INTERNATIONAL STANDARD

ISO 14181

First edition
2000-09-15

Animal feeding stuffs — Determination of residues of organochlorine pesticides — Gas chromatographic method

Aliments des animaux — Détermination des résidus de pesticides organochlorés — Méthode par chromatographie en phase gazeuse

Reference number
ISO 14181:2000(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14181 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 10, *Animal feeding stuffs*.

Annexes A and B of this International Standard are for information only.

Animal feeding stuffs — Determination of residues of organochlorine pesticides — Gas chromatographic method

1 Scope

This International Standard specifies a gas chromatographic method for the determination of residues of organochlorine pesticides in animal feeding stuffs.

The method is applicable to animal feeding stuffs containing residues of one or more of the following organochlorine pesticides and some of their isomers and degradation products: aldrin, *op'*-DDE, *pp'*-DDE, *op'*-DDT, *pp'*-DDT, dieldrin, endosulfan, endrin, HCB, α-HCH (α-BHC), β-HCH (β-BHC), γ-HCH (γ-BHC), δ-HCH (δ-BHC), heptachlor, heptachlor epoxide, *op'*-TDE (*op'*-DDD), *pp'*-TDE (*pp'*-DDD) and methoxychlor.

The lower determination limit for these organochlorine pesticides is 0,005 μg/g. However, the lower determination limit is 0,01 μg/g for *op'*-DDT and *pp'*-DDT, and 0,05 μg/g for methoxychlor.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative documents referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 6498, *Animal feeding stuffs — Preparation of test samples.*

3 Principle

A test portion is extracted with acetone. The filtered extract is diluted with water and saturated sodium chloride solution. The pesticides are partitioned into dichloromethane. The concentrated extract is purified on a chromatographic column of 10 % water-deactivated silica gel. The pesticide residues are determined by gas chromatography using an electron capture detector or a mass selective detector.

4 Reagents and materials

Use only reagents of recognized analytical grade and with a purity suitable for pesticide residue analysis.

Check the purity of the reagents by performing a blank test under the same conditions as used in the method. The chromatogram should not show any interfering impurity.

WARNING — Some of the organic solvents are suspected carcinogens. Use with care.

4.1 Water, complying with at least grade 3 in accordance with ISO 3696.

4.2 Hexane.

4.3 Acetone.

4.4 Dichloromethane.

4.5 Silica gel 60, with a mass fraction of water of 10 %.

Activate silica gel 60, particle size 63 μm to 200 μm, at 130 °C overnight, then cool in a desiccator. After cooling to room temperature, pour the silica gel into an airtight glass container and add sufficient distilled water to bring the final mass fraction of water to 10 %. Shake the container mechanically or by hand vigorously for 30 s and allow to stand for 30 min with occasional shaking. After 30 min the silica gel is ready for use. It should not be stored for more than 6 h.

4.6 Eluting solvent: dichloromethane in hexane, 20 % (by volume).

Mix 1 volume of dichloromethane with 4 volumes of hexane.

4.7 Inert gas, for example nitrogen.

4.8 Sodium sulfate, anhydrous.

4.9 Sodium chloride, saturated solution.

4.10 Pesticide reference standards, as follows:

— aldrin [(1*R*,4*S*,4*aS*,*5S*,8*R*,8*aR*)-1,2,3,4,10,10-hexachloro-1,4,4*a*,5,8,8*a*-hexahydro-1,4:5,8-dimethano-naphthalene];

— *op'*-DDE [*o,p'*-(1,1-dichloro-2,2-bis(4-chlorophenyl) ethylene)];

— *pp'*-DDE [*p,p'*-(1,1-dichloro-2,2-bis(4-chlorophenyl) ethylene)];

— *op'*-DDT [*o,p'*-(1,1,1-trichloro-2,2-bis(4-chlorophenyl) ethane)];

— *pp'*-DDT [*p,p'*-(1,1,1-trichloro-2,2-bis(4-chlorophenyl) ethane)];

— dieldrin [(1*R*,4*S*,4*aS*,5*R*,6*R*,7*S*,8*S*,8*aR*)-1,2,3,4,10,10-hexachloro-1,4,4*a*,5,6,7,8,8*a*-octahydro-6,7epoxy-1,4:5,8-dimethanonaphthalene];

— endosulfan (6,7,8,9,10,10-hexachloro-1,5,5*a*,6,9,9*a*-hexahydro-6,9-methano-2,4,3-benzodioxathiepin 3-oxide);

— endrin [(1*R*,4*S*,4*aS*,5*S*,6*S*,7*R*,8*R*,8*aR*)-1,2,3,4,10,10-hexachloro-1,4,4*a*,5,6,7,8,8*a*-octahydro-6,7-epoxy-1,4:5,8-dimethanonaphthalene];

— HCB (hexachlorobenzene);

— α-HCH (α-BHC) (α-1,2,3,4,5,6-hexachlorocyclohexane);

— β-HCH (β-BHC) (β-1,2,3,4,5,6-hexachlorocyclohexane);

— γ-HCH (γ-BHC; lindane) (γ-1,2,3,4,5,6-hexachlorocyclohexane);

— δ-HCH (δ-BHC) (δ-1,2,3,4,5,6-hexachlorocyclohexane);

— heptachlor (1,4,5,6,7,8,8-heptachloro-3*a*,4,7,7*a*-tetrahydro-4,7-methanoindene);

— heptachlor epoxide (heptachloroepoxy-tetrahydromethanoindene);

— *op'*-TDE (*op*-DDD) [*o,p'*-1,1-dichloro-2,2-bis(4-chlorophenyl) ethane];

— *pp'*-TDE (*pp'*-DDD) [*p,p'*-1,1-dichloro-2,2-bis(4-chlorophenyl) ethane];

— methoxychlor [1,1,1-trichloro-2,2-bis(4-methoxyphenyl) ethane].

4.11 Internal standard: mirex (1,3,5-tribromobenzene) or pentachloronitrobenzene.

4.12 Pesticide standard and internal standard solutions.

4.12.1 Stock solutions, of concentration 1 000 μg/ml.

Prepare a stock solution of each pesticide reference standard (4.10) and of the internal standard (4.11) as follows.

Weigh, to the nearest 0,1 mg, a mass of a pesticide reference standard (4.10) or the internal standard (4.11) which will result in a solution with a content of reference standard or internal standard of 1 000 μg/ml. While weighing, observe the cleanness of the standard matrial. Transfer the weighed mass into a volumetric flask, dissolve in hexane or in an other solvent such as toluene or iso octane. However, dissolve β-HCH in acetone. Dilute to volume with the same solvent and mix well.

These solutions are stable for 6 months when stored at 4 °C in the dark.

4.12.2 Intermediate solutions, of concentration 10 μg/ml.

Pipette 1 ml of each stock solution (4.12.1) into individual 100 ml volumetric flasks. Dilute to volume with hexane. The solutions are stable for at least 3 months when stored at 4 °C in the dark.

NOTE The stability of properly stored pesticide standards is very widely known. Investigations have shown that all neat pesticide standards tested are stable for 15 years when stored at –18 °C and that stock solutions of pesticide standards in toluene of 1mg/ml are stable for at least 3 years when stored at –18 °C.

The following recommended practice may be used for longer storage. Transfer portions of the prepared standard solutions to amber vials with PTFE-lined screwcaps. Weigh the vials and store at –20 °C. When needed, remove a vial from the freezer, bring to room temperature and weigh. If the accumulated loss in mass due to evaporation is 10 % or more of the prefrozen net mass, discard the vial. Weigh and refreeze stock standards and intermediate solutions that are in use for more than 3 months (usually in 25 ml vials). Otherwise the prepared standard solutions (usually in 2 ml vials) may be stored at 4 °C and shall be discarded after 3 months.

4.12.3 Working solution, of concentration 0,05 μg/ml.

Pipette a 0,5 ml of each intermediate solution (4.12.2) into a 100 ml volumetric flask and dilute to volume with hexane. The solution is stable for 1 month when stored at 4°C in the dark (see 4.12.2).

5 Apparatus

Before use, wash all glassware thoroughly with detergent free of interfering substances. Rinse with water, then with acetone and dry.

Avoid the use of plastics containers and do not lubricate the stopcocks with grease, otherwise impurities could be introduced into the solvents.

Usual laboratory equipment and, in particular, the following.

5.1 Separating funnels, of 500 ml capacity with polytetrafluoroethylene (PTFE) stopcocks and stoppers.

5.2 Filtering flasks, of 500 ml capacity.

5.3 Büchner funnel, of porcelain, with internal diameter 90 mm.

5.4 Graduated tubes, of 10 ml capacity, with polytetrafluoroethylene (PTFE) stoppers.

5.5 Glass chromatographic tube, about 300 mm in length, 8 mm to 10 mm internal diameter, with coarse fritted plate of porosity grade P 100 (pore size index 40 μm to 100 μm [2]) or glass wool plug.

5.6 Rotary vacuum evaporator, provided with round-bottom flasks of 100 ml and 500 ml capacities and a water bath at 40 °C ± 2 °C.

5.7 Mechanical shaker or **high-speed blender**.

5.8 Gas chromatographic system consisting of:

— splitless or on-column injection system;

— column;

— electron capture detector or mass selective detector;

— electrometer;

— mV recorder or integrator;

— data handling software and computer system.

Each injection port, column oven and detector shall be provided with an independent heating device controlled to the nearest 0,1 °C.

The chromatographic system shall be adjusted and the parameters optimized according to the characteristics of the instrument used.

5.8.1 Injection device

An autosampler or any other adequate injection device may be used. For manual injections, use a microsyringe of 1 μl to 5 μl capacity with a needle length suitable for the mode of injection (splitless or on-column).

Before injecting the solution into the gas chromatograph, rinse the syringe 10 times with pure solvent, then 5 times with solution. After injection, rinse the syringe 5 times with pure solvent.

5.8.2 Column

The use of capillary columns coated with mid-range polarity stationary phases (e.g. SE-30, SE-54, OV-17, or equivalent) is recommended.

Standard glass columns, of length 2 m to 4 m and of internal diameter 2 mm to 4 mm, packed with a mixture of 2,5 % QF1 + 1 % OV 11 + 0,5 % XE 60 on Chromosorb WHP 0,125 mm to 0,15 mm particle size, or any other stationary phases and inert supports recommended for organochlorine residue analyses could be used as an alternative.

The temperature programme for the column shall be chosen to separate the pesticides mixture specified in clause 1 into individual components (see annex A).

After installing a new column, it shall be conditioned for at least 24 h at a temperature slightly above the maximum proposed operating temperature, with carrier gas flowing through it and the end of the column disconnected from the detector.

5.8.3 Detector

Use an electron capture detector (ECD) operating either in constant current mode or in constant frequency mode at the polarization voltage, width, amplitude or frequency of the pulses at which a maximum of 0,05 ng heptachlor epoxide causes 40 % to 50 % full-scale deflection.

5.8.4 Carrier gas and make-up gas

Pure nitrogen (oxygen-free), pure helium or pure hydrogen, or a mixture of argon and methane [90 + 10, by volume or (95 + 5) % by volume].

5.9 Grinder

6 Sampling

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 6497 [1].

7 Preparation of test sample

Prepare the test sample in accordance with ISO 6498.

Grind a portion of the well-mixed laboratory sample (dry or low moisture products such as cereals and cereal products, oilseeds and oilseed meals, mixed feeds, hay, etc.) so that it passes completely through a sieve with 1 mm apertures. Mix thoroughly.

Chop up the high moisture products (e.g. grasses, silages, etc.) and mix thoroughly to obtain homogeneous samples.

8 Procedure

8.1 General

Carry out steps 8.2 to 8.4 using both the prepared test sample (clause 7) and a blank sample. The blank sample shall be free of residues at or above the limits of detection found from previous determinations. The blank extract is used to prepare the matrix-matched standard solution (see 8.5.3).

8.2 Test portion

Weigh, to the nearest 0,1 g, 50 g of the prepared test sample (clause 7) for dry or low moisture products, or 100 g for high moisture products, into a 1 000 ml conical flask.

8.3 Extraction

Add sufficient water to the test portion so that a total water amount of about 100 g is obtained. Allow the sample to soak for about 5 min. Add 200 ml of acetone. Close the flask tightly and shake for 2 h on a mechanical shaker or homogenize for 2 min in a high-speed blender.

Filter the homogenate with suction through a Büchner funnel (5.3) fitted with filter paper of medium porosity, into a 500 ml filtering flask (5.2). Wash the conical flask or the blender cup and the residue on the filter paper with two 25 ml portions of acetone, collecting the washings in the same filtering flask.

Measure the volume of the filtrate (V_1) and transfer one-fifth of the solution (V_2) to a 500 ml separating funnel. Add 250 ml of water, about 50 ml of saturated sodium chloride solution (4.9) and 100 ml of dichloromethane (4.4) to the separating funnel. Stopper and shake for 2 min.

Allow the phases to separate and draw off the lower phase (dichloromethane) into a second 500 ml separating funnel (5.1). Repeat the extraction twice with 50 ml of dichloromethane then combine the extracts in the same 500 ml separating funnel.

Wash the dichloromethane extract with two 100 ml portions of water, discarding the washings.

Filter the dichloromethane extract through a filter paper containing about 20 g of sodium sulfate (4.8) into a 500 ml flask of a vacuum evaporator. Rinse the separating funnel and the sodium sulfate with two 10 ml portions of dichloromethane and add to the flask.

Concentrate the solution to about 2 ml under vacuum at a temperature not exceeding 40 °C. Transfer the solution to a 10 ml graduated tube using 1 ml to 2 ml of hexane and concentrate under nitrogen to about 1 ml.

Do not allow the solution to dry completely or losses of pesticides may occur because of volatility or poor solubility.

8.4 Column clean-up

8.4.1 Preparation of the column

Transfer 5 g of silica gel (4.5) to a glass chromatographic tube (5.5). Add 5 g of anhydrous sodium sulfate (4.8) on the top of the silica gel. Wash the prepared column with 20 ml of hexane.

NOTE Prepacked silica or florisil cartridges (e.g. Millipore-SEP PAK) may be used instead of a silica gel column, after checking for efficiency and absence of interferences.

8.4.2 Purification

Transfer quantitatively the concentrated extract (8.3) on the top of the column (8.4.1) using 1 ml to 2 ml portions of hexane.

Elute the organochlorine pesticides with 50 ml of eluting solvent (4.6) and collect the eluate in the 100 ml flask of the rotary vacuum evaporator (5.6).

Concentrate the eluate to about 2 ml under vacuum at a temperature not exceeding 40 °C. Add about 10 ml of hexane and concentrate once more to about 1 ml. Repeat two times, leaving about 1 ml of hexane at the final evaporation. Transfer to a 10 ml graduated tube using 1 ml to 2 ml of hexane. Dilute the sample extract to 10 ml with hexane for determination by chromatography.

When an internal standard method is used, add 1 ml of the intermediate solution (4.12.2) of the internal standard (4.11) to the final extract before diluting to 10 ml with hexane. Keep the blank extract to prepare the matrix-matched standard solution (see 8.5).

8.5 Gas chromatography

8.5.1 Preparation of the system

Equilibrate the gas chromatographic system under the recommended operating conditions (5.8).

If the carrier gas volume flow rate through the column is below 25 ml/min, introduce a supplemental contribution of gas at the outlet of the column in order to ensure a sufficient flow of gas through the electron capture detector (make-up gas).

Dry the carrier gas by passing it through 0,5 nm molecular sieve traps, previously activated at 350 °C for 4 h to 8 h, installed in the carrier gas line.

Reactivate the molecular sieves each time a new gas cylinder is assembled and as often as needed.

8.5.2 Checking the linearity of the system

Check the linearity of the system from 0,05 ng to 0,5 ng of heptachlor epoxide.

Prepare working solutions with hetachlor epoxide contents ranging from 0,01 μg/ml to 0,1 μg/ml. Inject 1 μl.

Plot the response factor (area/mass, in nanograms, of heptachlor epoxide injected) versus the mass, in nanograms of heptachlor epoxide injected. The graph shall be straight line parallel to the x-axis. If not, establish the range of concentrations within which the detector response is linear.

8.5.3 Determination

Inject 1 μl to 2 μl of working standard solution (4.12.3), then the same volume of the sample extract (8.4.2). Dilute the sample extract if necessary.

Identify the individual pesticide peaks on the basis of retention times.

Determine the amount of pesticides by comparing the size of the sample peaks with those of the known amount of the corresponding pesticide peak in the working standard solution.

When the results based on solvent standards indicate residue levels at or above 50 % of the appropriate MRL, a matrix-matched solution should be used, prepared by adding to the blank extract (8.4.2) appropriate amounts of intermediate solutions (4.12.2) of those pesticides identified in the sample solution, so that the size of the peaks of this reference solution is within 25 % of the size of the peaks in the sample solution. Bring to 10 ml with hexane. Inject into the gas chromatograph the same volume as for the sample solution.

Determine the amount of pesticides by comparing the size of the sample peaks with those of the known amount of the corresponding pesticide peak in the matrix-matched standard solution.

9 Expression of results

9.1 Calculation

Calculate the residue of each individual pesticide in the test sample by the equation:

$$w = \frac{A \times A_{si} \times m_s \times V_2 \times V_3}{A_s \times A_i \times m \times V_1 \times V_4}$$

where

w is the content of an individual pesticide residue, in micrograms per gram, in the test sample;

A is the area (or height) of the pesticide peak in the sample solution;

A_s is the area (or height) of the corresponding standard pesticide peak in the working standard solution or in the matrix-matched standard solution;

A_i is the area (or height) of the internal standard peak in the sample solution;

A_{si} is the area (or height) of the internal standard peak in the working standard solution or in the matrix-matched standard solution;

m_s is the mass, in nanograms, of pesticide in the volume injected into the gas chromatograph;

V_1 is the total volume of filtrate, in millilitres, obtained in the extraction step;

V_2 is the volume of the filtrate, in millilitres, used in the purification step;

V_3 is the final volume, in millilitres, of the test solution, taking into account any further dilution that is necessary;

V_4 is the volume, in microlitres, of the sample solution injected into the gas chromatograph;

m is the mass, in grams, of the test portion.

9.2 Recovery

Verify the performance of the method by recovery experiments made on fortified blank samples at the 0,05 μg/g level.

Add to the test portion of a blank sample a known amount of pesticide solution. Let it stand for 30 min then analyse the fortified test portion together with the original blank sample with no added pesticides.

The recovery (%) is calculated for each pesticide as 100 × [(analysed amount in the fortified sample minus the original amount in the sample)/ (added amount)]

The recovery coefficient for each individual pesticide shall be between 70 % and 110 %.

When a residue exceeding a maximum residue limit (MLR) is to be confirmed, the concurrent recovery level should be approximately similar to that of the sample.

10 Confirmation of identity

Analyses for confirmation of the identity and quantity of pesticides shall be performed, particularly when the results obtained correspond to or exceed the maximum residue limits (MRLs), either by chromatography on a second column of significantly different polarity or, where the apparatus is available, by using GC-MS to quantify and confirm the identity of the pesticide.

11 Precision

11.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are given in annex B. The values derived from this test may not be applicable to concentration ranges and matrices other than those given.

11.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases exceed the repeatability limit r derived from Tables B.1 to B.17.

11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will in not more than 5 % of cases exceed the reproducibility limit R derived from Tables B.1 to B.17.

12 Test report

The test report shall specify:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with reference to this International Standard;

— all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

— the test result obtained, or the two test results obtained if the repeatability has been checked.

Annex A
(informative)

Examples of GC operating conditions for organochlorine pesticides

A.1 Example 1

Column:	fused silica capillary OV-1, length 25 m, internal diameter 0,25 mm, film thickness 0,25 μm
Column oven temperature:	50 °C for 1 min; 30 °C/min to 150 °C; 3 °C/min to 240 °C; 240 °C for 2 min
Injector:	splitless with 45 s delay, 250 °C, or on-column, initial oven temperature
Detector:	ECD, 300 °C or MSD

A.2 Example 2

Column:	fused silica capillary, SE-54 or OV-17, length 25 m, internal diameter 0,25 mm, film thickness 0,25 m
Column oven temperature:	4 °C/min from 60 °C to 240 °C
Injector:	splitless with 45 s delay, 250 °C, or on-column, ambient temperature
Detector:	ECD, 300 °C or MSD

Annex B
(informative)

Results of interlaboratory test

The precision of the method was established by an interlaboratory test organized by the Romanian Standardization institute (IRS) in 1996 and carried out in accordance with ISO 5725-2 [3]. In this test 12 laboratories participated. Samples of the following composition were investigated: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt, with organochlorine pesticide contents of 0,005 μg/g up to and including 0,5 μg/g.

Table B.1 — Statistical results for aldrin

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	12	12	12	12	12
Number of accepted results	24	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,0056	0,0124	0,045	0,084	0,438
Repeatability standard deviation (s_r), μg/g	0,0089	0,00122	0,0041	0,0054	0,027
Repeatability coefficient of variation, %	16,0	9,9	9,0	6,4	6,1
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0025	0,0034	0,011	0,015	0,076
Reproducibility standard deviation (s_R), μg/g	0,0014	0,00247	0,0068	0,009	0,051
Reproducibility coefficient of variation, %	25,3	19,9	15,1	10,8	11,6
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0039	0,0069	0,019	0,025	0,143

[a] 1: sample with a target aldrin content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target aldrin content of 0,01 μg/g; composition as for sample 1;

3: sample with a target aldrin content of 0,05 μg/g; composition as for sample 1;

4: sample with a target aldrin content of 0,1 μg/g; composition as for sample 1;

5: sample with a target aldrin content of 0,5 μg/g; composition: as for sample 1.

Table B.2 — Statistical results for *op'*-DDE

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	12	12	12	12	12
Number of accepted results	24	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,0068	0,0112	0,0438	0,083	0,427
Repeatability standard deviation (s_r), μg/g	0,0015	0,00148	0,0039	0,0064	0,0292
Repeatability coefficient of variation , %	21,7	13,2	8,9	7,7	6,8
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0042	0,0041	0,011	0,018	0,082
Reproducibility standard deviation (s_R), μg/g	0,0024	0,00247	0,0072	0,0115	0,067
Reproducibility coefficient of variation %	35,7	22,0	16,5	13,9	15,7
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0067	0,0069	0,020	0,032	0,188

[a] 1: sample with a target *op'*-DDE content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target *op'*-DDE content of 0,01 μg/g; composition as for sample 1;

3: sample with a target *op'*-DDE content of 0,05 μg/g; composition as for sample 1;

4: sample with a target *op'*-DDE content of 0,1 μg/g; composition as for sample 1;

5: sample with a target *op'*-DDE content of 0,5 μg/g; composition as for sample 1.

Table B.3 — Statistical results for *pp'*-DDE

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	12	12	12	12	12
Number of accepted results	24	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,0065	0,0093	0,043	0,0855	0,435
Repeatability standard deviation (s_r), μg/g	0,0013	0,0012	0,0035	0,0075	0,034
Repeatability coefficient of variation, %	19,5	12,9	8,2	8,7	7,8
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0036	0,0034	0,0098	0,021	0,095
Reproducibility standard deviation (s_R), μg/g	0,002	0,0021	0,0054	0,0135	0,069
Reproducibility coefficient of variation, %	30,8	22,6	12,6	15,8	15,9
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0056	0,0059	0,015	0,038	0,193

[a] 1: sample with a target *pp'*-DDE content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target *pp'*-DDE content of 0,01 μg/g; composition as for sample 1;

3: sample with a target *pp'*-DDE content of 0,05 μg/g; composition as for sample 1;

4: sample with a target *pp'*-DDE content of 0,1 μg/g; composition as for sample 1;

5: sample with a target *pp'*-DDE content of 0,5 μg/g; composition as for sample 1.

Table B.4 — Statistical results for *op'*-DDT

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	9	12	12	12	12
Number of accepted results	18	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,007	0,0094	0,045	0,087	0,46
Repeatability standard deviation (s_r), μg/g	0,00095	0,0013	0,0035	0,0049	0,029
Repeatability coefficient of variation, %	14,3	13,3	7,8	5,7	6,3
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0027	0,0036	0,010	0,014	0,081
Reproducibility standard deviation (s_R), μg/g	0,0022	0,0030	0,0098	0,013	0,041
Reproducibility coefficient of variation, %	31,6	26,2	18,0	15,0	8,8
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0062	0,0084	0,027	0,036	0,115

[a] 1: sample with a target *op'*-DDT content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target *op'*-DDT content of 0,01 μg/g; composition as for sample 1;

3: sample with a target *op'*-DDT content of 0,05 μg/g; composition as for sample 1;

4: sample with a target *op'*-DDT content of 0,1 μg/g; composition as for sample 1;

5: sample with a target *op'*-DDT content of 0,5 μg/g; composition as for sample 1.

Table B.5 — Statistical results for *pp'*-DDT

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	10	12	12	12	12
Number of accepted results	20	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,0077	0,0123	0,0495	0,088	0,46
Repeatability standard deviation (s_r), μg/g	0,00118	0,0017	0,0038	0,007	0,036
Repeatability coefficient of variation, %	15,4	14,0	7,7	8,0	7,9
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0033	0,0048	0,011	0,02	0,100
Reproducibility standard deviation (s_R), μg/g	0,0024	0,0037	0,0135	0,0142	0,047
Reproducibility coefficient of variation, %	31,2	30,0	27,3	16,2	10,2
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0067	0,010	0,038	0,040	0,132

[a] 1: sample with a target *pp'*-DDT content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target *pp'*-DDT content of 0,01 μg/g; composition as for sample 1;

3: sample with a target *pp'*-DDT content of 0,05 μg/g; composition as for sample 1;

4: sample with a target *pp'*-DDT content of 0,1 μg/g; composition as for sample 1;

5: sample with a target *pp'*-DDT content of 0,5 μg/g; composition as for sample 1.

Table B.6 — Statistical results for dieldrin

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	12	12	12	12	12
Number of accepted results	24	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,0047	0,0093	0,042	0,080	0,43
Repeatability standard deviation (s_r), μg/g	0,0008	0,0011	0,0041	0,0067	0,0347
Repeatability coefficient of variation, %	17,8	11,8	9,9	8,5	8,0
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0022	0,0031	0,0115	0,019	0,097
Reproducibility standard deviation (s_R), μg/g	0,0014	0,00176	0,0067	0,013	0,078
Reproducibility coefficient of variation, %	30,0	18,9	16,0	16,6	18,3
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0039	0,0049	0,0188	0,036	0,218

[a] 1: sample with a target dieldrin content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target dieldrin content of 0,01 μg/g; composition as for sample 1;

3: sample with a target dieldrin content of 0,05 μg/g; composition as for sample 1;

4: sample with a target dieldrin content of 0,1 μg/g; composition as for sample 1;

5: sample with a target dieldrin content of 0,5 μg/g; composition as for sample 1.

Table B.7 — Statistical results for endrin

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	11	11	11	11	11
Number of accepted results	22	22	22	22	22
Mean organochlorine pesticide content, μg/g	0,0046	0,0096	0,0446	0,084	0,448
Repeatability standard deviation (s_r), μg/g	0,00063	0,00105	0,0044	0,0057	0,036
Repeatability coefficient of variation, %	13,7	10,9	9,9	6,8	8,0
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0018	0,0029	0,0123	0,016	0,100
Reproducibility standard deviation (s_R), μg/g	0,00138	0,002	0,0053	0,0112	0,0494
Reproducibility coefficient of variation, %	30,0	20,8	11,9	13,4	11,0
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0039	0,0056	0,0148	0,031	0,138

[a] 1: sample with a target endrin content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target endrin content of 0,01 μg/g; composition: as sample 1;

3: sample with a target endrin content of 0,05 μg/g; composition: as sample 1;

4: sample with a target endrin content of 0,1 μg/g; composition: as sample 1;

5: sample with a target endrin content of 0,5 μg/g; composition: as sample 1.

Table B.8 — Statistical results for HCB

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	12	12	12	12	12
Number of accepted results	24	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,0047	0,0091	0,049	0,090	0,45
Repeatability standard deviation (s_r), μg/g	0,00077	0,0009	0,0035	0,0065	0,031
Repeatability coefficient of variation, %	16,5	9,8	7,1	7,2	6,9
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0022	0,0025	0,0098	0,0182	0,087
Reproducibility standard deviation (s_R), μg/g	0,00114	0,00179	0,006	0,0113	0,048
Reproducibility coefficient of variation, %	24,3	19,7	12,3	12,6	10,8
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0032	0,005	0,0168	0,032	0,134

[a] 1: sample with a target HCB content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target HCB content of 0,01 μg/g; composition as for sample 1;

3: sample with a target HCB content of 0,05 μg/g; composition as for sample 1;

4: sample with a target HCB content of 0,1 μg/g; composition as for sample 1;

5: sample with a target HCB content of 0,5 μg/g; composition as for sample 1.

Table B.9 — Statistical results α-HCH

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	12	12	12	12	12
Number of accepted results	24	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,0067	0,0137	0,055	0,092	0,48
Repeatability standard deviation (s_r), μg/g	0,0007	0,0014	0,0042	0,0064	0,035
Repeatability coefficient of variation, %	10,6	10,3	7,6	6,9	7,3
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0020	0,0039	0,0118	0,018	0,098
Reproducibility standard deviation (s_R), μg/g	0,00155	0,0027	0,0095	0,016	0,055
Reproducibility coefficient of variation, %	23,1	19,9	17,3	17,5	11,5
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0043	0,0076	0,027	0,045	0,154

[a] 1: sample with a target α-HCH content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target α-HCH content of 0,01 μg/g; composition as for sample 1;

3: sample with a target α-HCH content of 0,05 μg/g; composition as for sample 1;

4: sample with a target α-HCH content of 0,1 μg/g; composition as for sample 1;

5: sample with a target α-HCH content of 0,5 μg/g; composition as for sample 1.

Table B.10 — Statistical results for β-HCH

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	12	12	12	12	12
Number of accepted results	24	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,0088	0,0127	0,044	0,08	0,404
Repeatability standard deviation (s_r), μg/g	0,001	0,00134	0,0026	0,0051	0,0264
Repeatability coefficient of variation, %	11,9	10,6	5,9	6,5	6,5
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0028	0,0038	0,0073	0,0143	0,074
Reproducibility standard deviation (s_R), μg/g	0,0019	0,00255	0,005	0,011	0,036
Reproducibility coefficient of variation, %	21,9	20,0	11,4	13,8	9,0
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0053	0,0071	0,014	0,031	0,100

[a] 1: sample with a target β-HCH content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target β-HCH content of 0,01 μg/g; composition as for sample 1;

3: sample with a target β-HCH content of 0,05 μg/g; composition as for sample 1;

4: sample with a target β-HCH content of 0,1 μg/g; composition as for sample 1;

5: sample with a target β-HCH content of 0,5 μg/g; composition as for sample 1.

Table B.11 — Statistical results for γ-HCH

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	12	12	12	12	12
Number of accepted results	24	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,109	0,127	0,167	0,195	0,55
Repeatability standard deviation (s_r), μg/g	0,0069	0,0055	0,008	0,0063	0,030
Repeatability coefficient of variation, %	6,4	4,3	4,8	3,2	5,5
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,019	0,015	0,022	0,018	0,084
Reproducibility standard deviation (s_R), μg/g	0,017	0,0083	0,0168	0,0148	0,051
Reproducibility coefficient of variation, %	15,7	6,6	10,0	7,6	9,3
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,048	0,023	0,047	0,041	0,143

[a] 1: sample with a target γ-HCH content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target γ-HCH content of 0,01 μg/g; composition as for sample 1;

3: sample with a target γ-HCH content of 0,05 μg/g; composition as for sample 1;

4: sample with a target γ-HCH content of 0,1 μg/g; composition as for sample 1;

5: sample with a target γ-HCH content of 0,5 μg/g; composition as for sample 1.

Table B.12 — Statistical results for δ-HCH

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	12	12	12	12	12
Number of accepted results	24	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,0057	0,01	0,054	0,087	0,475
Repeatability standard deviation (s_r), μg/g	0,0007	0,00094	0,003	0,0059	0,036
Repeatability coefficient of variation, %	12,4	9,4	5,5	6,5	7,6
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0020	0,0026	0,0084	0,0165	0,100
Reproducibility standard deviation (s_R), μg/g	0,0011	0,00195	0,0062	0,0136	0,052
Reproducibility coefficient of variation, %	20,0	19,5	11,5	15,6	10,9
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0031	0,0055	0,017	0,038	0,146

[a] 1: sample with a target δ-HCH content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target δ-HCH content of 0,01 μg/g; composition as for sample 1;

3: sample with a target δ-HCH content of 0,05 μg/g; composition as for sample 1;

4: sample with a target δ-HCH content of 0,1 μg/g; composition as for sample 1;

5: sample with a target δ-HCH content of 0,5 μg/g; composition as for sample 1.

Table B.13 — Statistical results for heptachlor

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	12	12	12	12	12
Number of accepted results	24	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,005	0,01	0,046	0,088	0,445
Repeatability standard deviation (s_r), μg/g	0,00068	0,001	0,0042	0,0063	0,032
Repeatability coefficient of variation, %	13,5	10,0	9,2	7,2	7,2
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0019	0,0028	0,0118	0,0176	0,090
Reproducibility standard deviation (s_R), μg/g	0,0011	0,0018	0,0048	0,0109	0,056
Reproducibility coefficient of variation, %	22,2	18,4	10,5	12,3	12,7
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0031	0,0050	0,0134	0,031	0,157

[a] 1: sample with a target heptachlor content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target heptachlor content of 0,01 μg/g; composition as for sample 1;

3: sample with a target heptachlor content of 0,05 μg/g; composition as for sample 1;

4: sample with a target heptachlor content of 0,1 μg/g; composition as for sample 1;

5: sample with a target heptachlor content of 0,5 μg/g; composition as for sample 1.

Table B.14 — Statistical results for heptachlor epoxide

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	12	12	12	12	12
Number of accepted results	24	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,0054	0,0116	0,051	0,091	0,454
Repeatability standard deviation (s_r), μg/g	0,00063	0,00114	0,0033	0,005	0,027
Repeatability coefficient of variation, %	11,7	9,8	6,6	5,5	5,9
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0018	0,0032	0,0092	0,014	0,076
Reproducibility standard deviation (s_R), μg/g	0,00114	0,00226	0,0096	0,010	0,042
Reproducibility coefficient of variation, %	21,1	19,5	18,8	10,9	9,3
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0032	0,0063	0,027	0,039	0,213

[a] 1: sample with a target heptachlor epoxide content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target heptachlor epoxide content of 0,01 μg/g; composition as for sample 1;

3: sample with a target heptachlor epoxide content of 0,05 μg/g; composition as for sample 1;

4: sample with a target heptachlor epoxide content of 0,1 μg/g; composition as for sample 1;

5: sample with a target heptachlor epoxide content of 0,5 μg/g; composition as for sample 1.

Table B.15 — Statistical results for *op'*-DDD

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	8	12	12	12	12
Number of accepted results	16	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,0073	0,0095	0,038	0,08	0,40
Repeatability standard deviation (s_r), μg/g	0,00122	0,00126	0,0038	0,006	0,029
Repeatability coefficient of variation, %	16,8	13,3	10,0	7,5	7,3
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0034	0,0035	0,011	0,017	0,081
Reproducibility standard deviation (s_R), μg/g	0,0017	0,0020	0,0047	0,0128	0,061
Reproducibility coefficient of variation, %	23,3	21,6	12,5	16,0	15,2
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0048	0,0056	0,013	0,036	0,171

[a] 1: sample with a target *op'*-DDD content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target *op'*-DDD content of 0,01 μg/g; composition as for sample 1;

3: sample with a target *op'*-DDD content of 0,05 μg/g; composition as for sample 1;

4: sample with a target *op'*-DDD content of 0,1 μg/g; composition as for sample 1;

5: sample with a target *op'*-DDD content of 0,5 μg/g; composition as for sample 1.

Table B.16 — Statistical results for *pp'*-DDD

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	8	12	12	12	12
Number of accepted results	16	24	24	24	24
Mean organochlorine pesticide content, μg/g	0,007	0,011	0,049	0,084	0,42
Repeatability standard deviation (s_r), μg/g	0,00145	0,00145	0,0039	0,0068	0,033
Repeatability coefficient of variation, %	20,7	13,2	7,9	8,1	7,8
Repeatability limit (r) [r = 2,8 s_r], μg/g	0,0041	0,0041	0,011	0,019	0,092
Reproducibility standard deviation (s_R), μg/g	0,00197	0,00272	0,0064	0,0138	0,054
Reproducibility coefficient of variation, %	27,5	24,7	13,1	16,4	12,9
Reproducibility limit (R) [R = 2,8 s_R], μg/g	0,0054	0,0076	0,018	0,039	0,151

[a] 1: sample with a target *pp'*-DDD content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target *pp'*-DDD content of 0,01 μg/g; composition as for sample 1;

3: sample with a target *pp'*-DDD content of 0,05 μg/g; composition as for sample 1;

4: sample with a target *pp'*-DDD content of 0,1 μg/g; composition as for sample 1;

5: sample with a target *pp'*-DDD content of 0,5 μg/g; composition as for sample 1.

Table B.17 — Statistical results for methoxychlor

Parameter	Sample[a]				
	1	2	3	4	5
Number of laboratories retained after eliminating outliers	—	—	12	12	12
Number of accepted results	—	—	24	24	24
Mean organochlorine pesticide content, μg/g	—	—	0,046	0,091	0,414
Repeatability standard deviation (s_r), μg/g	—	—	0,0049	0,0085	0,036
Repeatability coefficient of variation, %	—	—	10,6	9,4	8,7
Repeatability limit (r) [r = 2,8 s_r], μg/g	—	—	0,014	0,024	0,100
Reproducibility standard deviation (s_R), μg/g	—	—	0,0072	0,0129	0,058
Reproducibility coefficient of variation, %	—	—	15,7	14,2	13,9
Reproducibility limit (R) [R = 2,8 s_R], μg/g	—	—	0,020	0,036	0,162

[a] 1: sample with a target methoxychlor content of 0,005 μg/g; composition: 50 % maize, 20 % barley, 20 % soya bean meal, 3 % fish meal, 3 % fat, 1 % premix, 1,5 % dicalcium phosphate, 1,2 % calcium carbonate and 0,3 % salt;

2: sample with a target methoxychlor content of 0,01 μg/g; composition as for sample 1;

3: sample with a target methoxychlor content of 0,05 μg/g; composition as for sample 1;

4: sample with a target methoxychlor content of 0,1 μg/g; composition as for sample 1;

5: sample with a target methoxychlor content of 0,5 μg/g; composition as for sample 1.

Bibliography

[1] ISO 6497, *Animal feeding stuffs — Sampling.*

[2] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

[3] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*.

ICS 65.120

Price based on 20 pages