \quad (1)$$

where:

V_{ISTD} is the volume of internal standard solution (3.7) added to the test portion;

C_{ISTD} is the concentration of internal standard solution (3.7);

V_1 is the volume of NaCl solution (2,5 ml);

V_2 is the volume of measuring flask used in 5.2 (10 ml);

V_3 is the volume used for solid supported liquid/liquid extraction (5 ml);

V_{ex} is the total volume of extraction solvents and natural water (30 ml);

V_{end} is the final volume of extract obtained after clean-up (0,5 ml).

NOTE The internal standard may correct for deviations from the correct extraction volume, a wrong estimate of water content of samples, losses of methanol during preparation of the final extract and fluctuations of instrument sensitivity during a batch of measurements. However, validation results in Annex B were obtained without internal standards.

3.10.3 Standard solutions prepared in blank matrix extracts (matrix-matched standards)

Prepare matrix-matched standards in the same way as the solvent-based standards, however, instead of pure methanol use extracts of blank samples (prepared as described in 5.2, 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, carrots for carrot samples, etc.).

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

3.11 5 ml cartridge for solid supported liquid/liquid extraction, 5 ml sample volume, diatomaceous earth, for example ChemElut CE 1005[2)]

3.12 20 ml cartridge for solid supported liquid/liquid extraction, 20 ml sample volume, diatomaceous earth, for example ChemElut CE 1020[2)]

4 Apparatus

Usual laboratory apparatus and, in particular, the following:

4.1 Carving board and knife, for chopping up food samples for analysis

4.2 Homogenizer or high speed blender, fitted with jar

4.3 Laboratory balance

4.4 Measuring flasks, 10 ml and 20 ml

4.5 Ultrasonic bath

4.6 Centrifuge tubes, 80 ml

4.7 Centrifuge, capable of producing a relative centrifugal force (RCF) of at least 3000 *g* (at the bottom of the tube)

4.8 Round bottom flasks, 50 ml and 250 ml

4.9 Glass syringe, minimum volume 2 ml

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

4.10 Microliter syringes, for sample fortification

4.11 Rotary evaporator, with temperature-controlled water bath

4.12 Syringe filters, 0,45 µm pore size, 4 mm diameter, polytetrafluoroethylene (PTFE) membrane

4.13 Glass vials and caps, 1,8 ml volume, suitable for an autosampler

4.14 LC-MS/MS system, triple quadrupole mass spectrometer with electrospray interface

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 supports 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 shall 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 [1].

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 analytes and usually results in smaller particle sizes and thus achieve a higher degree of homogeneity. Cutting the samples coarsely (e. g. 3 cm x 3 cm) with a knife and putting them into the freezer (e. g. -18 °C overnight) prior to comminution facilitates processing. Processing can be also assisted and improved by cryogenic milling (using dry ice or liquid nitrogen) by keeping the temperature below 0 °C. Especially in the case of fruits and vegetables, cryogenic milling is much more effective at homogenising commodities that have tough skins (e. g. tomatoes or grapes) compared to milling at ambient temperature. Given the fact that non-systemic pesticides often predominantly occur on the skin, cryogenic milling significantly reduces sub-sampling variability. When processing test samples at low temperatures, condensation caused by high humidity should be avoided. Residual carbon dioxide should be allowed to sufficiently dissipate so that its contribution to weigh of the sample will be negligible.

5.1.5 Test portion

Individual test portions each sufficient for one analysis are taken from the comminuted test sample. These test portions should be analysed immediately. If test portions cannot be analysed directly, the test sample or the test portions shall be frozen until required. If 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.

5.2 Extraction

Transfer a representative test portion of m_A = 10 g into a centrifuge tube (4.6). For dry sample materials like cereal products, weigh a homogenised portion of 5 g (m_A) into the centrifuge tube. Add sufficient water, that a total volume (added and natural) of 10 ml water is obtained. For typical water contents of crops and cereals, see Table 2. In the case of dry sample materials wait 10 min after addition of water. Add 20 ml of methanol (3.6) to the mixture and homogenise for 2 min using the high speed blender (4.2). Take at least 10 ml of the resulting extract of 30 ml (= V_{ex}) and centrifuge at approximately 3000 *g.* Pipette 2,5 ml of NaCl solution (20 %, w/w) *(*= V_1*)* into a 10 ml measuring flask *(*= V_2*)* (4.4), fill up to the mark with supernatant of centrifugation and mix.

As an option an internal standard can be used additionally. In that case add a small volume (<1 % of V_{ex}) of internal standard solution (=V_{ISTD}) to the test portion after addition of 20 ml of methanol.

Table 2 — Water content of selected foods and amount of water, which have to be added

Food group	Food	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
Fruits				
Citrus fruits	citrus juices	90	1,0	
	grapefruit	90	1,0	
	lemon	90	1,0	
	orange	85	1,5	
	orange peel	75	2,5	
	tangerine	90	1,0	
Pome fruit	apple	85	1,5	
	apple, dried	30		8,5
	apple sauce	80	2,0	
	apple juice	90	1,0	
	pear	85	1,5	
	quince	85	1,5	
Stone fruit	apricot	85	1,5	
	apricot, dried	30		8,5
	apricot nectar	85	1,5	
	cherry	85	1,5	
	mirabelle	80	2,0	
	nectarine	85	1,5	
	peach	90	1,0	
	peach, dried	20		9,0
	plum	85	1,5	
	plum, dried	20		9,0
Soft and small fruits	blackberry	85	1,5	
	blueberry	85	1,5	
	currant	85	1,5	
	elderberry	80	2,0	
	gooseberry	90	1,0	
	grapes	80	2,0	
	raspberry	85	1,5	
	raisin	20		9,0
	strawberry	90	1,0	
Other fruits	pineapple	85	1,5	
	banana	75	2,5	

Table 2 *(continued)*

Food group	Food	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
	fig, dried	20		9,0
	kiwi	85	1,5	
	mango	80	2,0	
	papaya	90	1,0	
Vegetables				
Root and tuber vegetables	beetroot	90	1,0	
	carrot	90	1,0	
	celeriac	90	1,0	
	horseradish	75	2,5	
	parsley root	90	1,0	
	radish	95	0,5	
	scorzonera (black salsify)	80	2,0	
	shallot	80	2,0	
Onions	garlic	60		7,0
	onion	90	1,0	
Fruiting vegetables	aubergine	90	1,0	
	cucumber	95	0,5	
	melon	90	1,0	
	pepper, sweet	90	1,0	
	pumpkin	95	0,5	
	tomato	95	0,5	
	zucchini (courgette)	95	0,5	
Cabbage	broccoli	90	1,0	
	Brussels sprouts	85	1,5	
	cauliflower	90	1,0	
	Chinese cabbage	95	0,5	
	kale	90	1,0	
	kohlrabi	90	1,0	
	red cabbage	90	1,0	
	savoy cabbage	90	1,0	
	white cabbage	90	1,0	

Table 2 *(continued)*

Food group	Food	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
Leafy vegetables and	butterhead lettuce	95	0,5	
herbs	chive	85	1,5	
	cress	90	1,0	
	endive	95	0,5	
	iceberg lettuce	95	0,5	
	lamb's lettuce	85	1,5	
	parsley	80	2,0	
	spinach	90	1,0	
	witloof chicory	95	0,5	
Stem vegetables	artichokes	85	1,5	
	asparagus	95	0,5	
	celery	95	0,5	
	leek	85	1,5	
	rhubarb	95	0,5	
Beans, peas (fresh)	beans	90	1,0	
	peas with pods	80	2,0	
Beans, peas (dried)	beans, peas, lentil	10		9,5
Other				
	beer	90	1,0	
	cereals (grain, flour, etc.)	10		9,5
	coffee (raw)	10		9,5
	mushrooms	90	1,0	
	must (grape)	90	1,0	
	potato	80	2,0	
	tea	10		9,5
	wine	90	1,0	

5.3 Clean-up

Apply 5 ml of the diluted centrifugate *(= V_3)* from 5.2 to a 5 ml cartridge (3.11). After 5 minutes, elute into a 50 ml round bottom flask (4.8), using 12,5 ml of dichloromethane (3.5). Repeat the elution with another 12,5 ml of dichloromethane. Reduce the combined eluates almost to dryness using the rotary evaporator (4.11). Remaining dichloromethane should be removed with a stream of nitrogen.

Add 500 µl methanol (3.6) to the round bottom flask and weigh with stopper. Carefully dissolve the residue by swirling the flask in the ultrasonic bath (4.5), but avoid losses of methanol. If losses of methanol occur (re-weighing), add methanol to obtain the previous total weight. Filter the obtained sample test solution of 0,5 ml *(= V_{end})* through a PTFE-filter (4.12) into a sample vial (4.13) for injection.

To obtain a larger amount of sample test solution for the preparation of matrix-matched standards (3.10.3) a 20 ml cartridge (3.12) can be used. In that case, 400 % of all volumes mentioned above have to be used.

NOTE The sample test solution contains the extractable components of 2,5 g sample per millilitre final extract (or 1,25 g/0,5 ml).

5.4 Determination

The sample test solutions (5.3) and calibration solutions (3.10.1, 3.10.2 or 3.10.3) are injected into the LC-MS/MS instrument in an appropriate sequence. This can involve bracketing of the sample extracts with the calibration solutions. In the injector needle of the HPLC system the sample test solution should be diluted with eluent A. The LC-MS/MS instrument shall be operated in the selected reaction monitoring (SRM) mode with transitions selective for the pesticides under investigation. For suitable experimental conditions see CEN/TR 15641 [4]. Nevertheless, individual tuning of the compounds on the instrument that is used for measurement usually provides better sensitivities.

The measurement can 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.

NOTE Most validation results listed in Annex B have been obtained after mixing of sample test solution with water in a LC vial and not in the injector of the HPLC system. In that case a ratio between methanolic extract and water of 1:4 (V/V) was used. Also, standard solutions were diluted with water in the volume ratio 1:4 (V/V). Most samples contained small amounts of co-extracted components that are not soluble in the resulting methanol/water mixture. As a result a turbid emulsion (or suspension) was obtained. It was recognized that recovery of some less polar pesticides was reduced, if such emulsions have formed.

5.5 Test for interference and recovery

Prepare reagent blanks and carry out spiked recovery tests at levels appropriate to the maximum residue level. The chromatogram of the reagent blank should not show any significant peak (e. g. 10 % of relevant MRL) at the retention time of the analytes.

6 Evaluation of results

6.1 Identification and quantification

To identify analytes compare the retention times obtained from the sample test solution with those obtained from the calibration solutions. Positive findings are confirmed by comparing the peak intensity ratios of the first and second compound specific m/z transition with the peak intensity ratios found in standards. If the peak ratio of a residue peak differs more than 20 % from the expected response ratio, check the EU-quality control guidelines described in the SANCO/2007/3131 document [2]. A different LC column, another eluent or an additional m/z transition may be used, if additional measures are necessary.

Use standard solutions (3.10.1 or 3.10.2) or matrix-matched standards (3.10.3) to check linearity and to determine the calibration functions for each active substance by plotting the peak areas or heights (if ISTDs are not used) or peak ratios (if ISTDs are used) of one SRM transition against the analyte concentration [ng/ml] of the standard solution.

For a first estimate of the residue level of pesticides in the food or to show their absence, the standard solutions (3.10.1 or 3.10.2) in pure methanol 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 (3.10.3) or the standard addition method should be preferred.

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 can be necessary to construct more than one calibration graph from the results of calibration measurements.

When using ISTDs 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 clean-up will result in an overestimation of analyte concentration. Such losses should thus be minimal. 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. 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 are mandatory.

6.2 Calculation of residue concentrations without standard addition

If standard addition method is not used, the residue level w_R of a pesticide in the food sample is calculated from the obtained peak area (or height) using Equation 2:

$$w_R = \frac{A-c}{b} \times \frac{V_{ex}}{m_a} \times \frac{V_{end} \times V_2}{(V_2 - V_1) \times V_3} \times 1000 \left(\frac{\text{mg}}{\text{kg}} \right) \quad (2)$$

where:

A is the peak area, peak height or peak ratio for one SRM transition measured, in arbitrary units (a.u) or without dimension;

c is the intercept of the corresponding calibration graph, in a.u. or without dimension;

b is the slope of the corresponding calibration graph, in a.u. $\times$ ml/ng (without ISTD) or ml/ng (with ISTD);

V_{ex} is the total volume of extraction solvents and natural water (30 ml);

m_a is the initial sample weight, in grams;

V_1 is the volume of NaCl solution (2,5 ml);

V_2 is the volume of measuring flask used in 5.2 (10 ml);

V_3 is the volume used for solid supported liquid/liquid extraction (5 ml);

V_{end} is the final volume of extract obtained after clean-up (0,5 ml);

1000 is the conversion factor.

If the results indicate that the amount of residue approaches or exceeds the maximum residue level, at least one further test portion shall be analysed.

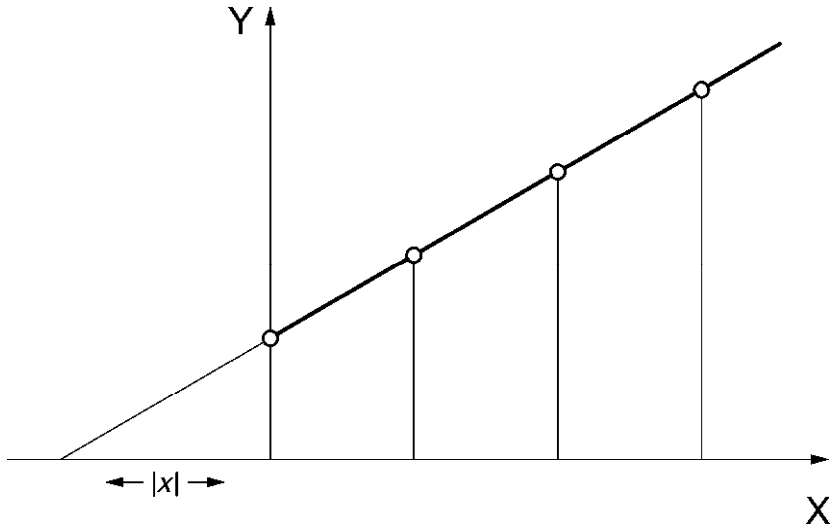
6.3 Calculation of residue concentrations with standard addition

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 known amounts of the analyte of interest. This procedure requires the knowledge of the approximate residue level w_R from preliminary analysis.

The standard solutions used for standard addition shall have nearly identical solvent composition compared to the sample test solution from 5.3. Assuming a sample (used sample amount 10 g) with an estimated residue level of w_R = 0,8 mg/kg, the following pipetting scheme may be appropriate. (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.) The amount of analyte in the sample is calculated using a graphical presentation of resulting response data as shown in Figure 1 via linear regression.

Table 3 — Pipetting scheme for standard addition approach

Additions	Vial 1	Vial 2	Vial 3	Vial 4
Volume of sample extract V_{aliq}	100 µl (= 0,25 g sample)	100 µl (= 0,25 g sample)	100 µl (= 0,25 g sample)	100 µl (= 0,25 g sample)
Volume of analyte standard solution (20 µg/ml)	0 µl	5 µl	10 µl	15 µl
Resulting mass of analyte added	0 µg	0,1 µg	0,2 µg	0,3 µg
Volume of solvent	15 µl	10 µl	5 µl	0 µl
Final volume	115 µl	115 µl	115 µl	115 µl



Key:

Y Peak area Analyte

X Added absolute amount of analyte in µg

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

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

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

If standard addition method is used, the residue level w_R of a pesticide in the food sample is calculated from the obtained peak area (or height) using Equation 3:

$$w_R = \frac{c}{b} \times \frac{V_{ex}}{m_a} \times \frac{V_2 \times V_{end}}{(V_2 - V_1) \times V_3 \times V_{aliq}} \left(\frac{\text{mg}}{\text{kg}} \right) \quad (3)$$

where:

c is the y-intercept of the calibration graph of the analyte in question in arbitrary units (a.u.);

b is the slope of the calibration graph of the analyte in question (a.u./µg);

V_{ex} is the total volume of extraction solvents and natural water (30 ml);

m_a is the initial sample weight (5 g or 10 g);

V_1 is the volume of NaCl solution (2,5 ml);

V_2 is the volume of measuring flask used in 5.2 (10 ml);

V_3 is the volume used for solid supported liquid/liquid extraction (5 ml);

V_{end} is the final volume of extract obtained after clean-up (0,5 ml);

V_{aliq} is the aliquot of final volume of extract obtained after clean-up (0,1 ml).

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 volative residue. For more information about the confirmation of identity refer to the EU-quality control guidelines described in the SANCO/2007/3131 document [2].

8 Precision

Details of the inter-laboratory test of the precision of the method according to ISO 5725-1 and ISO 5725-2 [3] are summarised in Annex B. The values derived from the inter-laboratory test may not be applicable to analyte 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;

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

— date of receipt of sample in the laboratory;

— date of test;

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

— any particular points observed 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)

Example for appropriate experimental conditions

The following LC-MS/MS operating conditions have been proven to be satisfactory.

A.1 HPLC system 1

For most LC-amenable compounds:

HPLC pump	HP1100 Binary Pump (G1312A)
Autosampler	HP1100 (G1313A)
Injector programme	draw 3 µl eluent A draw 2 µl sample wash needle with methanol draw 2 µl eluant A draw 2 µl sample wash needle with methanol draw 2 µl eluant A draw 2 µl sample wash needle with methanol draw 2 µl eluant A draw 2 µl sample wash needle with methanol draw 3 µl eluent A
Column	Phenomenex Aqua 5µ C18 125 Å, 50 mm × 2 mm
Mobile phase A	Methanol/water 2+8 (*V/V*) with 5 mmol/l ammonium formate (3.2)
Mobile phase B	Methanol/water 9+1 (*V/V*) with 5 mmol/l ammonium formate
Column temperature	20 °C

Table A.1 — Flow rate and elution gradient

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

A.2 HPLC-System 2

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	Ammonium formate solution in water, *c* = 5 mmol/l
Mobile phase B	Ammonium formate solution in methanol, *c* = 5 mmol/l
Column temperature	40 °C
Injection volume	5 µl

Table A.2 — Flow rate and elution gradient

Time min	**Flow rate** µl/min	**Mobile phase A** %	**Mobile phase B** %
0	300	50	50
20	300	0	100
25	300	0	100
26	300	50	50
30	300	50	50

A.3 HPLC-System 3

For polar compounds 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	Ammonium formate solution in water, *c* = 5 mmol/l
Mobile phase B	Ammonium formate solution in methanol, *c* = 5 mmol/l
Column temperature	40 °C
Injection volume	3 µl, automatically diluted with 3 µl of mobile phase A during injection procedure

Table A.3 — Flow rate and elution gradient:

Time min	**Flow rate** µl/min	**Mobile phase A** %	**Mobile phase B** %
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 A (1 : 1), and 6 µl thereof should be injected.

A.4 HPLC-System 4

Four acidic compounds:

Column	Zorbax XDB C18, length 150 mm, inner diameter 2,1 mm, particle size 3,5 µm
Mobile phase A	Acetic acid solution in water, σ = 0,1 ml glacial acetic acid /l
Mobile phase B	Acetic acid solution in acetonitrile, σ = 0,1 ml glacial acetic acid /l
Column temperature	40 °C
Injection volume	5 µl

Table A.4 — Flow rate and elution gradient:

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

A.5 MS/MS system 1

MS/MS instrument Applied Biosystems API 2000

Ion source Turbo Ion Spray (ESI)

Table A.5 — Ion source and general parameters

Curtain gas	nitrogen, 35 psi	Gas 2 temperature	400 °C
Collision gas	nitrogen, 2 units	Resolution MS 1	unit
Ion spray voltage	5500 V	Resolution MS 2	unit
Gas 1	nitrogen, 60 psi	Dwell time	25 ms
Gas 2	nitrogen, 60 psi	Focusing potential	360 V

A.6 MS/MS system 2

MS/MS instrument Micromass Quattro LC

Ion source Electrospray

Table A.6 — Ion source and general parameters

Nebulizer gas flow	nitrogen, 93 l/h	MS1 LM Resolution	14,7
Desolvation gas flow	nitrogen, 552 l/h	MS1 HM Resolution	14,7
Desolvation temp.	350 °C	MS2 LM Resolution	14,7
Capillary voltage	3500 V	MS2 HM Resolution	14,7
Gas cell	9,2 x 10^{-4} mbar		

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 validation trails. An updated version of validation data can be found on the website www.crl-pesticides-datapool.eu, which is run by the EU Community Reference Laboratories for Pesticides.

Table B.1 — Results of validation study of the German working group „Unterarbeitsgruppe Analytik der Bund-Länder-Arbeitsgruppe Pflanzenschutz- und Schädlingsbekämpfungsmittel“ (n approximately 12 000)

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
3,4,5-Trimethacarb	water containing	0,100	97	22	35	7
3,4,5-Trimethacarb	water containing	0,010	95	12	40	8
3,4,5-Trimethacarb	acidic	0,100	87	16	40	8
3,4,5-Trimethacarb	acidic	0,010	88	18	40	8
3,4,5-Trimethacarb	cereal (dry)	0,100	82	30	25	5
3,4,5-Trimethacarb	cereal (dry)	0,010	73	21	25	5
3,4,5-Trimethacarb	fatty	0,100	79	21	25	5
3,4,5-Trimethacarb	fatty	0,010	81	28	20	4
Acephate	water containing	0,100	87	20	35	7
Acephate	water containing	0,010	85	23	40	8
Acephate	acidic	0,100	81	28	40	8
Acephate	acidic	0,010	88	23	40	8
Acephate	cereal (dry)	0,100	85	29	25	5
Acephate	cereal (dry)	0,010	74	40	25	5
Acephate	fatty	0,100	85	21	25	5
Acephate	fatty	0,010	86	24	20	4
Aldicarb	water containing	0,100	85	37	30	6
Aldicarb	water containing	0,010	82	31	35	7
Aldicarb	acidic	0,100	79	31	35	7
Aldicarb	acidic	0,010	74	37	30	6
Aldicarb	cereal (dry)	0,100	92	20	25	5
Aldicarb	cereal (dry)	0,010	89	9	25	5
Aldicarb	fatty	0,100	97	18	25	5

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Aldicarb	fatty	0,010	88	9	20	4
Azoxystrobin	water containing	0,100	92	20	35	7
Azoxystrobin	water containing	0,010	97	11	35	7
Azoxystrobin	acidic	0,100	72	24	40	8
Azoxystrobin	acidic	0,010	70	24	40	8
Azoxystrobin	cereal (dry)	0,100	72	22	25	5
Azoxystrobin	cereal (dry)	0,010	69	10	25	5
Azoxystrobin	fatty	0,100	78	13	25	5
Azoxystrobin	fatty	0,010	81	33	20	4
Bendiocarb	water containing	0,100	101	26	30	6
Bendiocarb	water containing	0,010	98	18	35	7
Bendiocarb	acidic	0,100	92	12	35	7
Bendiocarb	acidic	0,010	95	28	35	7
Bendiocarb	cereal (dry)	0,100	84	40	20	4
Bendiocarb	cereal (dry)	0,010	91	35	25	5
Bendiocarb	fatty	0,100	95	53	25	5
Bendiocarb	fatty	0,010	95	35	20	4
Butocarboxim	water containing	0,100	61	62	30	6
Butocarboxim	water containing	0,010	70	75	35	7
Butocarboxim	acidic	0,100	62	70	35	7
Butocarboxim	acidic	0,010	60	63	25	5
Butocarboxim	cereal (dry)	0,100	88	21	20	4
Butocarboxim	cereal (dry)	0,010	81	14	20	4
Butocarboxim	fatty	0,100	91	25	20	4
Butocarboxim	fatty	0,010	76	44	15	3
Carbaryl	water containing	0,100	99	21	35	7
Carbaryl	water containing	0,010	96	17	40	8
Carbaryl	acidic	0,100	91	14	40	8
Carbaryl	acidic	0,010	84	33	40	8
Carbaryl	cereal (dry)	0,100	90	29	25	5
Carbaryl	cereal (dry)	0,010	78	26	25	5

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Carbaryl	fatty	0,100	81	20	25	5
Carbaryl	fatty	0,010	101	27	20	4
Carbendazim	water containing	0,100	67	70	35	7
Carbendazim	water containing	0,010	69	59	40	8
Carbendazim	acidic	0,100	84	26	40	8
Carbendazim	acidic	0,010	88	18	35	7
Carbendazim	cereal (dry)	0,100	80	49	25	5
Carbendazim	cereal (dry)	0,010	66	53	25	5
Carbendazim	fatty	0,100	63	59	25	5
Carbendazim	fatty	0,010	71	69	20	4
Carbofuran	water containing	0,100	104	24	35	7
Carbofuran	water containing	0,010	108	24	40	8
Carbofuran	acidic	0,100	102	21	40	8
Carbofuran	acidic	0,010	105	26	40	8
Carbofuran	cereal (dry)	0,100	104	27	25	5
Carbofuran	cereal (dry)	0,010	89	25	25	5
Carbofuran	fatty	0,100	100	22	25	5
Carbofuran	fatty	0,010	104	22	20	4
Cinosulfuron	water containing	0,100	83	25	35	7
Cinosulfuron	water containing	0,010	86	27	40	8
Cinosulfuron	acidic	0,100	84	15	40	8
Cinosulfuron	acidic	0,010	92	21	40	8
Cinosulfuron	cereal (dry)	0,100	95	28	25	5
Cinosulfuron	cereal (dry)	0,010	80	28	25	5
Cinosulfuron	fatty	0,100	84	19	25	5
Cinosulfuron	fatty	0,010	94	13	20	4
Cyprodinil	water containing	0,100	77	40	30	6
Cyprodinil	water containing	0,010	75	51	35	7
Cyprodinil	acidic	0,100	41	56	40	8
Cyprodinil	acidic	0,010	45	112	40	8
Cyprodinil	cereal (dry)	0,100	90	94	25	5

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Cyprodinil	cereal (dry)	0,010	67	41	20	4
Cyprodinil	fatty	0,100	11	105	25	5
Cyprodinil	fatty	0,010	49	89	10	2
Dimethoat	water containing	0,100	107	18	35	7
Dimethoat	water containing	0,010	104	25	35	7
Dimethoat	acidic	0,100	101	25	40	8
Dimethoat	acidic	0,010	108	25	40	8
Dimethoat	cereal (dry)	0,100	104	28	25	5
Dimethoat	cereal (dry)	0,010	92	35	25	5
Dimethoat	fatty	0,100	109	23	25	5
Dimethoat	fatty	0,010	108	31	20	4
Ethiofencarb	water containing	0,100	33	87	30	6
Ethiofencarb	water containing	0,010	22	114	35	7
Ethiofencarb	acidic	0,100	56	63	40	8
Ethiofencarb	acidic	0,010	47	84	40	8
Ethiofencarb	cereal (dry)	0,100	54	56	25	5
Ethiofencarb	cereal (dry)	0,010	49	60	25	5
Ethiofencarb	fatty	0,100	72	17	25	5
Ethiofencarb	fatty	0,010	87	26	20	4
Fenhexamid	water containing	0,100	87	42	35	7
Fenhexamid	water containing	0,010	75	65	35	7
Fenhexamid	acidic	0,100	69	41	40	8
Fenhexamid	acidic	0,010	69	52	35	7
Fenhexamid	cereal (dry)	0,100	86	29	25	5
Fenhexamid	cereal (dry)	0,010	81	23	20	4
Fenhexamid	fatty	0,100	78	16	25	5
Fenhexamid	fatty	0,010	70	47	15	3
Fenoxycarb	water containing	0,100	75	40	35	7
Fenoxycarb	water containing	0,010	75	53	40	8
Fenoxycarb	acidic	0,100	47	50	40	8
Fenoxycarb	acidic	0,010	51	65	35	7

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Fenoxycarb	cereal (dry)	0,100	56	23	25	5
Fenoxycarb	cereal (dry)	0,010	58	25	20	4
Fenoxycarb	fatty	0,100	32	38	25	5
Fenoxycarb	fatty	0,010	40	65	15	3
Fenpropimorph	water containing	0,100	72	35	35	7
Fenpropimorph	water containing	0,010	59	64	40	8
Fenpropimorph	acidic	0,100	72	22	40	8
Fenpropimorph	acidic	0,010	74	31	40	8
Fenpropimorph	cereal (dry)	0,100	50	41	25	5
Fenpropimorph	cereal (dry)	0,010	42	66	25	5
Fenpropimorph	fatty	0,100	13	74	20	4
Fenpropimorph	fatty	0,010	26	98	15	3
Flufenoxuron	water containing	0,100	65	36	30	6
Flufenoxuron	water containing	0,010	64	56	40	8
Flufenoxuron	acidic	0,100	29	95	30	6
Flufenoxuron	acidic	0,010	18	185	40	8
Flufenoxuron	cereal (dry)	0,100	45	37	25	5
Flufenoxuron	cereal (dry)	0,010	38	19	20	4
Flufenoxuron	fatty	0,100	18	83	15	3
Flufenoxuron	fatty	0,010	25	141	10	2
Imazalil	water containing	0,100	74	38	25	5
Imazalil	water containing	0,010	94	22	25	5
Imazalil	acidic	0,100	78	28	30	6
Imazalil	acidic	0,010	80	45	30	6
Imazalil	cereal (dry)	0,100	57	20	15	3
Imazalil	cereal (dry)	0,010	31	116	20	4
Imazalil	fatty	0,100	54	21	15	3
Imazalil	fatty	0,010	74	79	10	2
Imidacloprid	water containing	0,100	99	12	35	7
Imidacloprid	water containing	0,010	102	15	40	8
Imidacloprid	acidic	0,100	95	9	40	8

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Imidacloprid	acidic	0,010	103	16	40	8
Imidacloprid	cereal (dry)	0,100	96	23	25	5
Imidacloprid	cereal (dry)	0,010	86	21	25	5
Imidacloprid	fatty	0,100	98	16	25	5
Imidacloprid	fatty	0,010	96	2	20	4
Indoxacarb	water containing	0,100	71	38	35	7
Indoxacarb	water containing	0,010	62	67	35	7
Indoxacarb	acidic	0,100	28	125	40	8
Indoxacarb	acidic	0,010	44	107	35	7
Indoxacarb	cereal (dry)	0,100	50	27	25	5
Indoxacarb	cereal (dry)	0,010	44	16	15	3
Indoxacarb	fatty	0,100	21	68	20	4
Indoxacarb	fatty	0,010	27	73	15	3
Iprovalicarb	water containing	0,100	96	17	35	7
Iprovalicarb	water containing	0,010	96	14	40	8
Iprovalicarb	acidic	0,100	83	19	40	8
Iprovalicarb	acidic	0,010	83	15	40	8
Iprovalicarb	cereal (dry)	0,100	80	15	25	5
Iprovalicarb	cereal (dry)	0,010	73	9	25	5
Iprovalicarb	fatty	0,100	78	16	25	5
Iprovalicarb	fatty	0,010	85	32	20	4
Isoproturon	water containing	0,100	99	18	35	7
Isoproturon	water containing	0,010	93	40	40	8
Isoproturon	acidic	0,100	93	14	40	8
Isoproturon	acidic	0,010	92	22	40	8
Isoproturon	cereal (dry)	0,100	91	28	25	5
Isoproturon	cereal (dry)	0,010	92	12	20	4
Isoproturon	fatty	0,100	92	13	25	5
Isoproturon	fatty	0,010	87	14	15	3
Linuron	water containing	0,100	91	23	35	7
Linuron	water containing	0,010	87	30	40	8

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Linuron	acidic	0,100	72	28	40	8
Linuron	acidic	0,010	77	44	40	8
Linuron	cereal (dry)	0,100	81	27	25	5
Linuron	cereal (dry)	0,010	86	25	20	4
Linuron	fatty	0,100	61	26	25	5
Linuron	fatty	0,010	59	62	15	3
Metalaxyl	water containing	0,100	103	16	35	7
Metalaxyl	water containing	0,010	107	19	40	8
Metalaxyl	acidic	0,100	96	21	40	8
Metalaxyl	acidic	0,010	97	23	40	8
Metalaxyl	cereal (dry)	0,100	92	22	25	5
Metalaxyl	cereal (dry)	0,010	85	20	25	5
Metalaxyl	fatty	0,100	99	15	25	5
Metalaxyl	fatty	0,010	100	21	20	4
Methamidophos	water containing	0,100	77	22	35	7
Methamidophos	water containing	0,010	70	16	40	8
Methamidophos	acidic	0,100	68	15	40	8
Methamidophos	acidic	0,010	71	34	40	8
Methamidophos	cereal (dry)	0,100	73	29	25	5
Methamidophos	cereal (dry)	0,010	77	23	25	5
Methamidophos	fatty	0,100	75	24	25	5
Methamidophos	fatty	0,010	69	39	20	4
Methiocarb	water containing	0,100	91	21	35	7
Methiocarb	water containing	0,010	91	19	40	8
Methiocarb	acidic	0,100	73	26	40	8
Methiocarb	acidic	0,010	74	29	40	8
Methiocarb	cereal (dry)	0,100	71	27	25	5
Methiocarb	cereal (dry)	0,010	56	42	25	5
Methiocarb	fatty	0,100	53	46	25	5
Methiocarb	fatty	0,010	64	7	20	4
Methomyl	water containing	0,100	106	29	30	6

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Methomyl	water containing	0,010	108	34	35	7
Methomyl	acidic	0,100	96	17	35	7
Methomyl	acidic	0,010	106	13	35	7
Methomyl	cereal (dry)	0,100	120	64	25	5
Methomyl	cereal (dry)	0,010	107	25	25	5
Methomyl	fatty	0,100	124	57	25	5
Methomyl	fatty	0,010	114	29	15	3
Methoxyfenozid	water containing	0,100	98	10	30	6
Methoxyfenozid	water containing	0,010	85	50	30	6
Methoxyfenozid	acidic	0,100	83	13	35	7
Methoxyfenozid	acidic	0,010	72	48	35	7
Methoxyfenozid	cereal (dry)	0,100	71	24	20	4
Methoxyfenozid	cereal (dry)	0,010	62	28	15	3
Methoxyfenozid	fatty	0,100	79	19	20	4
Methoxyfenozid	fatty	0,010	82	15	15	3
Metolachlor	water containing	0,100	93	19	35	7
Metolachlor	water containing	0,010	107	50	40	8
Metolachlor	acidic	0,100	88	34	35	7
Metolachlor	acidic	0,010	77	34	40	8
Metolachlor	cereal (dry)	0,100	88	26	25	5
Metolachlor	cereal (dry)	0,010	83	29	25	5
Metolachlor	fatty	0,100	66	23	25	5
Metolachlor	fatty	0,010	71	32	20	4
Metsulfuron-methyl	water containing	0,100	94	14	35	7
Metsulfuron-methyl	water containing	0,010	92	18	40	8
Metsulfuron-methyl	acidic	0,100	83	14	40	8
Metsulfuron-methyl	acidic	0,010	91	16	40	8
Metsulfuron-methyl	cereal (dry)	0,100	78	31	25	5
Metsulfuron-methyl	cereal (dry)	0,010	83	40	25	5
Metsulfuron-methyl	fatty	0,100	96	24	25	5
Metsulfuron-methyl	fatty	0,010	100	13	20	4

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Monocrotophos	water containing	0,100	113	29	35	7
Monocrotophos	water containing	0,010	108	17	35	7
Monocrotophos	acidic	0,100	101	19	35	7
Monocrotophos	acidic	0,010	102	18	35	7
Monocrotophos	cereal (dry)	0,100	98	22	25	5
Monocrotophos	cereal (dry)	0,010	93	27	25	5
Monocrotophos	fatty	0,100	103	12	25	5
Monocrotophos	fatty	0,010	104	15	20	4
Oxamyl	water containing	0,100	109	19	35	7
Oxamyl	water containing	0,010	111	14	40	8
Oxamyl	acidic	0,100	104	12	40	8
Oxamyl	acidic	0,010	103	17	40	8
Oxamyl	cereal (dry)	0,100	100	33	25	5
Oxamyl	cereal (dry)	0,010	89	37	25	5
Oxamyl	fatty	0,100	99	39	25	5
Oxamyl	fatty	0,010	100	28	20	4
Oxydemeton-methyl	water containing	0,100	112	17	35	7
Oxydemeton-methyl	water containing	0,010	112	15	40	8
Oxydemeton-methyl	acidic	0,100	108	19	40	8
Oxydemeton-methyl	acidic	0,010	110	19	40	8
Oxydemeton-methyl	cereal (dry)	0,100	103	29	25	5
Oxydemeton-methyl	cereal (dry)	0,010	89	35	25	5
Oxydemeton-methyl	fatty	0,100	116	34	25	5
Oxydemeton-methyl	fatty	0,010	110	21	20	4
Picoxystrobin	water containing	0,100	72	33	35	7
Picoxystrobin	water containing	0,010	74	46	35	7
Picoxystrobin	acidic	0,100	47	46	40	8
Picoxystrobin	acidic	0,010	59	59	30	6
Picoxystrobin	cereal (dry)	0,100	53	37	25	5
Picoxystrobin	cereal (dry)	0,010	56	19	20	4
Picoxystrobin	fatty	0,100	26	87	25	5
Picoxystrobin	fatty	0,010	43	68	20	4

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Pirimicarb	water containing	0,100	94	20	35	7
Pirimicarb	water containing	0,010	97	13	40	8
Pirimicarb	acidic	0,100	93	12	40	8
Pirimicarb	acidic	0,010	98	21	40	8
Pirimicarb	cereal (dry)	0,100	92	20	25	5
Pirimicarb	cereal (dry)	0,010	87	15	25	5
Pirimicarb	fatty	0,100	97	12	25	5
Pirimicarb	fatty	0,010	103	19	20	4
Promecarb	water containing	0,100	92	18	35	7
Promecarb	water containing	0,010	94	20	40	8
Promecarb	acidic	0,100	78	21	40	8
Promecarb	acidic	0,010	77	30	40	8
Promecarb	cereal (dry)	0,100	84	25	25	5
Promecarb	cereal (dry)	0,010	74	20	25	5
Promecarb	fatty	0,100	70	21	25	5
Promecarb	fatty	0,010	73	33	20	4
Propamocarb	water containing	0,100	73	30	35	7
Propamocarb	water containing	0,010	68	38	40	8
Propamocarb	acidic	0,100	51	17	35	7
Propamocarb	acidic	0,010	46	25	30	6
Propamocarb	cereal (dry)	0,100	82	26	20	4
Propamocarb	cereal (dry)	0,010	73	36	25	5
Propamocarb	fatty	0,100	70	20	20	4
Propamocarb	fatty	0,010	79	19	20	4
Propoxur	water containing	0,100	100	21	35	7
Propoxur	water containing	0,010	107	24	40	8
Propoxur	acidic	0,100	97	13	40	8
Propoxur	acidic	0,010	98	14	40	8
Propoxur	cereal (dry)	0,100	109	11	20	4
Propoxur	cereal (dry)	0,010	96	11	20	4
Propoxur	fatty	0,100	107	12	20	4

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Propoxur	fatty	0,010	99	16	15	3
Prosulfuron	water containing	0,100	101	21	35	7
Prosulfuron	water containing	0,010	97	17	40	8
Prosulfuron	acidic	0,100	66	37	40	8
Prosulfuron	acidic	0,010	69	36	40	8
Prosulfuron	cereal (dry)	0,100	98	20	25	5
Prosulfuron	cereal (dry)	0,010	86	17	25	5
Prosulfuron	fatty	0,100	89	14	25	5
Prosulfuron	fatty	0,010	103	16	20	4
Pymetrozin	water containing	0,100	79	35	35	7
Pymetrozin	water containing	0,010	86	34	40	8
Pymetrozin	acidic	0,100	49	32	40	8
Pymetrozin	acidic	0,010	46	34	40	8
Pymetrozin	cereal (dry)	0,100	89	20	25	5
Pymetrozin	cereal (dry)	0,010	91	14	25	5
Pymetrozin	fatty	0,100	91	10	25	5
Pymetrozin	fatty	0,010	89	17	20	4
Pyraclostrobin	water containing	0,100	70	49	35	7
Pyraclostrobin	water containing	0,010	61	70	35	7
Pyraclostrobin	acidic	0,100	32	67	40	8
Pyraclostrobin	acidic	0,010	37	112	35	7
Pyraclostrobin	cereal (dry)	0,100	51	49	25	5
Pyraclostrobin	cereal (dry)	0,010	43	90	20	4
Pyraclostrobin	fatty	0,100	20	73	20	4
Pyraclostrobin	fatty	0,010	38	58	20	4
Pyrimethanil	water containing	0,100	86	28	35	7
Pyrimethanil	water containing	0,010	80	44	40	8
Pyrimethanil	acidic	0,100	75	29	40	8
Pyrimethanil	acidic	0,010	68	37	40	8
Pyrimethanil	cereal (dry)	0,100	77	14	25	5
Pyrimethanil	cereal (dry)	0,010	78	17	25	5

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Pyrimethanil	fatty	0,100	37	51	25	5
Pyrimethanil	fatty	0,010	47	74	20	4
Spiroxamine	water containing	0,100	72	42	35	7
Spiroxamine	water containing	0,010	72	45	35	7
Spiroxamine	acidic	0,100	77	17	40	8
Spiroxamine	acidic	0,010	81	24	40	8
Spiroxamine	cereal (dry)	0,100	64	13	25	5
Spiroxamine	cereal (dry)	0,010	56	13	25	5
Spiroxamine	fatty	0,100	30	67	25	5
Spiroxamine	fatty	0,010	40	85	20	4
Tebuconazol	water containing	0,100	83	32	35	7
Tebuconazol	water containing	0,010	96	33	30	6
Tebuconazol	acidic	0,100	66	33	40	8
Tebuconazol	acidic	0,010	60	39	40	8
Tebuconazol	cereal (dry)	0,100	74	17	25	5
Tebuconazol	cereal (dry)	0,010	81	21	25	5
Tebuconazol	fatty	0,100	56	33	25	5
Tebuconazol	fatty	0,010	64	43	20	4
Tebufenozid	water containing	0,100	84	41	35	7
Tebufenozid	water containing	0,010	80	42	40	8
Tebufenozid	acidic	0,100	64	41	40	8
Tebufenozid	acidic	0,010	64	44	40	8
Tebufenozid	cereal (dry)	0,100	68	26	25	5
Tebufenozid	cereal (dry)	0,010	60	21	25	5
Tebufenozid	fatty	0,100	68	25	25	5
Tebufenozid	fatty	0,010	70	38	20	4
Thiabendazol	water containing	0,100	85	27	35	7
Thiabendazol	water containing	0,010	87	38	40	8
Thiabendazol	acidic	0,100	77	25	40	8
Thiabendazol	acidic	0,010	78	29	40	8
Thiabendazol	cereal (dry)	0,100	81	27	25	5

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Thiabendazol	cereal (dry)	0,010	79	12	25	5
Thiabendazol	fatty	0,100	70	46	25	5
Thiabendazol	fatty	0,010	73	52	20	4
Thiacloprid	water containing	0,100	77	57	35	7
Thiacloprid	water containing	0,010	79	53	40	8
Thiacloprid	acidic	0,100	87	28	40	8
Thiacloprid	acidic	0,010	80	27	40	8
Thiacloprid	cereal (dry)	0,100	92	27	25	5
Thiacloprid	cereal (dry)	0,010	82	18	25	5
Thiacloprid	fatty	0,100	91	18	25	5
Thiacloprid	fatty	0,010	96	35	20	4
Thifensulfuron-methyl	water containing	0,100	95	13	35	7
Thifensulfuron-methyl	water containing	0,010	90	13	40	8
Thifensulfuron-methyl	acidic	0,100	89	16	40	8
Thifensulfuron-methyl	acidic	0,010	96	12	35	7
Thifensulfuron-methyl	cereal (dry)	0,100	73	30	25	5
Thifensulfuron-methyl	cereal (dry)	0,010	80	31	25	5
Thifensulfuron-methyl	fatty	0,100	89	17	25	5
Thifensulfuron-methyl	fatty	0,010	89	14	20	4
Thiofanox	water containing	0,100	78	39	30	6
Thiofanox	water containing	0,010	78	18	25	5
Thiofanox	acidic	0,100	79	38	40	8
Thiofanox	acidic	0,010	80	45	35	7
Thiofanox	cereal (dry)	0,100	56	69	25	5
Thiofanox	cereal (dry)	0,010	70	75	25	5
Thiofanox	fatty	0,100	72	60	25	5
Thiofanox	fatty	0,010	52	101	20	4
Vamidothion	water containing	0,100	101	36	30	6
Vamidothion	water containing	0,010	98	26	35	7
Vamidothion	acidic	0,100	92	22	35	7
Vamidothion	acidic	0,010	93	36	35	7

Table B.1 *(continued)*

Pesticide	Matrix type	Spiked amount	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Vamidothion	cereal (dry)	0,100	95	20	25	5
Vamidothion	cereal (dry)	0,010	83	29	25	5
Vamidothion	fatty	0,100	77	48	25	5
Vamidothion	fatty	0,010	80	46	20	4

[a] X = recovery, V = relative standard deviation (all individual results with equal weigh); n = number of results

Table B.2 — Results of single laboratory validations (n approximately 18 000)

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
2,4-D	water containing	0,050	107	6	7	1
2,4-D	acidic	0,050	143	23	9	1
2,4-D	fatty	0,100	10	31	6	1
2,4-D	sugar containing	0,050	63	18	9	1
3,4,5-Trimethacarb	water containing	0,100	83	19	67	3
3,4,5-Trimethacarb	cereal (dry)	0,050	96	13	9	1
3,4,5-Trimethacarb	acidic	0,100	169	19	9	1
3,4,5-Trimethacarb	fatty	0,050	102	14	10	1
3,4,5-Trimethacarb	sugar containing	0,050	91	8	9	1
3-Hydroxycarbofuran	water containing	0,100	98	17	88	5
3-Hydroxycarbofuran	cereal (dry)	0,100	112	15	14	1
3-Hydroxycarbofuran	acidic	0,100	128	33	13	1
3-Hydroxycarbofuran	fatty	0,100	116	30	6	1
3-Hydroxycarbofuran	sugar containing	0,100	92	15	14	1
5-Hydroxy-clethodim-sulfon	water containing	0,100	92	49	12	1
5-Hydroxy-clethodim-sulfon	cereal (dry)	0,100	22	74	12	1
5-Hydroxy-clethodim-sulfon	acidic	0,100	116	18	10	1
5-Hydroxy-clethodim-sulfon	fatty	0,100	62	28	13	1
5-Hydroxy-clethodim-sulfon	sugar containing	0,100	122	19	13	1
5-Hydroxythiabendazol	water containing	0,010	110	20	5	1
5-Hydroxythiabendazol	cereal (dry)	0,010	98	14	5	1
5-Hydroxythiabendazol	acidic	0,010	57	28	3	1
5-Hydroxythiabendazol	fatty	0,010	124	22	5	1
5-Hydroxythiabendazol	sugar containing	0,010	124	22	5	1
Abamectin	water containing	0,025	60	26	17	2
Acephat	water containing	0,025	78	25	200	5
Acephat	cereal (dry)	0,050	88	22	45	2
Acephat	acidic	0,072	102	37	20	3

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Acephat	fatty	0,100	84	25	17	2
Acephat	sugar containing	0,100	87	20	14	1
Acetamiprid	water containing	0,025	85	16	121	6
Acetamiprid	acidic	0,025	78	34	4	1
Aldicarb	water containing	0,025	76	37	148	5
Aldicarb	cereal (dry)	0,030	90	27	28	2
Aldicarb	acidic	0,100	118	36	16	2
Aldicarb	fatty	0,100	76	46	17	2
Aldicarb	sugar containing	0,100	83	57	13	1
Aldicarb-sulfoxid	water containing	0,100	108	27	98	4
Aldicarb-sulfoxid	cereal (dry)	0,100	105	15	14	1
Aldicarb-sulfoxid	acidic	0,100	127	32	11	1
Aldicarb-sulfoxid	fatty	0,100	132	26	14	1
Aldicarb-sulfoxid	sugar containing	0,100	113	34	14	1
Aldoxycarb	water containing	0,099	94	22	101	5
Aldoxycarb	cereal (dry)	0,100	112	16	14	1
Aldoxycarb	acidic	0,100	126	28	13	1
Aldoxycarb	fatty	0,100	114	13	14	1
Aldoxycarb	sugar containing	0,100	89	11	14	1
Amidosulfuron	water containing	0,100	91	21	66	2
Amidosulfuron	cereal (dry)	0,050	57	18	9	1
Amidosulfuron	acidic	0,100	177	22	9	1
Amidosulfuron	fatty	0,050	119	16	9	1
Amidosulfuron	sugar containing	0,050	90	29	9	1
Atrazin	water containing	0,100	88	17	71	2
Atrazin	cereal (dry)	0,100	102	12	14	1
Atrazin	acidic	0,100	133	29	12	1
Atrazin	fatty	0,100	97	8	14	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Atrazin	sugar containing	0,100	81	15	14	1
Azoxystrobin	water containing	0,050	82	21	183	7
Azoxystrobin	cereal (dry)	0,050	77	29	46	2
Azoxystrobin	acidic	0,097	109	38	18	2
Azoxystrobin	fatty	0,100	90	18	17	2
Azoxystrobin	sugar containing	0,100	84	13	14	1
Barban	water containing	0,025	74	16	17	1
Benalaxyl	water containing	0,025	73	20	44	2
Benalaxyl	acidic	0,025	69	24	3	1
Bendiocarb	water containing	0,075	101	25	90	4
Bendiocarb	cereal (dry)	0,030	105	38	16	1
Bendiocarb	acidic	0,097	114	31	18	1
Bendiocarb	fatty	0,050	111	28	20	1
Bendiocarb	sugar containing	0,050	95	21	19	1
Bensulfuron-methyl	water containing	0,100	89	17	65	2
Bensulfuron-methyl	cereal (dry)	0,050	85	23	9	1
Bensulfuron-methyl	acidic	0,050	172	22	9	1
Bensulfuron-methyl	fatty	0,050	129	15	9	1
Bensulfuron-methyl	sugar containing	0,050	78	17	9	1
Bentazon	cereal (dry)	0,010	60	74	8	1
Boscalid	water containing	0,025	83	16	30	3
Boscalid	cereal (dry)	0,055	79	15	10	1
Boscalid	acidic	0,055	82	16	10	1
Boscalid	fatty	0,100	108	49	7	1
Boscalid	sugar containing	0,055	98	15	10	1
Bromoxynil	water containing	0,050	112	4	7	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Bromoxynil	cereal (dry)	0,030	20	172	18	2
Bromoxynil	acidic	0,050	120	22	9	1
Bromoxynil	fatty	0,050	71	17	9	1
Bromoxynil	sugar containing	0,050	88	16	9	1
Bromuconazol	water containing	0,025	72	19	42	1
Bromuconazol	acidic	0,025	69	27	3	1
Bupirimat	water containing	0,100	94	17	3	1
Buprofezin	water containing	0,025	68	23	41	1
Buprofezin	acidic	0,025	48	40	3	1
Butocarboxim	water containing	0,100	78	49	74	3
Butocarboxim	cereal (dry)	0,100	105	11	14	1
Butocarboxim	acidic	0,100	129	33	14	1
Butocarboxim	fatty	0,100	68	61	15	1
Butocarboxim	sugar containing	0,100	78	59	12	1
Butocarboxim-sulfoxid	water containing	0,100	113	24	70	2
Butocarboxim-sulfoxid	cereal (dry)	0,100	106	15	14	1
Butocarboxim-sulfoxid	acidic	0,100	120	20	10	1
Butocarboxim-sulfoxid	fatty	0,100	124	17	14	1
Butocarboxim-sulfoxid	sugar containing	0,100	109	21	14	1
Butoxycarboxim	water containing	0,100	94	20	72	2
Butoxycarboxim	cereal (dry)	0,100	114	13	14	1
Butoxycarboxim	acidic	0,100	116	25	9	1
Butoxycarboxim	fatty	0,100	110	10	14	1
Butoxycarboxim	sugar containing	0,100	95	8	14	1
Cadusafos	water containing	0,025	75	22	45	2
Cadusafos	acidic	0,025	58	52	3	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Carbaryl	water containing	0,025	90	20	212	8
Carbaryl	cereal (dry)	0,050	90	23	56	2
Carbaryl	acidic	0,093	109	34	31	3
Carbaryl	fatty	0,100	102	26	27	2
Carbaryl	sugar containing	0,100	100	12	24	1
Carbendazim	water containing	0,025	68	42	150	7
Carbendazim	cereal (dry)	0,050	92	38	28	2
Carbendazim	acidic	0,092	87	39	23	4
Carbendazim	fatty	0,100	101	15	17	2
Carbendazim	sugar containing	0,100	100	24	14	1
Carbetamid	water containing	0,025	85	14	17	1
Carbofuran	water containing	0,025	90	19	155	6
Carbofuran	cereal (dry)	0,100	109	18	26	2
Carbofuran	acidic	0,072	112	32	30	3
Carbofuran	fatty	0,100	111	18	27	2
Carbofuran	sugar containing	0,100	100	16	24	1
Carbofuran-3-keto	water containing	0,099	93	13	3	1
Carvone	water containing	0,099	60	23	3	1
Chlorfenvinphos	water containing	0,100	73	31	5	1
Chloridazon	water containing	0,025	84	16	17	1
Chlorpropham	water containing	0,100	40	17	5	1
Chlorpyrifos	water containing	0,100	87	14	3	1
Chlorsulfuron	water containing	0,100	89	21	67	2
Chlorsulfuron	cereal (dry)	0,050	49	24	9	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Chlorsulfuron	acidic	0,100	172	26	8	1
Chlorsulfuron	fatty	0,050	97	9	9	1
Chlorsulfuron	sugar containing	0,050	63	18	9	1
Cinidon-ethyl	water containing	0,025	54	25	17	1
Cinosulfuron	water containing	0,100	93	17	64	3
Cinosulfuron	cereal (dry)	0,050	94	15	9	1
Cinosulfuron	acidic	0,550	125	20	4	1
Cinosulfuron	fatty	0,050	59	13	10	1
Cinosulfuron	sugar containing	0,050	44	24	9	1
Clethodim	water containing	0,100	78	28	27	1
Clethodim	cereal (dry)	0,100	91	27	29	1
Clethodim	acidic	0,100	49	82	19	1
Clethodim	fatty	0,100	73	61	13	1
Clethodim	sugar containing	0,100	54	48	15	1
Clethodim-imin-sulfon	water containing	0,100	114	10	12	1
Clethodim-imin-sulfon	cereal (dry)	0,100	113	10	14	1
Clethodim-imin-sulfon	acidic	0,100	118	28	9	1
Clethodim-imin-sulfon	fatty	0,100	103	15	14	1
Clethodim-imin-sulfon	sugar containing	0,100	93	13	13	1
Clethodim-imin-sulfoxid	water containing	0,100	108	13	12	1
Clethodim-imin-sulfoxid	cereal (dry)	0,100	110	12	14	1
Clethodim-imin-sulfoxid	acidic	0,100	117	26	10	1
Clethodim-imin-sulfoxid	fatty	0,100	96	11	14	1
Clethodim-imin-sulfoxid	sugar containing	0,100	78	21	14	1
Clethodim-sulfon	water containing	0,100	134	15	12	1
Clethodim-sulfon	cereal (dry)	0,100	109	23	14	1
Clethodim-sulfon	acidic	0,100	128	22	10	1
Clethodim-sulfon	fatty	0,100	115	22	14	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Clethodim-sulfon	sugar containing	0,100	117	25	14	1
Clethodim-sulfoxid	water containing	0,100	138	4	5	1
Clethodim-sulfoxid	cereal (dry)	0,100	114	26	14	1
Clethodim-sulfoxid	acidic	0,100	127	11	7	1
Clethodim-sulfoxid	fatty	0,100	125	27	8	1
Clethodim-sulfoxid	sugar containing	0,100	168	14	12	1
Clofentezin	water containing	0,025	49	35	17	1
Cycloxydim	water containing	0,010	60	40	38	2
Cycloxydim	cereal (dry)	0,100	65	57	17	2
Cycloxydim	acidic	0,010	72	50	3	2
Cymoxanil	water containing	0,025	66	29	15	2
Cyproconazol	water containing	0,025	74	24	21	2
Cyprodinil	water containing	0,025	71	24	159	7
Cyprodinil	cereal (dry)	0,030	73	36	28	2
Cyprodinil	acidic	0,072	79	56	22	3
Cyprodinil	fatty	0,100	51	53	16	2
Cyprodinil	sugar containing	0,100	70	32	14	1
Cyromazin	water containing	0,025	19	71	8	1
Demeton-S-methyl	water containing	0,025	78	29	171	6
Demeton-S-methyl	cereal (dry)	0,050	78	25	46	2
Demeton-S-methyl	acidic	0,050	119	29	19	3
Demeton-S-methyl	fatty	0,100	84	63	12	2
Demeton-S-methyl	sugar containing	0,100	118	32	8	1
Demeton-S-methylsulfon	water containing	0,025	98	21	192	7
Demeton-S-methylsulfon	cereal (dry)	0,050	93	27	52	2

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Demeton-S-methylsulfon	acidic	0,050	97	24	18	5
Demeton-S-methylsulfon	fatty	0,100	123	15	16	2
Demeton-S-methylsulfon	sugar containing	0,100	93	11	14	1
Desmedipham	water containing	0,100	115	27	40	1
Desmethylformamido-pirimicarb	water containing	0,030	92	5	6	1
Desmethylformamido-pirimicarb	cereal (dry)	0,010	99	18	5	1
Desmethylformamido-pirimicarb	fatty	0,030	101	39	6	1
Desmethylformamido-pirimicarb	sugar containing	0,010	102	8	5	1
Desmethyl-pirimicarb	water containing	0,030	88	4	6	1
Desmethyl-pirimicarb	cereal (dry)	0,010	90	12	5	1
Desmethyl-pirimicarb	fatty	0,030	82	38	6	1
Desmethyl-pirimicarb	sugar containing	0,030	94	40	6	1
Diazinon	water containing	0,075	86	16	4	1
Dichlofluanid	water containing	0,025	34	98	13	1
Dichlorprop	cereal (dry)	0,010	29	145	9	1
Dicrotophos	water containing	0,025	82	17	41	1
Dicrotophos	acidic	0,025	74	84	3	1
Diethofencarb	water containing	0,025	82	18	48	2
Diethofencarb	acidic	0,025	76	26	3	1
Difenoconazol	water containing	0,025	53	49	43	1
Difenoconazol	acidic	0,025	68	16	3	1
Diflubenzuron	water containing	0,025	74	35	88	3

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Diflubenzuron	cereal (dry)	0,100	73	30	25	2
Diflubenzuron	acidic	0,100	69	34	27	2
Diflubenzuron	fatty	0,100	75	55	22	2
Diflubenzuron	sugar containing	0,100	83	27	23	1
Diflufenican	water containing	0,025	52	43	18	1
Diflufenican	cereal (dry)	0,010	69	48	9	1
Dimethachlor	water containing	0,025	76	20	17	1
Dimethoat	water containing	0,025	90	19	206	6
Dimethoat	cereal (dry)	0,050	93	22	46	2
Dimethoat	acidic	0,100	120	29	19	2
Dimethoat	fatty	0,100	101	19	17	2
Dimethoat	sugar containing	0,100	98	12	14	1
Dimethomorph	water containing	0,025	63	24	19	1
Diniconazol	water containing	0,025	59	37	40	2
Diphenylamin	water containing	0,025	64	36	35	1
Diuron	water containing	0,100	82	18	67	2
Diuron	cereal (dry)	0,050	94	10	9	1
Diuron	acidic	0,100	171	25	7	1
Diuron	fatty	0,050	101	12	9	1
Diuron	sugar containing	0,050	83	13	9	1
Epoxiconazol	water containing	0,025	64	27	37	1
Ethiofencarb	water containing	0,025	57	39	90	6
Ethiofencarb	cereal (dry)	0,100	64	31	14	1
Ethiofencarb	acidic	0,100	114	35	17	1
Ethiofencarb	fatty	0,100	81	44	8	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Ethiofencarb	sugar containing	0,100	47	32	8	1
Ethiofencarbsulfon	water containing	0,100	104	19	78	3
Ethiofencarbsulfon	cereal (dry)	0,100	120	22	14	1
Ethiofencarbsulfon	acidic	0,100	122	31	12	1
Ethiofencarbsulfon	fatty	0,100	119	16	14	1
Ethiofencarbsulfon	sugar containing	0,100	87	13	14	1
Ethiofencarb-sulfoxid	water containing	0,100	148	23	65	3
Ethiofencarb-sulfoxid	cereal (dry)	0,100	152	20	11	1
Ethiofencarb-sulfoxid	acidic	0,100	140	18	9	1
Ethiofencarb-sulfoxid	fatty	0,100	149	18	14	1
Ethiofencarb-sulfoxid	sugar containing	0,100	157	14	14	1
Ethion	water containing	0,025	70	26	15	1
Ethofumesat	water containing	0,025	75	19	15	1
Ethoprophos	water containing	0,025	54	28	15	1
Etofenprox	water containing	0,025	52	52	6	1
Famoxadon	water containing	0,025	48	51	24	2
Fenamiphos	water containing	0,025	80	24	13	1
Fenamiphos-sulfon	water containing	0,025	88	14	15	1
Fenamiphos-sulfoxid	water containing	0,025	101	22	15	1
Fenarimol	water containing	0,025	66	22	15	1
Fenazaquin	water containing	0,025	58	43	12	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Fenbuconazol	water containing	0,025	58	29	37	1
Fenbutatin-oxid	water containing	0,025	27	34	8	1
Fenfuram	water containing	0,025	61	44	14	1
Fenhexamid	water containing	0,025	81	30	207	6
Fenhexamid	cereal (dry)	0,050	79	25	33	2
Fenhexamid	acidic	0,100	100	38	15	2
Fenhexamid	fatty	0,100	92	9	16	2
Fenhexamid	sugar containing	0,100	93	13	14	1
Fenitrothion	water containing	0,025	74	35	8	1
Fenoxaprop	cereal (dry)	0,010	75	43	9	1
Fenoxycarb	water containing	0,025	73	26	192	6
Fenoxycarb	cereal (dry)	0,050	63	23	22	2
Fenoxycarb	acidic	0,097	91	42	20	2
Fenoxycarb	fatty	0,100	62	27	16	2
Fenoxycarb	sugar containing	0,100	69	17	14	1
Fenpropimorph	water containing	0,025	67	29	163	5
Fenpropimorph	cereal (dry)	0,030	51	47	34	2
Fenpropimorph	acidic	0,097	98	44	16	2
Fenpropimorph	fatty	0,100	21	19	13	1
Fenpropimorph	sugar containing	0,100	47	68	14	1
Fenpyroximat	water containing	0,025	58	34	41	2
Fenthion	water containing	0,025	72	19	15	1
Fenthion-sulfon	water containing	0,025	74	19	15	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Fenthion-sulfoxid	water containing	0,025	95	19	15	1
Fipronil	water containing	0,025	70	27	34	1
Flazasulfuron	water containing	0,100	77	33	64	2
Flazasulfuron	cereal (dry)	0,050	71	9	9	1
Flazasulfuron	acidic	0,075	144	35	10	1
Flazasulfuron	fatty	0,050	110	18	9	1
Flazasulfuron	sugar containing	0,050	70	8	9	1
Florasulam	water containing	0,010	90	3	5	1
Florasulam	cereal (dry)	0,010	56	46	17	2
Florasulam	fatty	0,010	81	11	5	1
Florasulam	sugar containing	0,010	132	24	5	1
Fluazifop	water containing	0,025	70	18	31	1
Fluazifop-butyl	water containing	0,025	55	27	12	1
Fluazifop-P-butyl	water containing	0,100	60	28	74	3
Fluazifop-P-butyl	cereal (dry)	0,100	57	23	14	1
Fluazifop-P-butyl	acidic	0,100	49	57	15	1
Fluazifop-P-butyl	fatty	0,100	27	28	14	1
Fluazifop-P-butyl	sugar containing	0,100	50	33	14	1
Fludioxonil	water containing	0,010	68	37	95	4
Fludioxonil	cereal (dry)	0,050	91	17	11	2
Fludioxonil	acidic	0,050	90	30	13	2
Fludioxonil	fatty	0,050	46	60	11	2
Fludioxonil	sugar containing	0,050	69	34	9	1
Flufenacet	water containing	0,025	68	33	24	2
Flufenoxuron	water containing	0,025	63	37	154	52

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Flufenoxuron	cereal (dry)	0,100	58	42	26	2
Flufenoxuron	acidic	0,100	32	107	23	2
Flufenoxuron	fatty	0,100	28	70	21	2
Flufenoxuron	sugar containing	0,100	65	35	24	1
Fluquinconazol	water containing	0,025	70	43	29	1
Fluroxypyr	cereal (dry)	0,010	48	85	11	1
Flurtamone	water containing	0,025	73	24	12	1
Flusilazol	water containing	0,025	61	33	13	1
Flutriafol	water containing	0,100	94	16	15	1
Flutriafol	cereal (dry)	0,100	77	48	14	1
Flutriafol	acidic	0,055	101	28	20	1
Flutriafol	fatty	0,055	87	38	20	1
Flutriafol	sugar containing	0,100	102	14	19	1
Fosthiazate	water containing	0,010	88	5	5	1
Fosthiazate	cereal (dry)	0,010	99	17	5	1
Fosthiazate	fatty	0,010	101	7	5	1
Fosthiazate	sugar containing	0,010	104	14	5	1
Furathiocarb	water containing	0,100	65	27	91	4
Furathiocarb	cereal (dry)	0,100	49	7	14	1
Furathiocarb	acidic	0,100	71	53	15	1
Furathiocarb	fatty	0,100	32	18	14	1
Furathiocarb	sugar containing	0,100	57	20	14	1
Halofenozid	water containing	0,025	84	16	33	1
Haloxyfop	water containing	0,055	104	27	10	1
Haloxyfop	cereal (dry)	0,100	25	8	5	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Haloxyfop	acidic	0,055	93	12	10	1
Haloxyfop	fatty	0,055	74	17	10	1
Haloxyfop	sugar containing	0,055	109	10	10	1
Haloxyfop-ethoxyethyl	water containing	0,100	68	24	67	2
Haloxyfop-ethoxyethyl	cereal (dry)	0,050	62	36	9	1
Haloxyfop-ethoxyethyl	acidic	0,075	84	28	10	1
Haloxyfop-ethoxyethyl	fatty	0,050	43	14	9	1
Haloxyfop-ethoxyethyl	sugar containing	0,050	45	31	9	1
Haloxyfop-methyl	water containing	0,100	65	20	67	2
Haloxyfop-methyl	cereal (dry)	0,050	58	20	9	1
Haloxyfop-methyl	acidic	0,075	104	30	10	1
Haloxyfop-methyl	fatty	0,050	32	31	9	1
Haloxyfop-methyl	sugar containing	0,050	54	26	9	1
Hexaconazol	water containing	0,025	60	19	29	1
Hexaflumuron	water containing	0,025	59	44	47	3
Hexaflumuron	cereal (dry)	0,050	70	37	9	1
Hexaflumuron	acidic	0,050	49	52	10	1
Hexaflumuron	fatty	0,050	40	18	9	1
Hexaflumuron	sugar containing	0,050	50	41	9	1
Hexazinon	water containing	0,025	79	13	11	1
Hexythiazox	water containing	0,025	62	33	35	3
Imazalil	water containing	0,025	68	40	176	8
Imazalil	cereal (dry)	0,050	66	36	46	2
Imazalil	acidic	0,093	124	31	21	3
Imazalil	fatty	0,100	73	24	16	2
Imazalil	sugar containing	0,100	73	27	14	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Imidacloprid	water containing	0,025	87	25	159	8
Imidacloprid	cereal (dry)	0,100	110	18	26	2
Imidacloprid	acidic	0,100	109	32	26	2
Imidacloprid	fatty	0,100	101	18	26	2
Imidacloprid	sugar containing	0,100	94	23	24	1
Imidacloprid hydroxid	water containing	0,100	82	16	15	1
Imidacloprid hydroxid	cereal (dry)	0,100	86	20	13	1
Imidacloprid hydroxid	acidic	0,100	90	8	10	1
Imidacloprid hydroxid	fatty	0,100	88	13	9	1
Imidacloprid hydroxid	sugar containing	0,100	81	21	10	1
Imidacloprid olefin	water containing	0,100	100	11	5	1
Imidacloprid olefin	cereal (dry)	0,100	80	15	5	1
Imidacloprid olefin	acidic	0,100	112	13	5	1
Imidacloprid olefin	fatty	0,100	101	20	4	1
Imidacloprid olefin	sugar containing	0,100	109	14	5	1
Indoxacarb	water containing	0,010	57	52	42	4
Indoxacarb	cereal (dry)	0,100	68	32	15	1
Indoxacarb	acidic	0,100	16	292	8	2
Indoxacarb	fatty	0,100	42	38	15	1
Indoxacarb	sugar containing	0,100	54	37	14	1
Iodosulfuron-methyl	water containing	0,010	91	10	5	1
Iodosulfuron-methyl	cereal (dry)	0,010	83	8	5	1
Iodosulfuron-methyl	acidic	0,010	171	3	3	1
Iodosulfuron-methyl	fatty	0,010	107	9	5	1
Iodosulfuron-methyl	sugar containing	0,010	127	20	5	1
Ioxynil	water containing	0,050	106	4	7	1
Ioxynil	cereal (dry)	0,010	32	96	20	2
Ioxynil	acidic	0,050	114	17	9	1
Ioxynil	fatty	0,050	88	15	9	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Ioxynil	sugar containing	0,050	93	9	9	1
Iprovalicarb	water containing	0,050	94	20	32	4
Iprovalicarb	cereal (dry)	0,100	86	12	14	1
Iprovalicarb	acidic	0,100	106	33	10	1
Iprovalicarb	fatty	0,100	91	10	15	1
Iprovalicarb	sugar containing	0,100	86	10	14	1
Isoproturon	water containing	0,100	85	18	77	3
Isoproturon	cereal (dry)	0,050	93	24	26	2
Isoproturon	acidic	0,100	134	24	15	1
Isoproturon	fatty	0,100	108	9	14	1
Isoproturon	sugar containing	0,100	99	9	14	1
Isoxaflutole	water containing	0,100	91	40	71	2
Isoxaflutole	cereal (dry)	0,100	77	27	12	1
Isoxaflutole	acidic	0,100	96	40	15	2
Isoxaflutole	fatty	0,100	84	29	14	1
Isoxaflutole	sugar containing	0,050	109	15	9	1
Kresoxim-methyl	water containing	0,025	69	28	14	2
Lenacil	water containing	0,025	74	22	13	1
Linuron	water containing	0,100	81	21	90	5
Linuron	cereal (dry)	0,100	96	12	14	1
Linuron	acidic	0,100	126	40	16	1
Linuron	fatty	0,100	86	14	14	1
Linuron	sugar containing	0,100	89	18	14	1
Lufenuron	water containing	0,025	62	48	76	3
Lufenuron	acidic	0,010	54	139	3	1
MCPA	water containing	0,050	116	9	7	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
MCPA	cereal (dry)	0,030	15	60	4	1
MCPA	acidic	0,050	139	21	9	1
MCPA	fatty	0,050	16	19	9	1
MCPA	sugar containing	0,050	81	14	9	1
Mecarbam	water containing	0,025	59	26	12	1
Mecoprop-P	water containing	0,050	110	13	7	1
Mecoprop-P	cereal (dry)	0,050	8	91	9	1
Mecoprop-P	acidic	0,050	137	16	9	1
Mecoprop-P	fatty	0,050	36	24	9	1
Mecoprop-P	sugar containing	0,050	83	15	9	1
Mepanipyrim	water containing	0,025	79	20	16	1
Mepronil	water containing	0,025	76	23	35	2
Mesotrion	water containing	0,055	45	18	10	1
Mesotrion	acidic	0,100	175	22	3	1
Mesotrion	fatty	0,055	34	26	10	1
Metalaxyl	water containing	0,025	88	21	196	7
Metalaxyl	cereal (dry)	0,050	82	24	46	7
Metalaxyl	acidic	0,100	111	31	19	7
Metalaxyl	fatty	0,100	100	10	17	7
Metalaxyl	sugar containing	0,100	91	12	14	7
Metamitron	water containing	0,050	65	30	82	4
Metamitron	cereal (dry)	0,050	96	14	9	1
Metamitron	acidic	0,100	66	25	9	1
Metamitron	fatty	0,050	94	14	9	1
Metamitron	sugar containing	0,050	79	20	9	1
Metconazol	water containing	0,025	47	42	31	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Methabenzthiazuron	water containing	0,025	75	34	13	1
Methamidophos	water containing	0,025	68	29	193	5
Methamidophos	cereal (dry)	0,050	80	22	46	2
Methamidophos	acidic	0,097	104	30	20	2
Methamidophos	fatty	0,100	91	24	17	2
Methamidophos	sugar containing	0,100	88	22	14	1
Methidathion	water containing	0,025	66	33	31	1
Methiocarb (Mercaptodimethur)	water containing	0,050	86	24	188	6
Methiocarb (Mercaptodimethur)	cereal (dry)	0,050	77	24	45	2
Methiocarb (Mercaptodimethur)	acidic	0,100	93	38	28	2
Methiocarb (Mercaptodimethur)	fatty	0,100	82	35	25	2
Methiocarb (Mercaptodimethur)	sugar containing	0,100	82	25	24	1
Methiocarbsulfon	water containing	0,099	93	24	22	4
Methiocarbsulfon	cereal (dry)	0,055	110	8	10	1
Methiocarbsulfon	acidic	0,055	123	15	10	1
Methiocarbsulfon	fatty	0,010	113	29	9	1
Methiocarbsulfon	sugar containing	0,055	105	9	10	1
Methiocarb-sulfoxid	water containing	0,099	106	10	12	3
Methomyl	water containing	0,025	94	35	145	8
Methomyl	cereal (dry)	0,010	116	31	24	2
Methomyl	acidic	0,100	120	26	13	2
Methomyl	fatty	0,050	181	26	11	2
Methomyl	sugar containing	0,100	101	10	12	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Methoxyfenozide	water containing	0,025	84	24	87	5
Methoxyfenozide	cereal (dry)	0,100	86	10	27	2
Methoxyfenozide	acidic	0,097	94	17	30	2
Methoxyfenozide	fatty	0,100	98	34	23	2
Methoxyfenozide	sugar containing	0,100	93	16	25	1
Metobromuron	water containing	0,050	105	15	12	2
Metobromuron	cereal (dry)	0,050	92	14	10	1
Metobromuron	acidic	0,055	103	7	10	1
Metobromuron	fatty	0,055	117	35	10	1
Metobromuron	sugar containing	0,055	104	8	10	1
Metolachlor	water containing	0,100	83	16	75	3
Metolachlor	cereal (dry)	0,100	83	11	14	1
Metolachlor	acidic	0,100	134	34	16	1
Metolachlor	fatty	0,100	82	19	15	1
Metolachlor	sugar containing	0,100	89	13	14	1
Metosulam	water containing	0,025	97	19	9	1
Metsulfuron-methyl	water containing	0,100	91	18	61	2
Metsulfuron-methyl	cereal (dry)	0,010	59	40	21	2
Metsulfuron-methyl	acidic	0,097	151	30	8	1
Metsulfuron-methyl	fatty	0,050	99	15	10	1
Metsulfuron-methyl	sugar containing	0,050	79	11	9	1
Monocrotophos	water containing	0,025	91	18	177	6
Monocrotophos	cereal (dry)	0,050	98	13	34	2
Monocrotophos	acidic	0,097	120	36	18	2
Monocrotophos	fatty	0,100	101	17	16	2
Monocrotophos	sugar containing	0,100	87	14	14	1
Monolinuron	water containing	0,025	90	16	25	3
Monolinuron	cereal (dry)	0,055	95	15	10	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Monolinuron	acidic	0,055	100	7	10	1
Monolinuron	fatty	0,055	114	29	10	1
Monolinuron	sugar containing	0,055	98	9	10	1
Myclobutanil	water containing	0,025	77	29	35	1
Nicosulfuron	water containing	0,100	85	23	65	2
Nicosulfuron	cereal (dry)	0,050	42	18	8	1
Nicosulfuron	acidic	0,100	162	26	7	1
Nicosulfuron	fatty	0,050	85	18	9	1
Nicosulfuron	sugar containing	0,050	76	43	9	1
Nitenpyram	water containing	0,025	51	69	30	2
Ofurace	water containing	0,025	82	21	29	1
Omethoat	water containing	0,025	90	21	182	6
Omethoat	cereal (dry)	0,050	96	21	46	2
Omethoat	acidic	0,100	120	30	15	2
Omethoat	fatty	0,100	108	16	16	2
Omethoat	sugar containing	0,100	82	20	14	1
Oxadixyl	water containing	0,010	97	14	3	1
Oxamyl	water containing	0,025	96	22	118	5
Oxamyl	cereal (dry)	0,100	119	21	16	2
Oxamyl	acidic	0,100	125	37	17	2
Oxamyl	fatty	0,100	103	16	17	2
Oxamyl	sugar containing	0,100	95	8	14	1
Oxydemetonmethyl	water containing	0,025	100	29	139	7
Oxydemetonmethyl	cereal (dry)	0,030	105	26	28	2
Oxydemetonmethyl	acidic	0,100	124	17	11	2
Oxydemetonmethyl	fatty	0,100	116	16	17	2

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Oxydemetonmethyl	sugar containing	0,100	98	12	14	1
Penconazol	water containing	0,025	70	35	31	1
Pencycuron	water containing	0,025	64	27	29	1
Phenmedipham	water containing	0,100	114	31	39	1
Phorate	water containing	0,025	46	29	11	1
Phorate sulfoxid	water containing	0,100	123	7	5	1
Phorate sulfoxid	cereal (dry)	0,100	140	10	5	1
Phorate sulfoxid	acidic	0,100	111	4	5	1
Phorate sulfoxid	fatty	0,100	107	4	5	1
Phorate sulfoxid	sugar containing	0,025	108	3	5	1
Phosalon	water containing	0,099	11	42	3	1
Picoxystrobin	water containing	0,025	63	33	22	3
Picoxystrobin	cereal (dry)	0,010	56	49	17	2
Picoxystrobin	acidic	0,100	59	37	6	1
Picoxystrobin	fatty	0,100	50	25	6	1
Picoxystrobin	sugar containing	0,100	77	2	5	1
Pirimicarb	water containing	0,025	85	26	190	5
Pirimicarb	cereal (dry)	0,050	86	16	34	2
Pirimicarb	acidic	0,100	116	30	14	2
Pirimicarb	fatty	0,100	93	17	17	2
Pirimicarb	sugar containing	0,100	87	17	14	1
Primisulfuron-methyl	cereal (dry)	0,050	65	89	9	1
Primisulfuron-methyl	acidic	0,050	48	26	9	1
Primisulfuron-methyl	fatty	0,050	113	12	9	1
Primisulfuron-methyl	sugar containing	0,050	78	13	9	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Profenofos	water containing	0,025	66	23	29	1
Promecarb	water containing	0,100	86	20	99	4
Promecarb	cereal (dry)	0,100	96	16	24	1
Promecarb	acidic	0,100	101	33	24	1
Promecarb	fatty	0,100	97	35	24	1
Promecarb	sugar containing	0,100	94	13	24	1
Propamocarb	water containing	0,025	52	40	119	6
Propamocarb	cereal (dry)	0,100	56	46	14	1
Propamocarb	acidic	0,072	65	33	12	1
Propamocarb	fatty	0,100	68	32	15	1
Propamocarb	sugar containing	0,100	35	13	14	1
Propargit	water containing	0,025	57	40	37	1
Propiconazol	water containing	0,025	62	22	30	1
Propoxur	water containing	0,025	87	19	203	6
Propoxur	cereal (dry)	0,050	89	23	56	2
Propoxur	acidic	0,100	116	28	27	2
Propoxur	fatty	0,100	112	16	27	2
Propoxur	sugar containing	0,100	99	13	24	1
Propyzamid	water containing	0,025	77	18	15	1
Prosulfuron	water containing	0,100	98	21	65	2
Prosulfuron	cereal (dry)	0,050	89	22	9	1
Prosulfuron	acidic	0,093	140	34	11	1
Prosulfuron	fatty	0,050	71	17	10	1
Prosulfuron	sugar containing	0,050	83	36	9	1
Pymetrozin	water containing	0,050	72	30	119	6

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Pymetrozin	cereal (dry)	0,100	94	14	24	1
Pymetrozin	acidic	0,100	71	32	26	1
Pymetrozin	fatty	0,100	80	17	24	1
Pymetrozin	sugar containing	0,100	65	27	24	1
Pyraclostrobin	water containing	0,025	82	36	22	3
Pyraclostrobin	cereal (dry)	0,010	63	49	17	2
Pyraclostrobin	acidic	0,100	33	22	6	1
Pyraclostrobin	fatty	0,100	31	33	6	1
Pyraclostrobin	sugar containing	0,100	68	3	5	1
Pyrazophos	water containing	0,025	62	19	30	1
Pyridaben	water containing	0,025	59	45	37	1
Pyridaphenthion	water containing	0,025	76	17	31	1
Pyridat	water containing	0,025	59	70	63	3
Pyridat	cereal (dry)	0,075	15	88	8	1
Pyridat	acidic	0,075	42	57	10	1
Pyridat	fatty	0,050	37	26	9	1
Pyridat	sugar containing	0,050	28	65	9	1
Pyrifenox	water containing	0,025	61	21	13	1
Pyrimethanil	water containing	0,025	81	20	191	6
Pyrimethanil	cereal (dry)	0,050	69	27	34	2
Pyrimethanil	acidic	0,100	127	32	19	2
Pyrimethanil	fatty	0,100	67	17	17	2
Pyrimethanil	sugar containing	0,100	73	25	14	1
Pyriproxyfen	water containing	0,025	62	25	31	1
Quinmerac	water containing	0,050	99	40	9	2

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Quinmerac	cereal (dry)	0,075	6	7	4	1
Quinmerac	acidic	0,100	130	27	13	1
Quinmerac	fatty	0,050	7	18	9	1
Quinmerac	sugar containing	0,100	73	17	14	1
Quizalofop-ethyl	water containing	0,100	61	22	65	2
Quizalofop-ethyl	cereal (dry)	0,050	59	33	9	1
Quizalofop-ethyl	acidic	0,050	74	32	9	1
Quizalofop-ethyl	fatty	0,075	30	37	10	1
Quizalofop-ethyl	sugar containing	0,050	41	36	9	1
Rimsulfuron	water containing	0,100	83	24	66	3
Rimsulfuron	cereal (dry)	0,010	71	37	21	2
Rimsulfuron	acidic	0,075	73	62	8	1
Rimsulfuron	fatty	0,010	174	24	3	1
Rimsulfuron	sugar containing	0,050	55	26	9	1
Spinosad	water containing	0,025	50	32	16	2
Spiroxamin	water containing	0,025	69	32	120	5
Spiroxamin	cereal (dry)	0,050	68	43	27	2
Spiroxamin	acidic	0,100	99	38	11	1
Spiroxamin	fatty	0,100	55	22	15	1
Spiroxamin	sugar containing	0,100	64	27	14	1
Tebuconazol	water containing	0,025	75	24	195	6
Tebuconazol	cereal (dry)	0,050	75	23	56	2
Tebuconazol	acidic	0,100	86	56	29	2
Tebuconazol	fatty	0,100	87	38	25	2
Tebuconazol	sugar containing	0,100	91	25	24	1
Tebufenozid	water containing	0,025	86	20	124	7
Tebufenozid	cereal (dry)	0,100	76	19	16	2
Tebufenozid	acidic	0,097	101	50	18	2

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Tebufenozid	fatty	0,100	90	22	17	2
Tebufenozid	sugar containing	0,100	83	15	14	1
Tebufenpyrad	water containing	0,025	64	36	29	1
Teflubenzuron	water containing	0,025	77	36	53	3
Teflubenzuron	cereal (dry)	0,100	66	31	19	1
Teflubenzuron	acidic	0,100	34	60	19	1
Teflubenzuron	fatty	0,100	43	92	16	1
Teflubenzuron	sugar containing	0,100	55	51	16	1
Terbuthylazin	water containing	0,025	75	21	15	1
Tetraconazol	water containing	0,025	64	32	39	3
Thiabendazol	water containing	0,025	70	31	144	8
Thiabendazol	cereal (dry)	0,050	94	23	29	2
Thiabendazol	acidic	0,100	109	27	19	2
Thiabendazol	fatty	0,100	108	12	17	2
Thiabendazol	sugar containing	0,100	86	8	14	1
Thiacloprid	water containing	0,050	84	26	111	7
Thiacloprid	cereal (dry)	0,100	105	17	16	2
Thiacloprid	acidic	0,100	129	29	17	2
Thiacloprid	fatty	0,100	111	17	17	2
Thiacloprid	sugar containing	0,100	84	14	14	1
Thiamethoxam	water containing	0,040	82	20	23	5
Thiamethoxam	cereal (dry)	0,010	97	18	9	1
Thiamethoxam	acidic	0,100	101	14	11	1
Thiamethoxam	fatty	0,055	101	12	10	1
Thiamethoxam	sugar containing	0,055	97	27	10	1
Thifensulfuron-methyl	water containing	0,050	110	9	7	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Thifensulfuron-methyl	cereal (dry)	0,050	57	15	9	1
Thifensulfuron-methyl	acidic	0,093	173	17	9	1
Thifensulfuron-methyl	fatty	0,050	92	13	10	1
Thifensulfuron-methyl	sugar containing	0,050	81	14	9	1
Thiodicarb	water containing	0,050	68	47	117	6
Thiodicarb	cereal (dry)	0,010	42	91	17	2
Thiodicarb	acidic	0,100	70	26	18	2
Thiodicarb	fatty	0,010	20	236	5	2
Thiodicarb	sugar containing	0,100	92	9	14	1
Thiofanox	water containing	0,100	68	42	58	4
Thiofanox	cereal (dry)	0,100	90	17	14	1
Thiofanox	acidic	0,100	117	38	14	1
Thiofanox	fatty	0,100	78	60	12	1
Thiofanox	sugar containing	0,100	96	39	9	1
Thiofanox-sulfon	water containing	0,010	90	5	5	1
Thiofanox-sulfon	cereal (dry)	0,030	97	41	6	1
Thiofanox-sulfon	fatty	0,050	114	48	9	1
Thiofanox-sulfon	sugar containing	0,030	101	41	6	1
Thiofanox-sulfoxid	water containing	0,050	125	34	7	1
Thiofanox-sulfoxid	cereal (dry)	0,050	96	55	8	1
Thiofanox-sulfoxid	fatty	0,100	89	81	7	1
Thiofanox-sulfoxid	sugar containing	0,050	160	44	9	1
Thiophanat-methyl	water containing	0,025	20	112	29	3
Thiophanat-methyl	cereal (dry)	0,100	27	68	10	1
Thiophanat-methyl	acidic	0,100	72	47	14	1
Thiophanat-methyl	fatty	0,100	71	54	7	1
Thiophanat-methyl	sugar containing	0,100	51	41	9	1
Tolylfluanid	water containing	0,025	63	64	16	2

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Triadimefon	water containing	0,025	79	19	17	2
Triasulfuron	water containing	0,100	84	25	65	2
Triasulfuron	cereal (dry)	0,050	103	15	9	1
Triasulfuron	acidic	0,100	147	34	5	1
Triasulfuron	fatty	0,050	104	10	9	1
Triasulfuron	sugar containing	0,050	72	10	9	1
Triazophos	water containing	0,025	72	24	28	1
Tribenuron-methyl	cereal (dry)	0,010	53	67	20	2
Tribenuron-methyl	fatty	0,050	97	11	9	1
Tribenuron-methyl	sugar containing	0,050	13	142	9	1
Trichlorfon	water containing	0,055	87	13	10	1
Trichlorfon	cereal (dry)	0,055	99	21	10	1
Trichlorfon	acidic	0,055	111	6	10	1
Trichlorfon	fatty	0,100	96	26	5	1
Trichlorfon	sugar containing	0,055	104	9	10	1
Trifloxystrobin	water containing	0,025	60	44	20	2
Triflumizol	water containing	0,025	42	55	25	1
Triflumuron	water containing	0,025	62	36	68	3
Triflumuron	cereal (dry)	0,050	62	35	29	3
Triflumuron	acidic	0,050	38	48	29	1
Triflumuron	fatty	0,100	55	77	22	1
Triflumuron	sugar containing	0,050	63	33	29	1
Triflusulfuron-methyl	water containing	0,050	101	10	7	1
Triflusulfuron-methyl	cereal (dry)	0,050	106	18	9	1
Triflusulfuron-methyl	acidic	0,050	119	34	9	1

Table B.2 *(continued)*

Pesticide	Matrix type	Spiked amount (median)	Recovery [a]			Number of labs
		mg/kg	X %	V %	n	
Triflusulfuron-methyl	sugar containing	0,075	74	30	8	1
Triforin	water containing	0,025	81	27	45	4
Triforin	cereal (dry)	0,055	86	13	10	1
Triforin	acidic	0,055	95	26	10	1
Triforin	fatty	0,100	113	46	7	1
Triforin	sugar containing	0,055	112	16	10	1
Vamidothion	water containing	0,100	83	39	72	3
Vamidothion	cereal (dry)	0,100	111	15	14	1
Vamidothion	acidic	0,100	121	29	14	1
Vamidothion	fatty	0,100	73	43	15	1
Vamidothion	sugar containing	0,100	77	46	13	1

[a] X = recovery, V = relative standard deviation (all individual results with equal weigh); n = number of results

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