Progetto Trieste – Pesticide Residues

FINAL REPORT

Laboratory Proficiency Testing for Food Analysis

PTP N° 0008 P

2023

Pesticide Residues

Round of October

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Round coordinator
27/12/2023 rev.0

INTRODUCTION

Test Veritas S.r.l has been accredited EN ISO/IEC 17043:2010 "Conformity assessment - General requirements for proficiency testing" by Accredia.
Also, the quality management system has been assessed and certified by SGS Italia as meeting the requirements of UNI EN ISO 9001:2015 for development and production of materials for agri-food analyses and proficiency supply.

77 laboratories participated to *"Progetto Trieste – Pesticide Residues Proficiency Testing 2023, round of October*".

The test materials were dispatched on October 16th and 17th 2023. The deadline for sending the results was November 20th 2023.
58 laboratories returned test results.

4 out of 58 laboratories (participants n. 177, 187, 192 and 2192) sent back the test results after the deadline.

Participants were from 35 countries.

This Proficiency Testing Scheme offered different sets of test materials (biological matrices, see Table a).

Table a: test materials.

Test material Code	matrices	
VF3700	lettuce	60g x 1
SF3701	salmon	50g x 1
E3702	eggs	50g x 1
H3703	honey	70g x 1
N3704	pistachio	40g x 1

The molecules that could be present in the test materials are shown in Table b.

Table b: Molecules that have been spiked to the sample.

Test Material	Molecules
VF3700	azoxystrobin*, boscalid, coumaphos*, cypermethrin (sum of constituent isomers), cyproconazole*, imidacloprid*, linuron, omethoate, oxadixyl*, vinclozolin
SF3701*	cyfluthrin (sum of constituent isomers), cyhalothrin-lambda, deltamethrin, HCH-A (alpha hexachlorocyclohexane), heptachlor, hexaflumuron
E3702*	aldrin, amitraz (sum of amitraz and all metabolites containing the 2,4-DMA moiety), chlordane (cis), diazinon, fipronil-sulfone, pirimiphos-methyl
H3703*	acetamiprid, amitraz (sum of amitraz and all metabolites containing the 2,4-DMA moiety), clothianidin, coumaphos, glyphosate, thiacloprid, thiamethoxam
N3704*	acetamiprid, clotiadin, imidacloprid, thiametoxam

* test/measurand not ISO/IEC 17043:2010 accredited

Acreditation excludes, in any case, Accredia from any responsibility regarding the offered service.

PREPARATION OF TEST MATERIALS, HOMOGENEITY, DISTRIBUTION

Lettuce: spiked and blank

Blank matrices (from organic farming) were homogenized with a cutter mixer. One portion of them was used in order to obtain blank sample: the other portion was added with spiking solution. The blank sample was checked for the absence of pesticides. Both samples separately, were homogenized with a magnetic stirrer and lyophilized. After mixing thoroughly, samples were dispensed into vials and kept at controlled temperature until dispatch. Spiked sample was checked for homogeneity.

Salmon: spiked and blank

Blank matrices were homogenized with a cutter mixer. One portion of them was used in order to obtain blank sample: the other portion was added with spiking solution. The blank sample was checked for the absence of pesticides. Both samples separately, were homogenized with a cutter mixer and lyophilized. After mixing thoroughly, samples were dispensed into vials and kept at controlled temperature until dispatch. Spiked sample was checked for homogeneity.

Eggs (albumen and yolk): spiked and blank

Blank matrices (from organic farming) were homogenized with a cutter mixer. One portion of them was used in order to obtain blank sample: the other portion was added with spiking solution. The blank sample was checked for the absence of pesticides. Both samples separately, were homogenized with a cutter mixer and lyophilized. After mixing thoroughly, samples were dispensed into vials and kept at controlled temperature until dispatch. Spiked sample was checked for homogeneity.

Honey: spiked and blank

Blank matrices (from organic farming) were homogenized. One portion of them was used in order to obtain blank sample: the other portion was added with spiking solution. The blank sample was checked for the absence of pesticides. Both samples separately, were homogenized. After mixing thoroughly, samples were dispensed into vials and kept at controlled temperature until dispatch. Spiked sample was checked for homogeneity.

Pistachio: contaminated and blank

Blank matrices (from organic farming) were homogenized and checked for the absence of pesticides. Contamined matrices were provided from a competent laboratory. Both samples separately, were homogenized with a mixer. The obtained test materials were dispensed into foil-laminate pouches and kept at controlled temperature until dispatch. Contaminated sample was checked for homogeneity.

Subcontractor

In order to check the batch's homogeneity, the analyses were performed by a competent subcontractor.

Homogeneity and stability

Appropriate homogeneity studies assessed that test materials are sufficiently homogeneous for the measurands under study.
Appropriate stability studies assessed that test materials are stable for the time that elapsed between the dispatch of the samples and the deadline for submission of results.
Information about homogeneity and stability studies are available under request.

Labels

To avoid labelling errors, the Test Material blank and contaminated are always labelled on different days.

Distribution

In accordance with the stability established, the test materials were shipped in proper containers and conditions.

Dispatch was followed by the courier's parcel tracking system on the web site.

Shipments outside the EU can follow different procedures.

The parcel contains:

- A number of units of test material.
- A covering letter with the necessary instructions for use and storage of the materials, together with indications for reporting results in the on-line form.

An e-mail informed participants that materials were shipped. The e-mail contained an attached copy of the covering letter, a letter with recommendations on how to use lyophilized samples and SDS.

PERFORMANCE EVALUATION CRITERIA

Laboratory code and confidentiality

Confidentiality is guaranteed. Participants are identified in the Final Report by a randomly assigned code.

The laboratory codes were communicated to participants through the web restricted area.

Result and concentration value

The compounds are generally detected by HPLC, GC, UPLC, etc…

Each participation includes the possibility of being evaluated for 2 different results (obtained with different methods or different technicians).

Participants were asked to report quantitative or semiquantitative (”lower than” or “higher than”) results. Results can be given as:.

- “=…” means that the analyte was detected and quantified
- “< of…” means that the analyte was not detected.
- “> of…” means that the analyte was detected but not quantified.
- “Not Searched” means that the laboratory did not perform the analysis.

Limit of quantification (LOQ)

Limit of quantification for the relative compounds was claimed by the laboratories.

“-” means “Not Reported”.

False negative results

In case of false negative results, no evaluation is given.

If the laboratory does not detect the analyte that is effectively present, as its method does not allow it, this is an information concerning the capability of the method. The result is “congruent” but the participant should take into consideration if his method has the appropriate capability in respect of his requirement.

Other compounds detected in the test material

Additional compounds detected by participants are shown on pages 35, 48, 60 and 73.

Elaboration of laboratory data

In the statistical data processing, all the data submitted by the participants are elaborated considering two decimal places. In case there are not declared decimal places, they are considered as corresponding to "zero" (E.g. 25=25,00 - 25,3=25,30 - 25,32=25,32).

CALCULATION OF THE ASSIGNED VALUE

The Assigned Value x_{pt}

The Assigned Value $x_{pt,}$ is the value attributed to a particular property of proficiency test items (definition from ISO13528:2022).

The procedure for determining the Assigned Value x_{pt} is described below.

After excluding results that are identified as invalid the data population was checked for normality and for the presence of outliers by applying appropriate statistics and visual presentations. x_{pt} represents the value of concentration obtained from Algorithm A (ISO 13528:2022) or from the median. The chosen value will be reported under the assigned value table.

Sometimes very low concentrations are quantified. When it occurs, the concentration value is assigned only if statistics described in this paragraph are applicable.

The value is not assigned when $p < 8$, where "*p"* is the number of data after invalid results rejection.

The standard uncertainty of the assigned value $u(x_{pt})$

The standard uncertainty $u(x_{pt})$ is calculated as:

$u(x_{pt}) = s^* / \sqrt{p}$

where:

- s^* is the robust estimate of the participant standard deviation;
- p is the number of participants.

In case of not negligible effects of inhomogeneity and instability, if $0.1 < [u(x_{pt})]^2/\sigma_{pt}^2 \leq 0.5$, the standard uncertainty is expanded by the factor 1.25:

$u(xpt) = 1{,}25\ [s^*/ \sqrt{p}]$

In case of median as estimator, the standard deviation is calculated as $s^* = MADe$ (where MADe is the Median Absolute Deviation).

When the standard uncertainty is too high, the assigned value could be inaccurate.

Therefore:

- In case $[u(x_{pt})]^2/\sigma_{pt}^2 > 0.5$, the consensus value is not determined and individual laboratory performance scores are not reported. Summary statistics are provided only for information.
- In case $0.1 < [u(x_{pt})]^2/\sigma_{pt}^2 \leq 0.5$, the uncertainty is not negligible. The effects of uncertainty are introduced in the calculation of the z-score (that will be calculated as z'-score). The standard uncertainty $u(x_{pt})$ is expanded by factor 1.25 only in case inhomogeneity and instability effects are not negligible.

It may happen that only few participants confirm the presence of some analytes in the test materials. In such cases, the presence of an analyte is considered:

- "unconfirmed", when less than 25 % of participants detect the compound;
- "confirmed", when 25% of participants, or more, detect the compound (the minimum number of positive results is anyway three).

z-score, z'-score and σ_{pt} (standard deviation for proficiency assessment):

When the number of quantitative data is $p \geq 8$, the participant's result are converted into a z-score according to the equation:

z-score $= (x_i - x_{pt}) / \sigma_{pt}$

where:

- x_i is the analyte concentration value reported by the laboratory;
- x_{pt} is the assigned value;
- σ_{pt} is the standard deviation for proficiency assessment calculated from $b\ x_{pt}$.
- b = %RSD / 100, (RSD = Relative Standard Deviation)

the %RSD value comes from the Horwitz equation (Horwitz, W., 1988, *Pure Appl. Chem.* 60, 855-864)

$\%RSD = 2^{(1-0.5 \log X_{pt})}$

where x_{pt} is expressed as a dimensionless concentration.

σ_{pt} is related to the concentration of the analyte: it comes from Horwitz equation (unless otherwise specified); in case of contamination less than 10 ppb the Thompson equation modified Horwitz equation (Thompson, M., 2000, *Analyst* 125, 385-386). In particular circumstance σ_{pt} is chosen from Proficiency Test provider's (PTp) experience, derived from previous rounds. The adopted criteria is reported in the specific test material table.

If $0.1 < [u(x_{pt})]^2/\sigma_{pt}^2 \leq 0.5$, participants' results are converted into a z'-score according to the equation:

$z'\text{-score} = (x_i - x_{pt}) / \sqrt{\sigma^2_{pt} + u^2(x_{pt})}$

where:

- x_i is the analyte concentration value reported by the laboratory;
- x_{pt} is the assigned value;
- σ_{pt} is the standard deviation for proficiency assessment calculated from $b\ x_{pt}$
- $u^2(x_{pt})$ is the standard uncertainty calculated as previously described.

In case of z'-score, the assigned value will be given in *italics* when the uncertainty is not negligible, with underlined font where inhomogeneity and instability effects are not negligible.

The laboratory performance evaluation was established taking into account the following criteria for z-score:

acceptable (satisfactory)	when	$\|z\| \leq 2.0$
warning signal (questionable)	when	$2.0 < \|z\| < 3.0$
action signal (unsatisfactory)	when	$\|z\| \geq 3.0$

PARTICIPANTS (ordered by Country)

- Centre national de toxicologie NIPA (Nouvel institut Pasteur), Dély Ibrahim, Algeria
- CER Groupe FUP, Marloie, Belgium
- FLVVT - Federaal Laboratorium voor Voedselveiligheid Tervuren Sectie Microscopie & Mycotoxines, Tervuren, Belgium
- Laboratoire Fédérale pour la Sécurité de la Chaîne Alimentaire Liège - LFSAL, Wandre, Belgium
- LABORATORIUM ECCA NV, Merelbeke, Belgium
- LOVAP NV, Geel, Belgium
- LCSSA - Labratoire Central de Contrôle de la Sécurité Sanitaire des Aliments, Cotonou, Benin
- JU Veterinarski Institut R.S."De Vaso Butozan", Banja Luka, Bosnia and Herzegovina
- NSF Brasil - Prestação de Serviços de Análises e Certificação Ltda, Viamão , Brasile
- LFDA/MG - Laboratório Federal de Defesa Agropecuária, Pedro Leopoldo - Minas Gerais, Brazil
- Laboratoire national de santé publique. Contrôle des Aliments et de la Nutrition Appliquée, Ouagadougou, Burkina Faso
- HYDRAC Laboratory, Douala, Cameroun
- AGQ Chile S.A. (Agriquem America S.A), Santiago, Chile
- CEIMIC CHILE, Santiago, Chile
- Comercial Analab Chile S.A., Macul (Santiago), Chile
- Corthorn Quality Chile S.A., Santiago, Chile
- INIA - Instituto de Investigaciones Agropecuarias - La Platina, Santiago, Chile
- MAG - Ministerio de Agricultura y Ganadería
- SENASA - Servicio Nacional de Salud Animal
- LANASEVE - Laboratorio Nacional de Servicios Veterinarios, Heredia, Costa Rica
- Animal Health Service of Bavaria - Tiergesundheitsdienst Bayern, Poing, Germany
- Institut für Hygiene und Umwelt, Hamburg, Germany
- Labor Kneißler GmbH & Co. KG, Burglengenfeld, Germany
- Landesamt für Umwelt und Arbeitsschutz, Saarbrücken, Germany
- Marjaan khatam, Tehran, Germany
- Ghana Standards Authority, Accra, Ghana
- Biolab Mike, Ioannina, Greece
- VELTIA Labs for Life, Sindos and Attika, Greece
- VELTIA LABS FOR LIFE VELTIA SA, Athens, Greece
- Kimron Veterinary Institute National Residue Control Laboratory, Bet-Dagan, Israel
- BonassisaLab S.p.A, Foggia, Italy
- Chimica Applicata Depurazione Acque di Giglio Filippo e C. S.n.c., Menfi, Italy
- Istituto Zooprofilattico Sperimentale del Piemonte, Liguria e Valle D Aosta “I. Altara”, Genova, Italy
- Istituto Zooprofilattico Sperimentale della Sardegna "G. Pegreffi", Sassari, Italy
- Istituto Zooprofilattico Sperimentale della Sicilia “A. Mirri”, Palermo, Italy
- LAB&CO S.R.L., Bronte, Italy
- Micro-B S.r.l., Asola, Italy
- Tetralab S.r.l., Giammoro - Pace del Mela, Italy
- Malawi Bureau of Standards Head Office. Testing Services, Blantyre, Malawi
- Makmal Kesihatan Awam Veterinar - Veterinary Public Health Laboratory, Sepang SELANGOR, Malaysia
- Agricultural Chemistry Division/Food Technology Laboratory, Ministry of AgroIndustry and Food Security, Reduit, Mauritius
- AGROLAB Dr. Verwey B.V., Barendrecht, Netherlands
- Eurofins Lab Zeeuws-Vlaanderen B.V., Graauw, Netherlands
- NofaLab B.V., Schiedam, Netherlands

- Normec Groen Agro Control DELFT RESEARCH GROUP B.V., Delfgauw, Netherlands
- AsureQuality Ltd, Lower Hutt, New Zealand
- NAFDAC - National Agency for Food and Drug Administration and Control Central Laboratory Complex, Lagos, Nigeria
- MAFWR - Central Laboratory of Food Safety - Food Safety and Quality Center, Seeb, Muscat, Oman
- ALS LS PERU S. A. C, Lima, Perù
- Eurofins Polska Sp. z o.o., Lodz, Poland
- Ministry of Public Health, Public Health Department, Central Food Laboratories - Food Safety & Environmental Health Division
- Central Food Laboratories, Doha, Qatar
- Laboratorio de Diagnóstico e Investigación Veterinaria Dr. Gerardino Medina H".Unidad de Residuos Tóxicos. Direccion Nacional de Salud Animal (DINASA) Ministero de Desarollo Agropecuniario (MIDA), Panama, Rep. Panama
- Public Health Authority, Mahe, Republic of Seychelles
- Fotometric Research Laboratory SRL, Voluntari, Romania
- IISPV Institutul de Igiena si Sanatate Publica Veterinar
- Institute for Hygiene and Veterinary Public Health, Sector 2, Bucharest, Romania
- Régional Center for Research in Toxicology and Environmental Security.Unité Qualité des Eaux et des Engrais (UQEE).
- CERES - LOCUSTOX Fondation, Dakar, Senegal
- Ainia - Parque tecnológico de Valencia, Paterna, Spain
- Laboratorio De Salud Pública De Lugo - Conselleria de Samdade - Xefarura Territorial de Lugo, Lugo, Spain
- Bureau of Quality Control of Livestock Products, Mueng, Pathumtanee, Thailand
- Arian Toxins Identification and Research Think Tank Laboratory, Kerman, Turkey
- Directorate of Government Analytical Laboratory, Kampala, Uganda
- Zambia Bureau of Standards, Lusaka, Zambia

ABSTRACT

Table e: Overall evaluation.

Test Material Code	analyte	number of evaluation	|Z-score| or |Z'-score| < 2 (n)	|Z-score| or |Z'-score| < 2 (%)
VF3700	azoxystrobin	12	11	92
	boscalid	12	11	92
	coumaphos	11	10	91
	cypermethrin (sum of constituent isomers)	11	9	82
	cyproconazole	12	10	83
	imidacloprid	10	7	70
	linuron	12	10	83
	oxadixyl	10	9	90
SF3701	cyhalothrin-lambda	9	6	67
	deltamethrin	10	8	80
	HCH-A (alpha hexachlorocyclohexane)	10	7	70
	heptachlor	10	6	60
E3702	aldrin	8	7	88
	chlordane (cis)	8	8	100
	fipronil-sulfone	12	11	92
H3703	acetamiprid	9	8	89
	clothianidin	9	8	89
	coumaphos	8	7	88
	thiacloprid	9	7	78
	thiamethoxam	9	7	78
N3704	acetamiprid	10	9	90
	clotiadin	9	9	100
	imidacloprid	9	8	89
	thiametoxam	8	8	100

REFERENCES

"Progetto Trieste" is managed in agreement to:

- UNI CEI EN ISO/IEC 17043:2010 Conformity assessment – General requirements for proficiency testing
- EURACHEM Selection, Use and Interpretation of Proficiency Testing (PT) Schemes, 3rd edition, 2021
- ISO 13528:2022 Statistical method for use in proficiency testing by interlaboratory comparisons
- ISO GUIDE 35 Reference materials – General and statistical principles for certification, 2017
- UNI CEI EN ISO 17034:2017 General requirements for the competence of reference materials producers
- IUPAC Technical Report The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, 2006

RESULTS

Proficiency test VF3700, result contents

pesticides in lettuce;

- **Test material VF3700**

Test material VF3700, lettuce

Table 1: results of analyses performed on test material VF3700 (spiked sample): pesticides detection.

VF3700 azoxystrobin, assigned value: 74,86 µg/kg satisfactory range: 36,71 – 113,00 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	**z'-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	NS	-		-	-
16	90	110	**0,79**	10	NO
26	72	>95	**-0,15**	10	NO
83	<10	-		<10	-
136	NS	-		-	-
153	80	90	**0,27**	10	NO
169	103	97	**1,48**	10	NO
176*	68	80	**-0,36**	<10	YES
177	92	83	**0,90**	>10	YES
180	76,99	97,80	**0,11**	10	YES
187	59,6	-	**-0,80**	<5	NO
188	56,30	-	**-0,97**	<5	-
190	35,0	>223	**-2,09**	10	NO
213	69	102	**-0,31**	10	YES
2213	78	95	**0,16**	10	YES

*Outlier: The participant declares doubts on reconstitution.

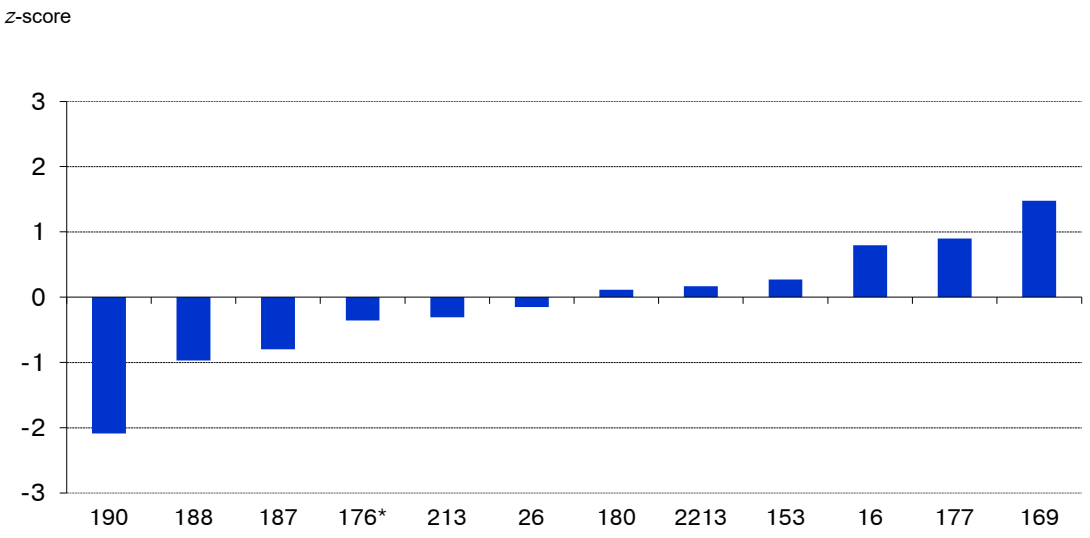
Test material VF3700, lettuce

Concentration, arithmetic mean	n= 12	73,32	ug/Kg
Standard deviation	n= 12	18,05	ug/Kg
Coefficient of variation	n= 12	24,61	%

Table 2: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
azoxystrobin	11	74,86	18,89	25,24	7,12	0,24	19,07

$0.1 < [u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.5$



* outliers

Test material VF3700, lettuce

Table 3: results of analyses performed on test material VF3700 (spiked sample): pesticides detection.

VF3700 boscalid, assigned value: 101,79 µg/kg satisfactory range: 55,85 – 147,74 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	NS	-		-	-
16	121	100	**0,84**	10	NO
26	85	>80	**-0,73**	10	NO
83	<10	-		<10	-
136	NS	-		-	-
153	123	90	**0,92**	10	NO
169	117	114	**0,66**	10	NO
176*	99	80	**-0,12**	<10	YES
177	123	87	**0,92**	>10	YES
180	98,48	99,03	**-0,14**	10	YES
187	98,5	-	**-0,14**	<5	NO
188	95,67	-	**-0,27**	<5	-
190	49,80	88,1	**-2,26**	10	NO
213	95	103	**-0,30**	10	YES
2213	90	92	**-0,51**	10	YES

*Outlier: The participant declares doubts on reconstitution.

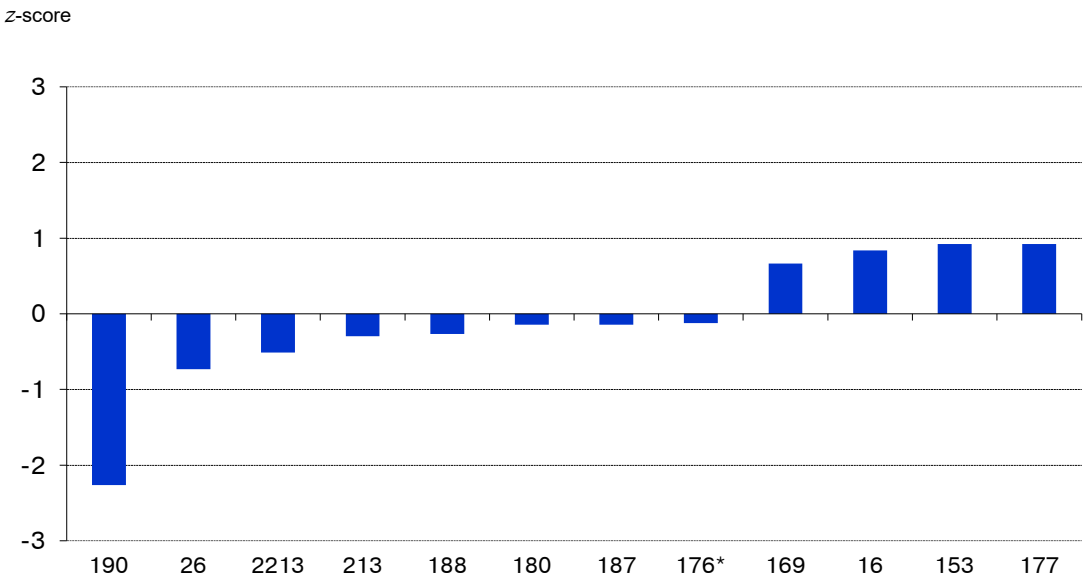
Test material VF3700, lettuce

Concentration, arithmetic mean	n= 12	99,62	ug/Kg
Standard deviation	n= 12	20,63	ug/Kg
Coefficient of variation	n= 12	20,71	%

Table 4: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
boscalid	11	101,79	19,13	18,80	5,77	0,23	22,97

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material VF3700, lettuce

Table 5: results of analyses performed on test material VF3700 (spiked sample): pesticides detection.

VF3700 coumaphos, assigned value: 99,73 µg/kg satisfactory range: 46,93 – 152,54 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	**z'-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	62,42	IS93,00	**-1,41**	10,00	NO
16	135	100	**1,34**	10	NO
26	NS	-		-	-
83	NS	-		-	-
136	NS	-		-	-
153	100	90	**0,01**	10	NO
169	162	79	**2,36**	10	NO
176*	94	80	**-0,22**	<10	YES
177	121	84	**0,81**	>10	YES
180	102,95	99,32	**0,12**	>10	YES
187	<5	-		<5	-
188	87,90	-	**-0,45**	<5	-
190	51,40	-	**-1,83**	10	-
213	96	102	**-0,14**	10	YES
2213	89	95	**-0,41**	10	YES

*Outlier: The participant declares doubts on reconstitution.

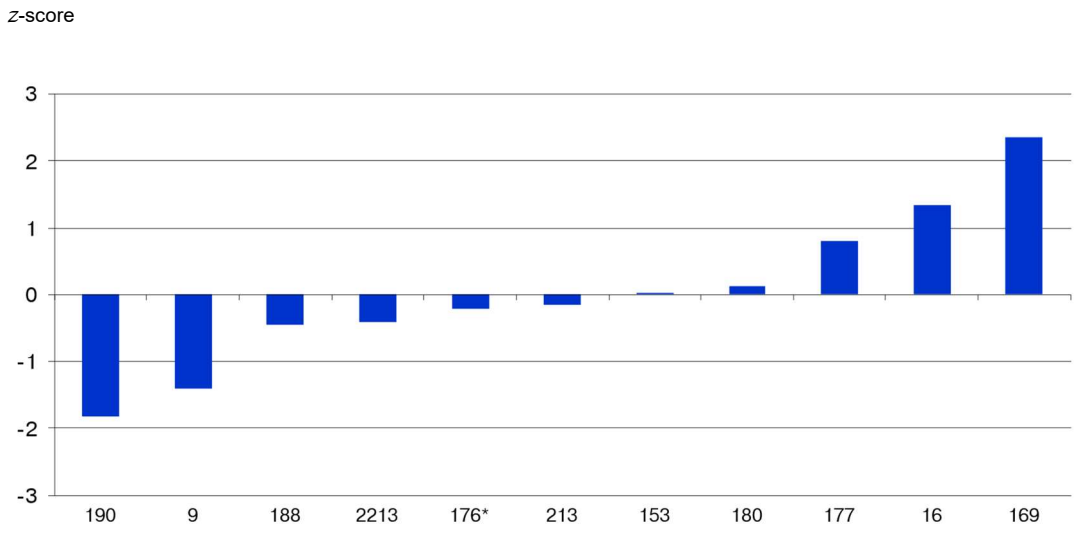
Test material VF3700, lettuce

Concentration, arithmetic mean	n= 11	100,15	ug/Kg
Standard deviation	n= 11	31,00	ug/Kg
Coefficient of variation	n= 11	30,96	%

Table 6: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
coumaphos	10	99,73	34,63	34,72	13,69	0,23	26,40

$0.1 < [u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.5$



* outliers

Test material VF3700, lettuce

Table 7: results of analyses performed on test material VF3700 (spiked sample): pesticides detection.

VF3700 cypermethrin (sum of constituent isomers), assigned value: *73,47* µg/kg satisfactory range: *36,61 – 112,34* µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	60,15	IS93,00	***-0,69***	10,00	NO
16	76	130	***0,13***	10	NO
26	NS	-		-	-
83	12,4	98	***-3,14***	>12,4	NO
136	NS	-		-	-
153	98	85	***1,26***	10	NO
169	87	105	***0,70***	10	NO
176*	76	80	***0,13***	<10	-
177	60	83	***-0,69***	>10	YES
180	71,17	90,75	***-0,12***	>10	YES
187	NS	-		-	-
188	85,67	-	***0,63***	<5	-
190	49,80	53	***-1,22***	10	NO
213	NS	-		-	-
2213	120	98	***2,39***	10	YES

*Outlier: The participant declares doubts on reconstitution.

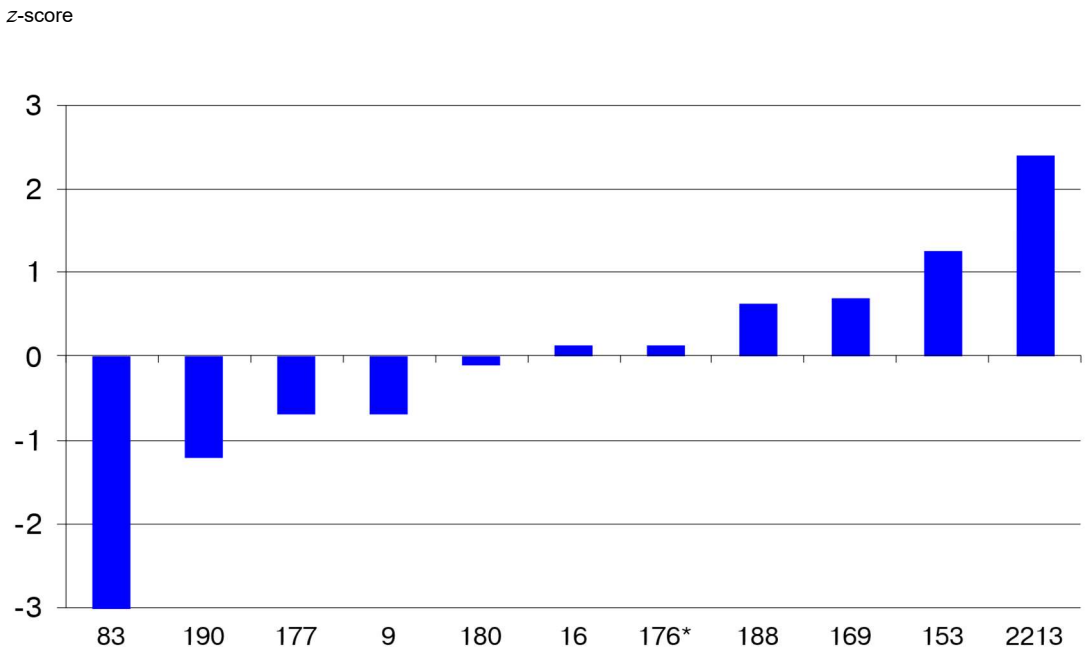
Test material VF3700, lettuce

Concentration, arithmetic mean	n= 11	72,38	ug/Kg
Standard deviation	n= 11	27,84	ug/Kg
Coefficient of variation	n= 11	38,47	%

Table 8: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
cypermethrin (sum of constituent isomers)	10	73,47	27,27	37,12	8,62	0,24	19,43

$0.1 < [u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.5$



* outliers

Test material VF3700, lettuce

Table 9: results of analyses performed on test material VF3700 (spiked sample): pesticides detection.

VF3700 cyproconazole, assigned value: 81,35 µg/kg satisfactory range: 43,37 – 119,32 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	NS	-		-	-
16	78	100	**-0,18**	10	NO
26	180	>80	**5,20**	10	NO
83	NS	-		-	-
136	NS	-		-	-
153	85	90	**0,19**	10	NO
169	30	84	**-2,70**	10	NO
176*	67	80	**-0,76**	<10	-
177	99	84	**0,93**	>10	YES
180	94,94	93,83	**0,72**	>10	YES
187	74,7	-	**-0,35**	<5	NO
188	73,67	-	**-0,40**	<5	-
190	66,80	151	**-0,77**	10	NO
213	94	98	**0,67**	10	YES
2213	66	90	**-0,81**	10	YES

*Outlier: The participant declares doubts on reconstitution.

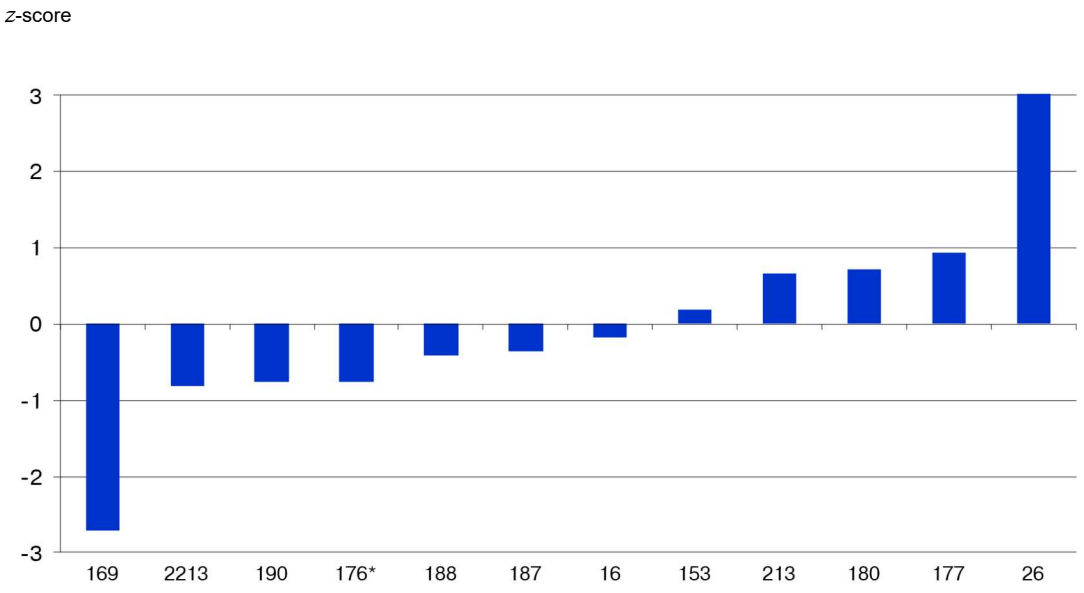
Test material VF3700, lettuce

Concentration, arithmetic mean	n= 12	84,09	ug/Kg
Standard deviation	n= 12	35,31	ug/Kg
Coefficient of variation	n= 12	41,99	%

Table 10: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
cyproconazole	11	81,35	19,60	24,09	5,91	0,23	18,99

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material VF3700, lettuce

Table 11: results of analyses performed on test material VF3700 (spiked sample): pesticides detection.

VF3700 imidacloprid, assigned value: 75,00 µg/kg satisfactory range: 39,56 µg/kg – 110,44 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	NS	-		-	-
16	125	70	**2,82**	10	NO
26	75	>80	**0,00**	10	NO
83	NS	-		-	-
136	NS	-		-	-
153	77	90	**0,11**	10	NO
169	136	104	**3,44**	10	NO
176*	62	80	**-0,73**	<10	YES
177	93	84	**1,02**	>10	YES
180	<10	-		>10	-
187	64,5	-	**-0,59**	<5	NO
188	73,70	-	**-0,07**	<5	-
190	33,48	77,4	**-2,34**	10	NO
213	NS	-		-	-
2213	74	92	**-0,06**	10	YES

*Outlier: The participant declares doubts on reconstitution.

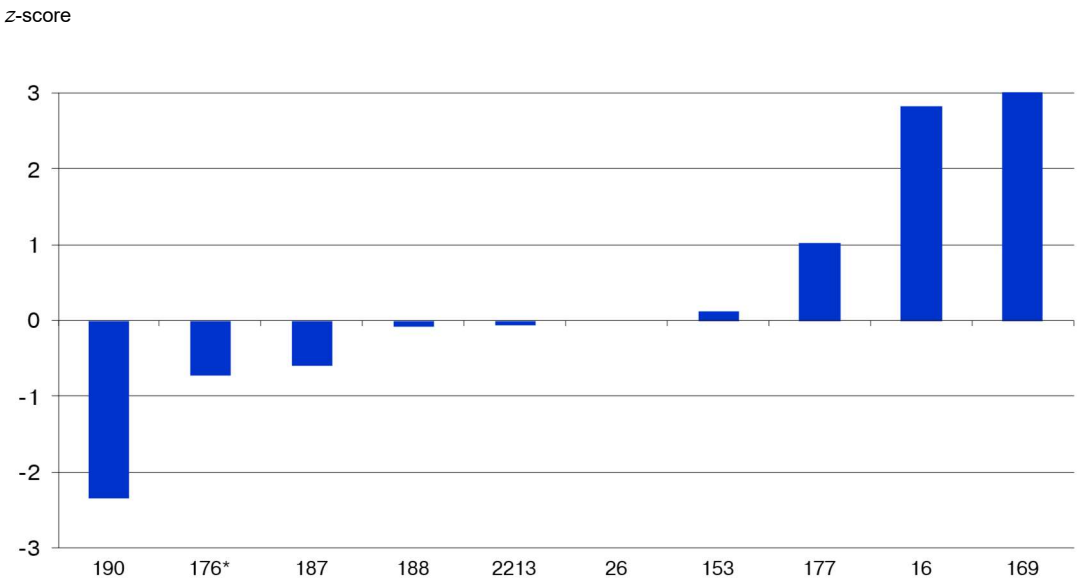
Test material VF3700, lettuce

Concentration, arithmetic mean	n= 10	81,37	ug/Kg
Standard deviation	n= 10	30,09	ug/Kg
Coefficient of variation	n= 10	36,98	%

Table 12: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Median	MADe	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
imidacloprid	9	75,00	15,57	20,76	5,19	0,24	17,72

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material VF3700, lettuce

Table 13: results of analyses performed on test material VF3700 (spiked sample): pesticides detection.

VF3700 linuron, assigned value: 64,58 µg/kg satisfactory range: 33,37 – 95,79 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	NS	-		-	-
16	67	120	**0,16**	10	NO
26	56	>90	**-0,55**	10	NO
83	NS	-		-	-
136	NS	-		-	-
153	73	90	**0,54**	10	NO
169	124	130	**3,81**	10	NO
176*	59	80	**-0,36**	<10	YES
177	79	87	**0,92**	>10	YES
180	53,29	100,39	**-0,72**	>10	YES
187	69,8	-	**0,33**	<5	NO
188	29,20	-	**-2,27**	<5	-
190	40,12	96,1	**-1,57**	10	NO
213	69	100	**0,28**	10	YES
2213	74	86	**0,60**	10	YES

*Outlier: The participant declares doubts on reconstitution.

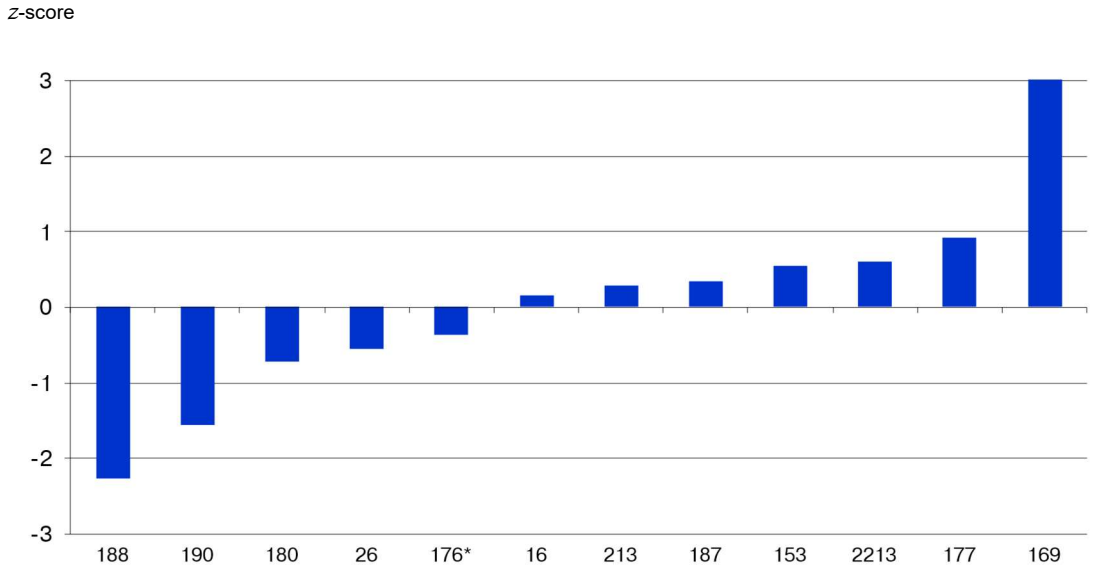
Test material VF3700, lettuce

Concentration, arithmetic mean	n= 12	66,12	ug/Kg
Standard deviation	n= 12	23,36	ug/Kg
Coefficient of variation	n= 12	35,34	%

Table 14: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
linuron	11	64,58	19,27	29,83	5,81	0,24	15,61

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material VF3700, lettuce

Table 15: results of analyses performed on test material VF3700 (spiked sample): pesticides detection.

VF3700 oxadixyl, assigned value: 192,87 µg/kg satisfactory range: 113,80 -271,94 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	NS	-		-	-
16	255	140	**1,57**	10	NO
26	NS	-		-	-
83	NS	-		-	-
136	NS	-		-	-
153	165	90	**-0,71**	10	NO
169	194	106	**0,03**	10	NO
176*	163	80	**-0,76**	<10	YES
177	200	83	**0,18**	>10	YES
180	NS	-		-	-
187	221,7	-	**0,73**	<5	NO
188	169,40	-	**-0,59**	<5	-
190	107,40	-	**-2,16**	10	NO
213	200	100	**0,18**	10	YES
2213	200	95	**0,18**	10	YES

*Outlier: The participant declares doubts on reconstitution.

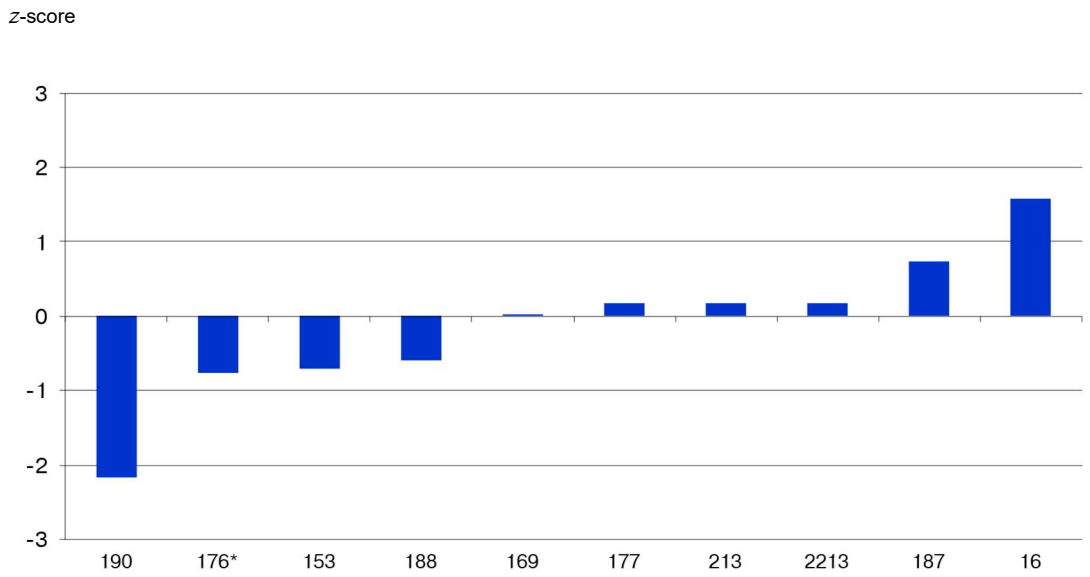
Test material VF3700, lettuce

Concentration, arithmetic mean	n= 10	187,55	ug/Kg
Standard deviation	n= 10	39,56	ug/Kg
Coefficient of variation	n= 10	21,09	%

Table 16: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
oxadixyl	9	192,87	36,57	18,96	12,19	0,20	39,53

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material VF3700, lettuce

Table 17: results of analyses performed on test material VF3700 (spiked sample): pesticides detection.

VF3700 vinclozolin, presence				
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	NS	-	-	-
16	47	110	10	NO
26	NS	-	-	-
83	NS	-	-	-
136	NS	-	-	-
153	55	90	10	NO
169	41	112	10	NO
176*	49	80	<10	YES
177	44	84	>10	YES
180	41,52	96,98	>10	YES
187	<5	-	<5	-
188	42,67	-	<5	-
190	<10	-	10	-
213	NS	-	-	-
2213	37	94	10	YES

*Outlier: The participant declares doubts on reconstitution.

Test material VF3700, lettuce

Concentration, arithmetic mean	n= 8	44,65	ug/Kg
Standard deviation	n= 8	5,57	ug/Kg
Coefficient of variation	n= 8	12,49	%

The value is not assigned when p < 8, where "p" is the number of data after invalid results rejection.

Test material VF3700, lettuce

Table 18: results of analyses performed on test material VF3700 (spiked sample): pesticides detection.

VF3700 omethoate, presence				
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	NS	-	-	-
16	68	120	10	NO
26	67	>90	10	NO
83	NS	-	-	-
136	NS	-	-	-
153	75	90	10	NO
169	32	117	10	NO
176*	41	80	<10	YES
177	26	83	>10	YES
180	>10	-	>10	-
187	43,7	-	<5	NO
188	42,60	-	<5	-
190	25,92	94,7	10	NO
213	67	98	10	YES
2213	63	95	10	YES

*Outlier: The participant declares doubts on reconstitution.

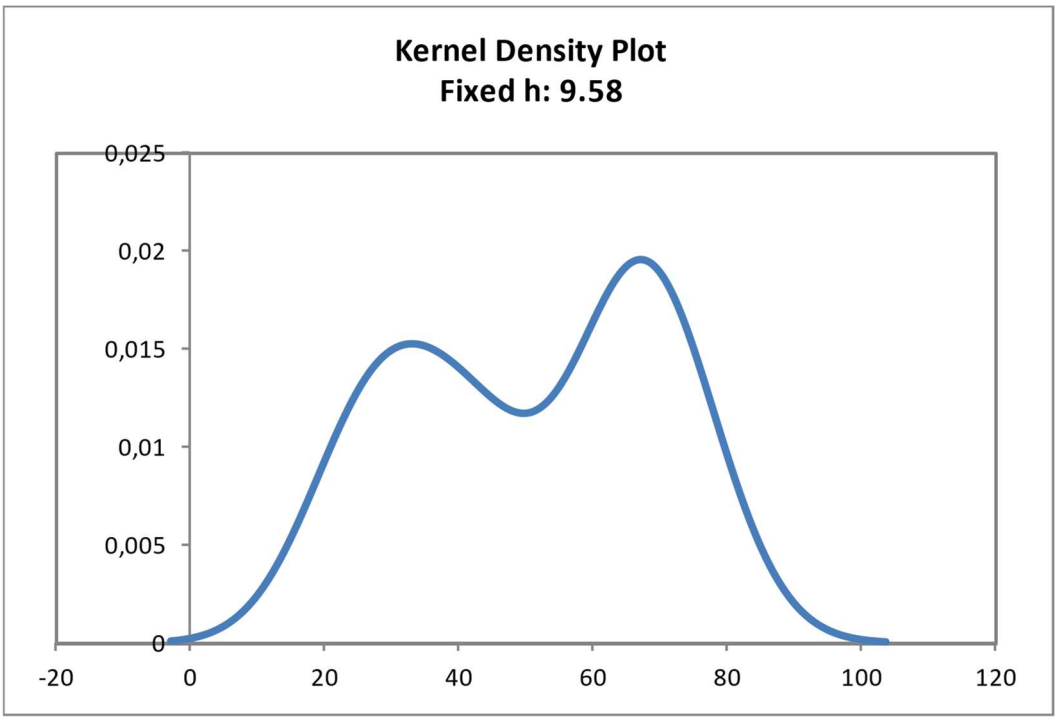
Test material VF3700, lettuce

Concentration, arithmetic mean	n= 11	50,11	ug/Kg
Standard deviation	n= 11	18,31	ug/Kg
Coefficient of variation	n= 11	36,53	%

It was not possible to assign a value, because the confirmatory data distribution is bimodal (see Kernel diagram).

In case of bimodal distribution ISO 13528 does not allow to give the assigned value.

A group of 5 participants have found a concentration about 36 μg/kg, another group of 5 participants have found a concentration about 69 μg/kg.



Test material VF3700, lettuce

Table 19: other compounds detected in the test material:

VF3700	
Lab. Code	**Other compounds (µg/kg)**
09	none
16	none
26	difenoconazole (79)
83	chlorpyrifos-methyl (23,03) cyhalothrin-lambda (35,21) diuron (33,59) metalaxyl (21,95) permethrin (70,25)
136	none
153	none
169	none
176	none
177	none
180	none
187	none
188	none
190	none
213	none
2213	none

Proficiency test SF3701, result contents

pesticides in salmon;

- **Test material SF3701**

Test material SF3701, salmon

Table 20: results of analyses performed on test material SF3701 (spiked sample): pesticides detection.

SF3701 cyhalothrin-lambda, assigned value: *77,00* µg/kg satisfactory range *35,23 – 118,77* µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z'*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
02	30,63	113	***-2,22***	2	NO
18	19,62	-	***-2,75***	<10	YES
21	87	134	***0,48***	10	YES
83	53,2	95	***-1,14***	53,2	NO
89	79	-	***0,10***	<10	YES
98	292	-	***10,30***	292	-
103	56	IS	***-1,01***	5	-
124	NS	-		-	-
146	<20	-		20	-
147	NS	-		-	-
168	NS	-		-	-
189	96	97	***0,91***	<10	-
200	77,0	IS	***0,00***	5	NO

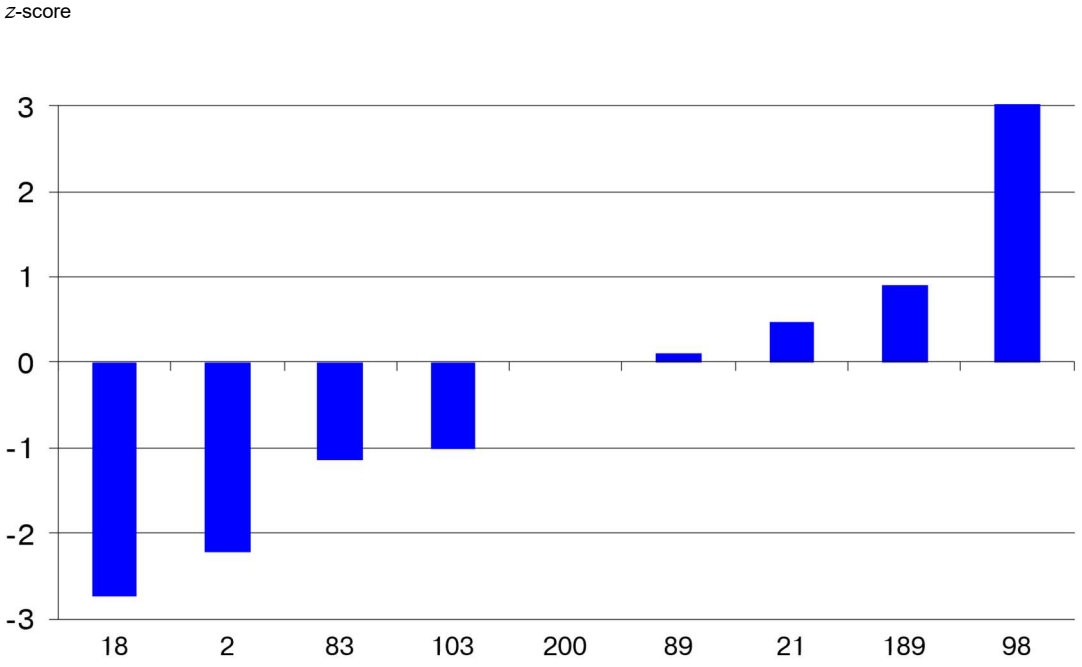
Test material SF3701, salmon

Concentration, arithmetic mean	n= 9	87,83	ug/Kg
Standard deviation	n= 9	80,69	ug/Kg
Coefficient of variation	n= 9	91,87	%

Table 21: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Median	MADe	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
cyhalothrin-lambda	9	*77,00*	*31,13*	40,43	10,38	0,24	20,88

$0.1 < [u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.5$



* outliers

Test material SF3701, salmon

Table 22: results of analyses performed on test material SF3701 (spiked sample): pesticides detection.

SF3701 deltamethrin, assigned value: 47,69 µg/kg satisfactory range: 23,56 – 71,81 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
02	43,21	93	**-0,37**	2	NO
18	13,4	-	**-2,84**	<5	YES
21	42	119	**-0,47**	10	NO
83	<10	-		<10	-
89	46	-	**-0,14**	<10	YES
98	228	-	**14,95**	228	-
103	58	IS	**0,85**	5	-
124	NS	-		-	-
146	54	-	**0,52**	20	YES
147	37	218	**-0,89**	10	YES
168	NS	-		-	-
189	52	99	**0,36**	<10	-
200	49,3	IS	**0,13**	5	NO

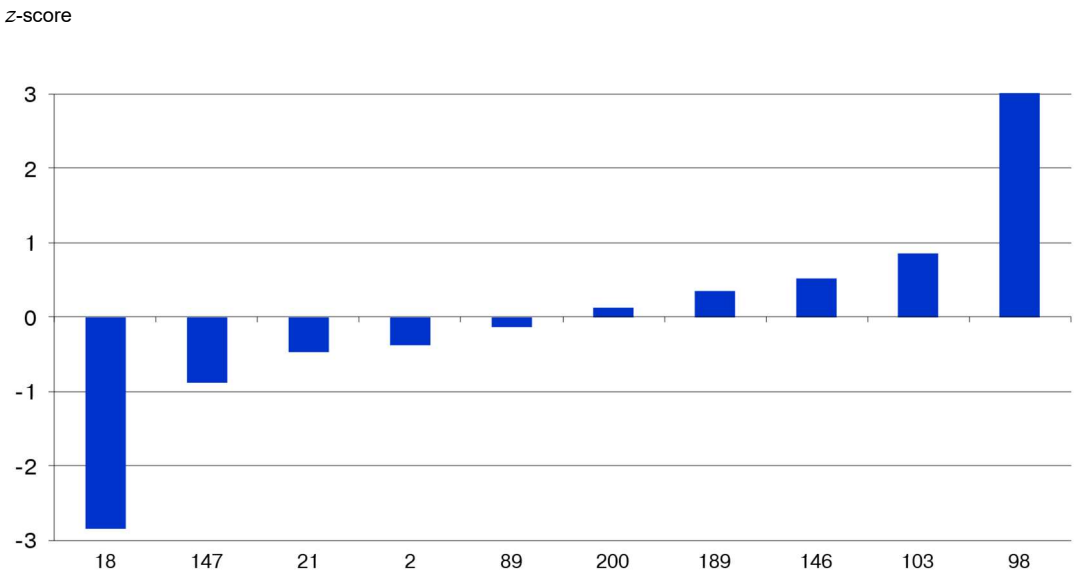
Test material SF3701, salmon

Concentration, arithmetic mean	n= 10	62,29	ug/Kg
Standard deviation	n= 10	59,53	ug/Kg
Coefficient of variation	n= 10	95,56	%

Table 23: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
deltamethrin	10	47,69	11,59	24,31	3,67	0,25	12,06

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material SF3701, salmon

Table 24: results of analyses performed on test material SF3701 (spiked sample): pesticides detection.

SF3701 HCH-A (alpha hexachlorocyclohexane), assigned value: *110.00* µg/kg satisfactory range: *52,67 – 167,33* µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z'*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
02	38,17	91	***-2,51***	2	NO
18	6,06	-	***-3,63***	<1	YES
21	132	94	***0,77***	10	NO
83	NS	-		-	-
89	110	-	***0,00***	<10	YES
98	466	-	***12,42***	466	-
103	140	IS	***1,05***	3	-
124	NS	-		-	-
146	<10	-		10	YES
147	110	109	***0,00***	10	YES
168	66,21	-	***-1,53***	2	YES
189	145	89	***1,22***	<10	-
200	82,3	IS	***-0,97***	2,5	NO

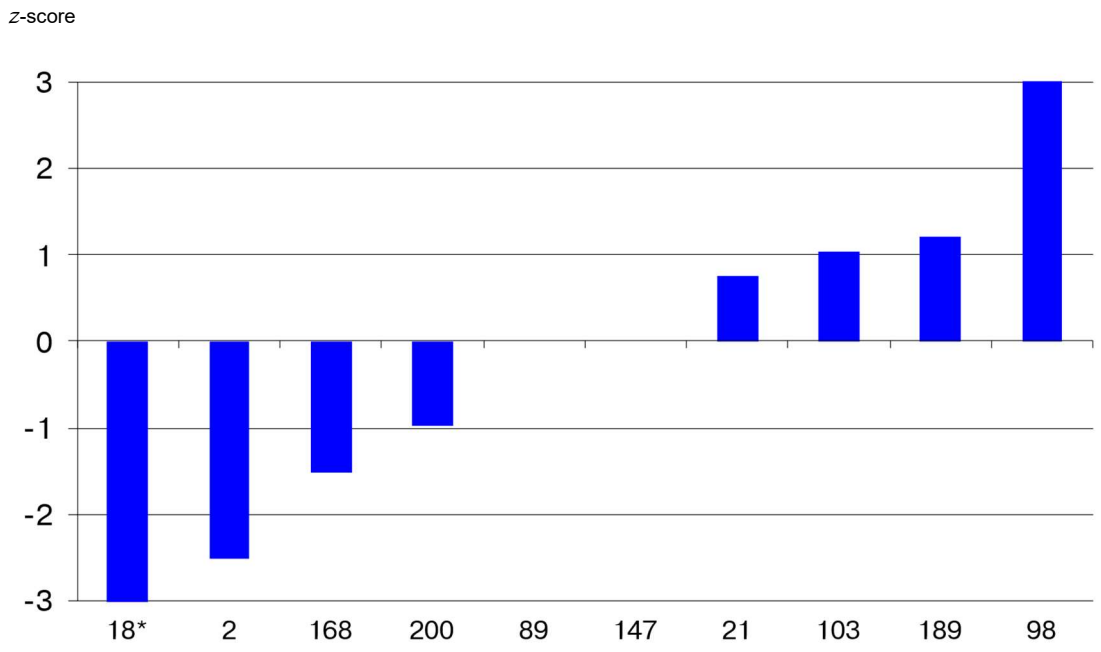
Test material SF3701, salmon

Concentration, arithmetic mean	n= 10	129,57	ug/Kg
Standard deviation	n= 10	126,56	ug/Kg
Coefficient of variation	n= 10	97,68	%

Table 25: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Median	MADe	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
HCH-A (alpha hexachlorocyclohexane)	9	*110,00*	*44,48*	40,43	14,83	0,22	28,67

$0.1 < [u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.5$



* outliers

Test material SF3701, salmon

Table 26: results of analyses performed on test material SF3701 (spiked sample): pesticides detection.

SF3701 heptachlor, assigned value: *87,50* µg/kg satisfactory range: *43,55 - 131,45* µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z'*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
02	24,18	83	***-2,88***	2	NO
18	1	-	***-3,94***	<1	YES
21	90	84	***0,11***	10	NO
83	NS	-		-	-
89	66	-	***-0,98***	<10	YES
98	344	-	***11,67***	344	-
103	<3	-		3	-
124	NS	-		-	-
146	105	-	***0,80***	10	YES
147	81	115	***-0,30***	10	YES
168	36,28	-	***-2,33***	2	YES
189	89	91	***0,07***	<10	-
200	87,5	IS	***0,00***	2,5	NO

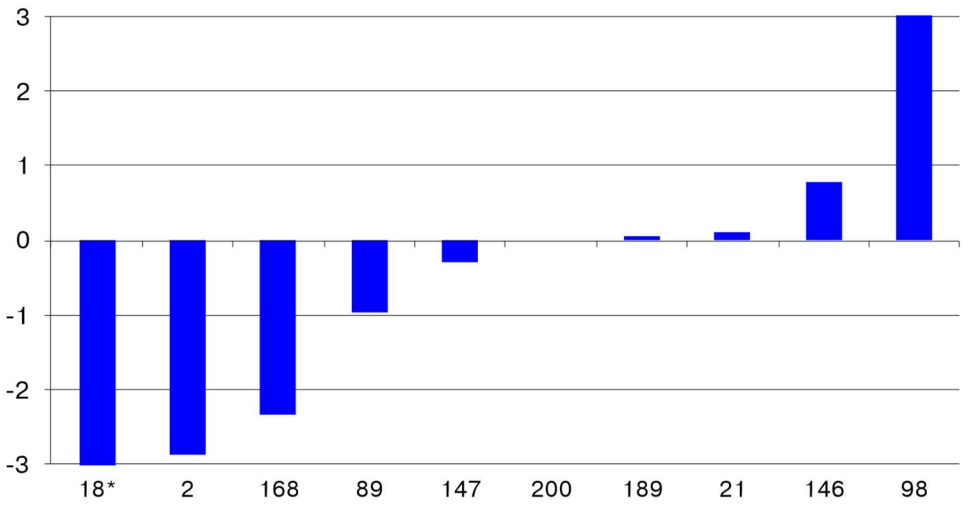
Test material SF3701, salmon

Concentration, arithmetic mean	n= 10	92,40	ug/Kg
Standard deviation	n= 10	94,57	ug/Kg
Coefficient of variation	n= 10	102,35	%

Table 27: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Median	MADe	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
heptachlor	9	*87,50*	*25,95*	29,65	8,65	0,23	21,97

$0.1 < [u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.5$



* outliers

Test material SF3701, salmon

Table 28: results of analyses performed on test material SF3701 (spiked sample): pesticides detection.

SF3701 cyfluthrin (sum of constituent isomers), presence				
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
02	<2	-	2	-
18	50,55	-	<10	YES
21	198	128	10	YES
83	NS	-	-	-
89	178	-	<10	YES
98	608	-	608	-
103	170	IS	5	-
124	NS	-	-	-
146	<20	-	20	-
147	<20	116	10	YES
168	NS	-	-	-
189	179	94	<10	-
200	202,0	IS	5	NO

Concentration, arithmetic mean	n= 7	226,51	ug/Kg
Standard deviation	n= 7	175,93	ug/Kg
Coefficient of variation	n= 7	77,67	%

The value is not assigned when p < 8, where "p" is the number of data after invalid results rejection.

Test material SF3701, salmon

Table 29: results of analyses performed on test material SF3701 (spiked sample): pesticides detection.

SF3701 hexaflumuron, presence				
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
02	NS	-	-	-
18	NS	-	-	-
21	142	70	10	NO
83	NS	-	-	-
89	25	-	<10	YES
98	689	-	689	-
103	160	IS	25	-
124	NS	-	-	-
146	NS	-	-	-
147	NS	-	-	-
168	NS	-	-	-
189	195	89	<10	-
200	NS	-	-	-

Concentration, arithmetic mean	n= 5	242,20	ug/Kg
Standard deviation	n= 5	257,79	ug/Kg
Coefficient of variation	n= 5	106,44	%

The value is not assigned when p < 8, where "p" is the number of data after invalid results rejection.

Test material SF3701, salmon

Table 30: other compounds detected in the test material:

SF3701	
Lab. code	**Other compounds (µg/kg)**
02	none
18	none
21	none
83	cypermethrin (sum of constituent isomers) (21,05) permethrin (sum of isomers) (171,4)
89	none
98	none
103	none
124	none
146	HCB (hexachlorobenzene) (169)
147	none
168	none
189	none
200	none

Proficiency test E3702, result contents

fipronil and other pesticides in eggs;

- **Test material E3702**

Test material E3702, eggs

Table 31: results of analyses performed on test material E3702 (spiked sample): fipronil and other pesticides detection.

E3702 aldrin, assigned value: 49,10 µg/kg satisfactory range: 24,37 – 73,84 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
02	41,29	76	**-0,63**	2	NO
09	20,43	-	**-2,32**	10,00	-
21	48	111	**-0,09**	10	NO
32	NS	-		-	-
57	58,40	89,54	**0,75**	3,00	NO
78	NS	-		-	-
82	<0,00	-		-	NO
83	NS	-		-	-
85	NS	-		-	-
91	49	IS	**-0,01**	5	NO
134	NS	-		-	-
146	63	-	**1,12**	10	YES
147	61	131	**0,96**	10	YES
162	NS	-		-	-
197	NS	-		-	-
257	42,33	79,64	**-0,55**	3,00	NO
332	NS	-		-	-

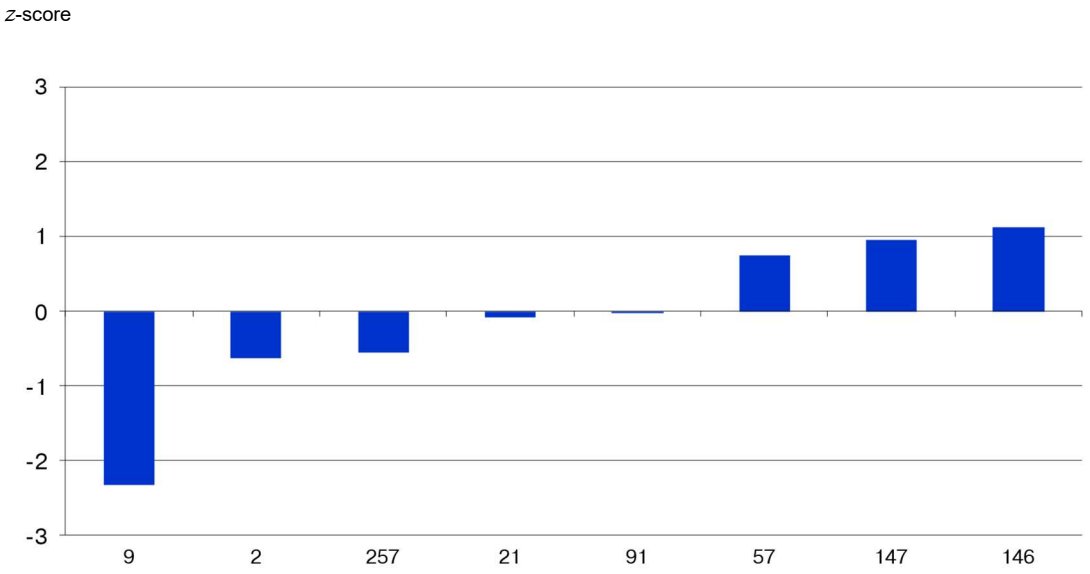
Test material E3702, eggs

Concentration, arithmetic mean	n= 8	47,93	ug/Kg
Standard deviation	n= 8	13,84	ug/Kg
Coefficient of variation	n= 8	28,87	%

Table 32: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
aldrin	8	49,10	12,86	26,19	4,55	0,25	12,37

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material E3702, eggs

Table 33: results of analyses performed on test material E3702 (spiked sample): fipronil and other pesticides detection.

E3702 chlordane (cis), assigned value: 54,45 µg/kg satisfactory range: 27,45 – 81,45 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
02	45,10	93	**-0,69**	2	NO
09	39,15	-	**-1,13**	10,00	-
21	50	95	**-0,33**	10	NO
32	NS	-		-	-
57	65,85	103,57	**0,84**	3,00	NO
78	NS	-		-	-
82	<0,00	-		-	NO
83	NS	-		-	-
85	NS	-		-	-
91	58	IS	**0,26**	5	NO
134	<10	-		10	-
146	66	-	**0,86**	10	-
147	57	140	**0,19**	10	YES
162	NS	-		-	-
197	NS	-		-	-
257	54,48	87,67	**0,00**	3,00	NO
332	NS	-		-	-

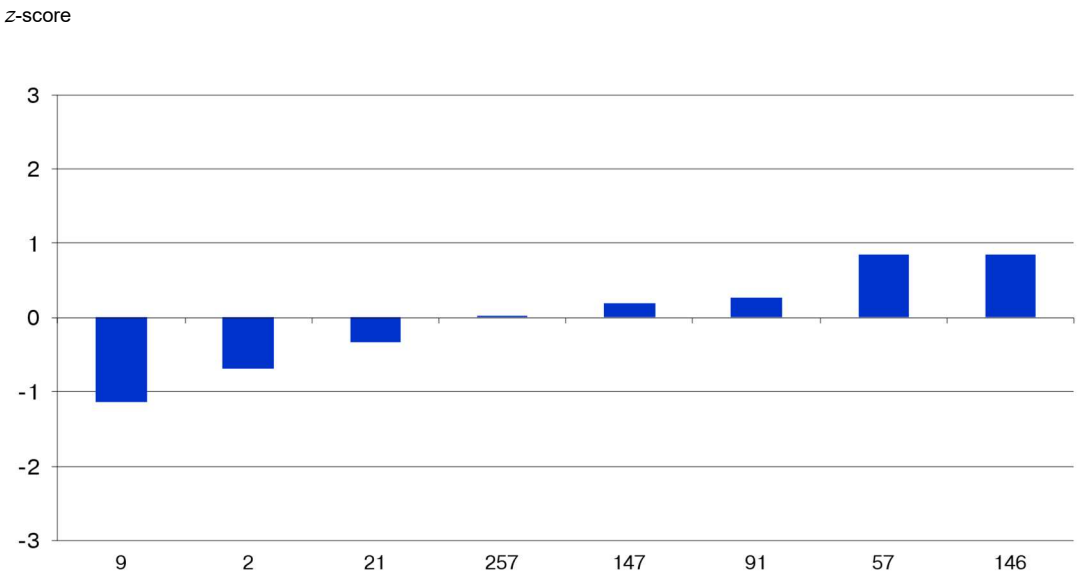
Test material E3702, eggs

Concentration, arithmetic mean	n= 8	54,45	ug/Kg
Standard deviation	n= 8	9,44	ug/Kg
Coefficient of variation	n= 8	17,34	%

Table 34: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
chlordane (cis)	8	54,45	10,70	19,65	3,78	0,25	13,50

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material E3702, eggs

Table 35: results of analyses performed on test material E3702 (spiked sample): fipronil and other pesticides detection.

E3702 fipronil-sulfone, assigned value: 41,51 µg/kg satisfactory range: 20,07 – 62,95 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
02	45,30	104	**0,35**	2	NO
09	NS	-		-	-
21	34	114	**-0,70**	5	NO
32	35,81	100,25	**-0,53**	2	NO
57	42,13	103,12	**0,06**	1,00	NO
78	38,3	101	**-0,30**	5	NO
82	<0,00	-		-	NO
83	NS	-		-	-
85	42,6	97,8	**0,10**	5	NO
91	NS	-		-	-
134	65,55	110	**2,24**	2	-
146	42	-	**0,05**	2,5	YES
147	41	97	**-0,05**	10	YES
162	42,2	99	**0,06**	3	YES
197	41,5	-	**0,00**	5	NO
257	44,24	99,29	**0,25**	1,00	NO
332	NS	-		-	-

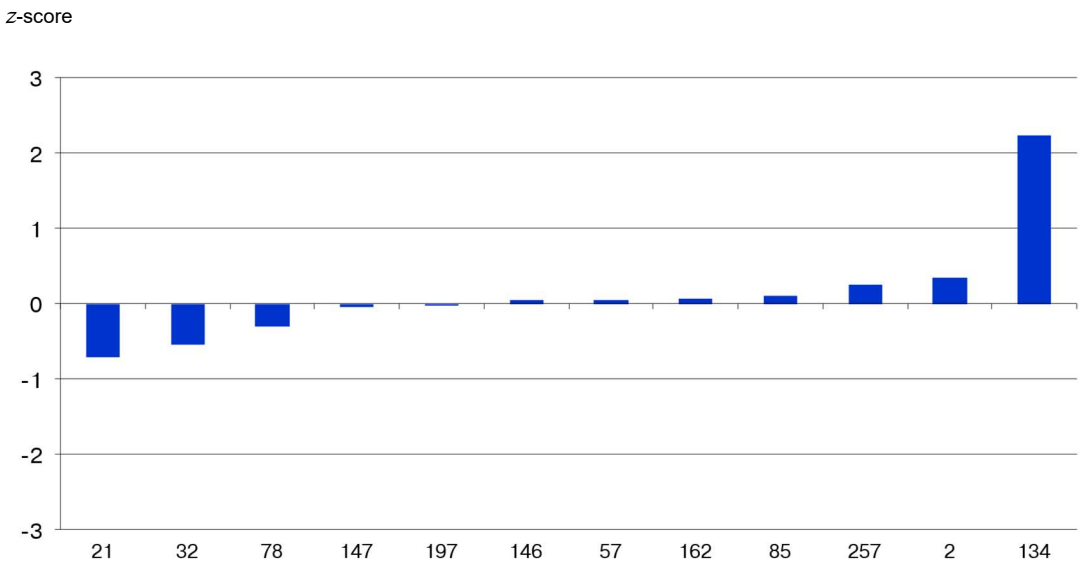
Test material E3702, eggs

Concentration, arithmetic mean	n= 12	42,89	ug/Kg
Standard deviation	n= 12	7,86	ug/Kg
Coefficient of variation	n= 12	18,32	%

Table 36: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
fipronil-sulfone	12	41,51	4,07	9,79	1,17	0,26	10,72

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material E3702, eggs

Table 37: results of analyses performed on test material E3702 (spiked sample): fipronil and other pesticides detection.

E3702 amitraz (sum of amitraz and all metabolites containing the 2,4-DMA moiety), presence				
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
02	NS	-	-	-
09	NS	-	-	-
21	63	80	10	NO
32	NS	-	-	-
57	NS	-	-	-
78	NS	-	-	-
82	<0,00	-	-	NO
83	26,13	98	>26,13	NO
85	NS	-	-	-
91	NS	-	-	-
134	<10	-	10	-
146	46	-	2,5	YES
147	NS	-	-	-
162	NS	-	-	-
197	NS	-	-	-
257	NS	-	-	-
332	NS	-	-	-

Concentration, arithmetic mean	n= 3	45,04	ug/Kg
Standard deviation	n= 3	18,45	ug/Kg
Coefficient of variation	n= 3	40,97	%

The value is not assigned when p < 8, where "p" is the number of data after invalid results rejection.

Test material E3702, eggs

Table 38: results of analyses performed on test material E3702 (spiked sample): fipronil and other pesticides detection.

E3702 diazinon, assigned value: presence				
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
02	57,52	100	2	NO
09	<10,00	-	10,00	-
21	52	121	10	YES
32	NS	-	-	-
57	NS	-	-	-
78	NS	-	-	-
82	<0,02	-	0,02	NO
83	NS	-	-	-
85	NS	-	-	-
91	54	IS	5	NO
134	34,97	81	10	-
146	113	-	10	YES
147	61	106	10	YES
162	NS	-	-	-
197	NS	-	-	-
257	NS	-	-	-
332	NS	-	-	-

Concentration, arithmetic mean	n= 6	62,08	ug/Kg
Standard deviation	n= 6	26,52	ug/Kg
Coefficient of variation	n= 6	42,72	%

The value is not assigned when p < 8, where "p" is the number of data after invalid results rejection.

Test material E3702, eggs

Table 39: results of analyses performed on test material E3702 (spiked sample): fipronil and other pesticides detection.

E3702 pirimiphos-methyl, presence				
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
02	37,53	102	2	NO
09	<10,00	-	10,00	-
21	31	134	10	YES
32	NS	-	-	-
57	NS	-	-	-
78	NS	-	-	-
82	<0,02	-	0,02	NO
83	17,91	97	>17,91	NO
85	NS	-	-	-
91	32	IS	5	NO
134	34,02	66	10	-
146	45	-	10	YES
147	29	91	10	YES
162	NS	-	-	-
197	NS	-	-	-
257	NS	-	-	-
332	NS	-	-	-

Concentration, arithmetic mean	n= 7	32,35	ug/Kg
Standard deviation	n= 7	8,28	ug/Kg
Coefficient of variation	n= 7	25,59	%

The value is not assigned when p < 8, where "p" is the number of data after invalid results rejection.

Test material E3702, eggs

Table 40: other compounds detected in the test material:

E3702	
Lab. code	**Other compounds (µg/kg)**
02	none
09	none
21	none
32	none
57	none
78	none
82	none
83	none
85	none
91	none
134	chlordane (trans) (23,8)
146	none
147	none
162	none
197	none
257	none
332	PCB 52 (2,9)

Proficiency test H3703, result contents

pesticides in honey;

- **Test material H3703**

Test material H3703, honey

Table 41: results of analyses performed on test material H3703 (spiked sample): pesticides detection.

H3703 acetamiprid, assigned value: 112,33 µg/kg satisfactory range: 62,38 – 162,28 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	NS	-		-	-
26	120	>80	**0,31**	<10	NO
35	110,59	IS	**-0,07**	10	-
82	<0,00	-		-	NO
108	106,00	91	**-0,25**	10	NO
121	49,05	IS	**-2,53**	5	YES
147	130	108	**0,71**	10	NO
149	132,24	71,60	**0,80**	5,0	NO
175	114,44	IS	**0,08**	10	YES
210	93,4	88,2	**-0,76**	50	NO
3175	118,10	IS	**0,23**	10	YES

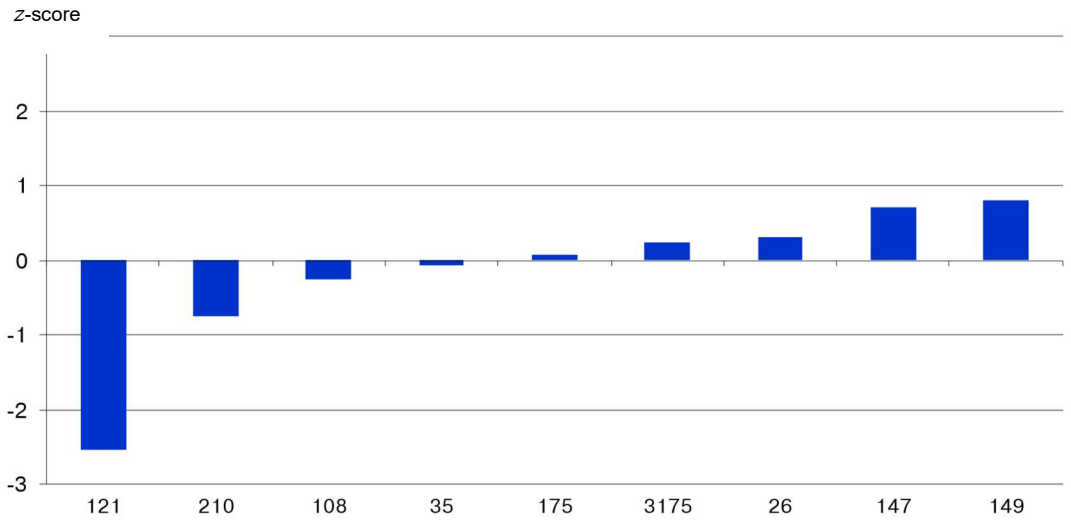
Test material H3703, honey

Concentration, arithmetic mean	n= 9	108,20	ug/Kg
Standard deviation	n= 9	25,14	ug/Kg
Coefficient of variation	n= 9	23,24	%

Table 42: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u(x_{pt})$	b	σ_{pt}
acetamiprid	9	112,33	17,42	15,50	5,81	0,22	24,98

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material H3703, honey

Table 43: results of analyses performed on test material H3703 (spiked sample): pesticides detection.

H3703 clothianidin, assigned value: 76,08 µg/kg satisfactory range: 36,95 – 115,22 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z'*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	NS	-		-	-
26	84	>90	***0,40***	<10	YES
35	90,26	IS	***0,72***	10	-
82	<0,00	-		-	NO
108	79,00	91	***0,15***	10	NO
121	50,65	IS	***-1,30***	5	YES
147	110	105	***1,73***	10	NO
149	28,97	<5,0	***-2,41***	5,0	NO
175	84,45	IS	***0,43***	10	YES
210	76,4	92,2	***0,02***	50	NO
3175	69,09	IS	***-0,36***	10	YES

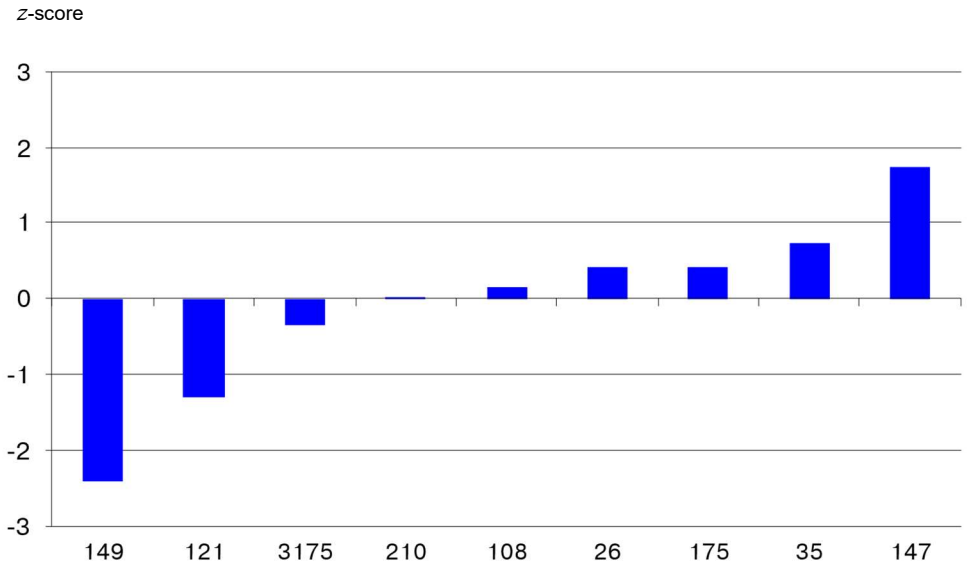
Test material H3703, honey

Concentration, arithmetic mean	n= 9	74,76	ug/Kg
Standard deviation	n= 9	23,43	ug/Kg
Coefficient of variation	n= 9	31,34	%

Table 44: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u(x_{pt})$	b	σ_{pt}
clothianidin	9	*76,08*	*23,45*	30,83	7,82	0,24	19,57

$0.1 < [u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.5$



* outliers

Test material H3703, honey

Table 45: results of analyses performed on test material H3703 (spiked sample): pesticides detection.

H3703 coumaphos, assigned value: 86,14 µg/kg satisfactory range: 46,27 – 126,01 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	239,4	IS140	**7,69**	10,00	NO
26	NS	-		-	-
35	73,30	IS	**-0,64**	10	-
82	<0,5	-		0,5	NO
108	75,00	89	**-0,56**	10	NO
121	94,03	IS	**0,40**	5	YES
147	93	88	**0,34**	10	NO
149	NS	-		-	-
175	77,25	IS	**-0,45**	10	YES
210	78,7	89,6	**-0,37**	50	NO
3175	91,80	-	**0,28**	10	YES

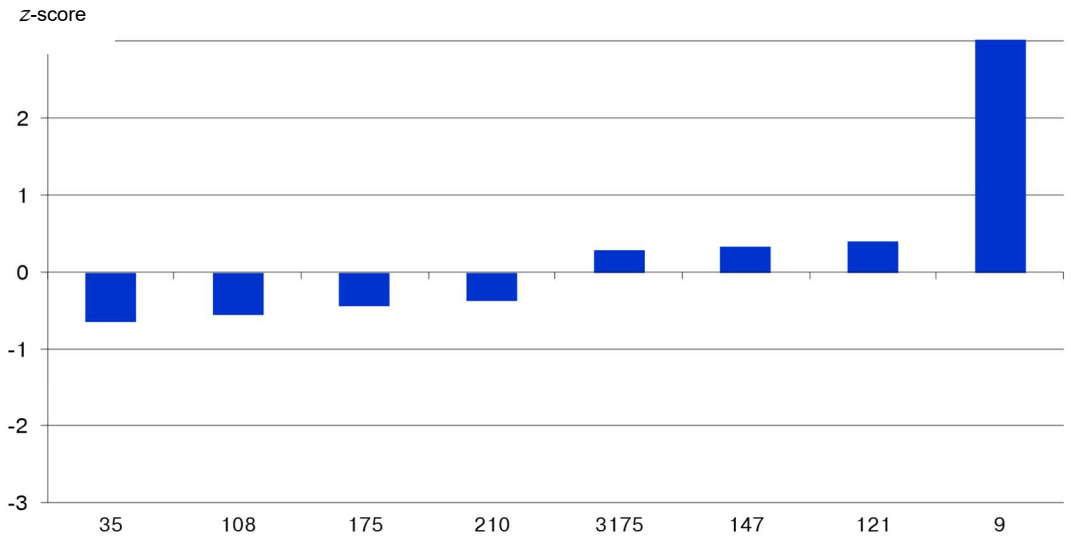
Test material H3703, honey

Concentration, arithmetic mean	n= 8	102,81	ug/Kg
Standard deviation	n= 8	55,84	ug/Kg
Coefficient of variation	n= 8	54,32	%

Table 46: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u(x_{pt})$	b	σ_{pt}
coumaphos	8	86,14	13,29	15,42	4,70	0,23	19,93

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material H3703, honey

Table 47: results of analyses performed on test material H3703 (spiked sample): pesticides detection.

H3703 thiacloprid, assigned value: 90,17 µg/kg satisfactory range: 48,72 – 131,61 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	NS	-		-	-
26	90	>85	**-0,01**	<10	NO
35	90,67	IS	**0,02**	10	-
82	<0,00	-		-	NO
108	92,00	108	**0,09**	10	NO
121	41,00	IS	**-2,37**	5	YES
147	140	128	**2,40**	10	NO
149	95,46	97,80	**0,26**	5,0	NO
175	92,45	IS	**0,11**	10	YES
210	82,3	98	**-0,38**	50	NO
3175	88,30	IS	**-0,09**	10	YES

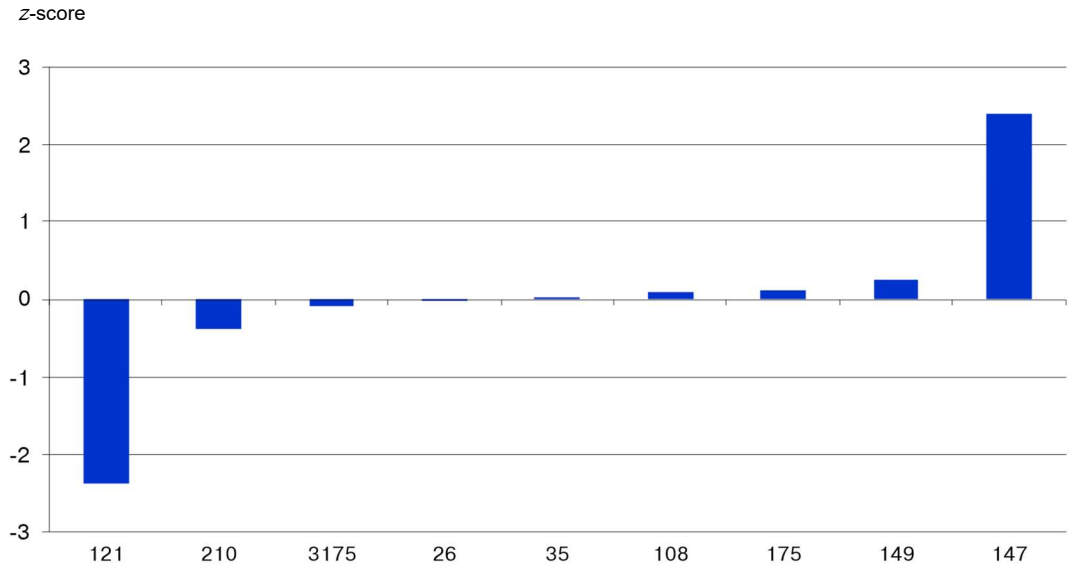
Test material H3703, honey

Concentration, arithmetic mean	n= 9	90,24	ug/Kg
Standard deviation	n= 9	25,01	ug/Kg
Coefficient of variation	n= 9	27,71	%

Table 48: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u(x_{pt})$	b	σ_{pt}
thiacloprid	9	90,17	7,69	8,53	2,56	0,23	20,72

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material H3703, honey

Table 49: results of analyses performed on test material H3703 (spiked sample): pesticides detection.

H3703 thiamethoxam, assigned value: 141,19 µg/kg satisfactory range: 80,53 – 201,86 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
09	NS	-		-	-
26	120	>90	**-0,70**	<10	NO
35	144,16	IS	**0,10**	10	-
82	<0,00	-		-	NO
108	149,00	107	**0,26**	10	NO
121	75,66	IS	**-2,16**	5	YES
147	220	132	**2,60**	10	NO
149	145,08	99,90	**0,13**	5,0	NO
175	153,85	IS	**0,42**	10	YES
210	137,2	94,6	**-0,13**	50	NO
3175	139,07	IS	**-0,07**	10	YES

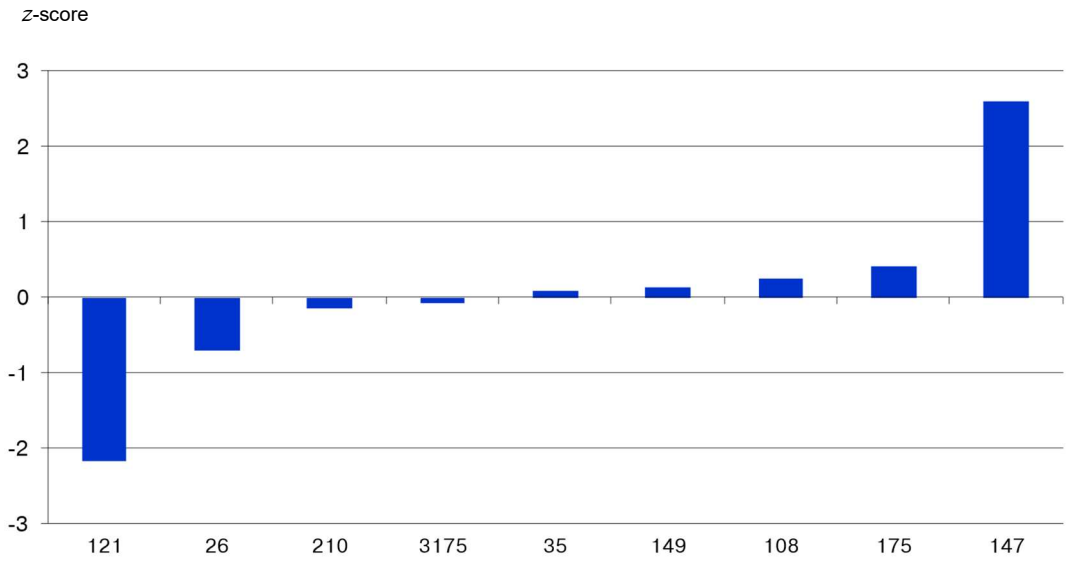
Test material H3703, honey

Concentration, arithmetic mean	n= 9	142,67	ug/Kg
Standard deviation	n= 9	37,42	ug/Kg
Coefficient of variation	n= 9	26,23	%

Table 50: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u(x_{pt})$	b	σ_{pt}
thiamethoxam	9	141,19	20,33	14,40	6,78	0,21	30,33

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material H3703, honey

Table 51: other compounds detected in the test material.

	H3703
Lab. code	**Other compounds (µg/kg)**
09	none
26	none
35	amitraz (sum of amitraz and all metabolites containing the 2,4-DMA moiety) (101,75) DMPF (N-2,4-dimethylphenylformamide) (50,35) DMF (N-2,4-dimethylformamide) (31,60) glyphosate (141,12)
82	none
108	none
121	none
147	DMPF (N-2,4-dimethylphenylformamide) (24)
149	none
175	none
210	none
3175	none

Proficiency test N3704, result contents

pesticides in pistachio;

- **Test material N3704**

Test material N3704, pistachio

Table 52: results of analyses performed on test material N3704: pesticides detection.

N3704 acetamiprid, assigned value: 27,03 µg/kg satisfactory range: 12,14 – 41,93 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
10	24	-	**-0,41**	10	-
21	33	103	**0,80**	10	NO
76	33,00	88	**0,80**	10	NO
137	23,9	84	**-0,42**	<10	NO
196	23	96,9	**-0,54**	<5,0	NO
213	40	102	**1,74**	10	-
231	20	IS70	**-0,94**	10	NO
232	17,2	76,38	**-1,32**	5,0	NO
2192	422,4	92	**53,09**	0,01	NO
2213	30	90	**0,40**	10	YES

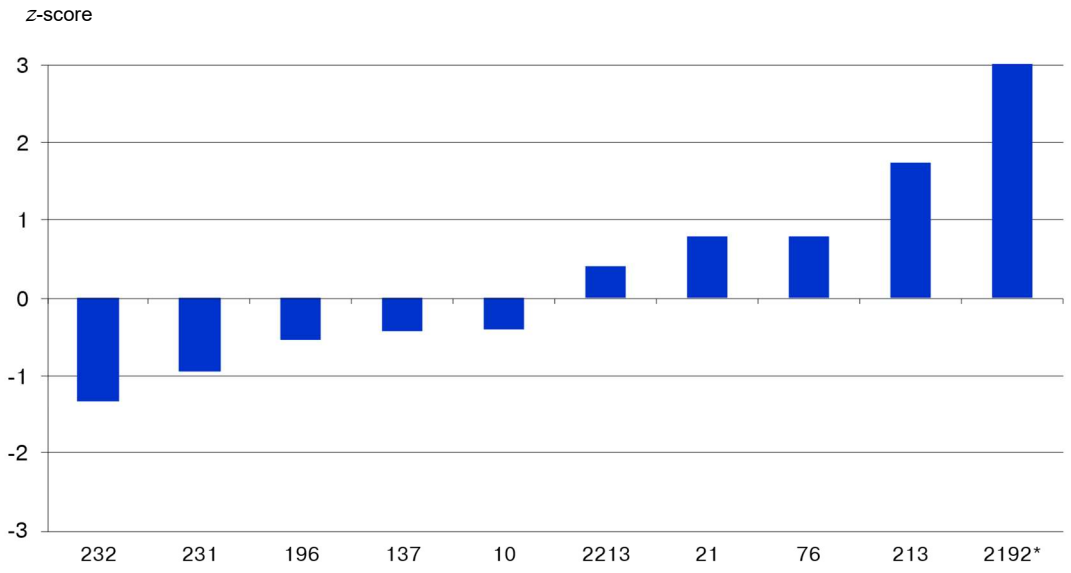
Test material N3704, pistachio

Concentration, arithmetic mean	n= 10	66,65	ug/Kg
Standard deviation	n= 10	125,19	ug/Kg
Coefficient of variation	n= 10	187,83	%

Table 53: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u(x_{pt})$	b	σ_{pt}
acetamiprid	9	27,03	8,11	29,99	2,70	0,28	7,45

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material N3704, pistachio

Table 54: results of analyses performed on test material N3704: pesticides detection.

N3704 clotiadin, assigned value: *45,64* µg/kg satisfactory range: *20,30 – 70,98* µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z'*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
10	45	-	***-0,05***	10	-
21	54	80	***0,66***	10	NO
76	57,00	90	***0,90***	10	NO
137	46,4	103	***0,06***	<10	NO
196	58	83,1	***0,98***	<5,0	NO
213	60	100	***1,13***	10	-
231	25	IS70	***-1,63***	10	NO
232	25,4	104,7	***-1,60***	5,0	NO
2192	NS	-		-	-
2213	40	95	***-0,45***	10	YES

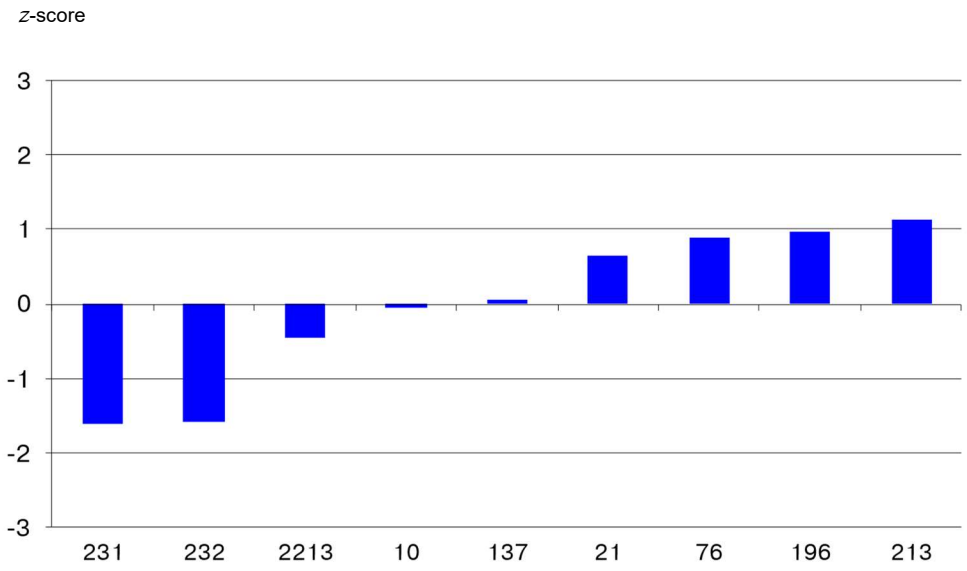
Test material N3704, pistachio

Concentration, arithmetic mean	n= 9	45,64	ug/Kg
Standard deviation	n= 9	13,35	ug/Kg
Coefficient of variation	n= 9	29,25	%

Table 55: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
clotiadin	9	*45,64*	*15,13*	33,16	5,04	0,25	12,67

$0.1 < [u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.5$



* outliers

Test material N3704, pistachio

Table 56: results of analyses performed on test material N3704: pesticides detection.

N3704 imidacloprid, assigned value: 33,68 µg/kg satisfactory range: 15,72 – 51,63 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
10	37	-	**0,37**	10	-
21	36	102	**0,26**	10	NO
76	35,00	96	**0,15**	10	NO
137	32,9	88	**-0,09**	<10	NO
196	30	99,4	**-0,41**	<5,0	NO
213	NS	-		-	-
231	36	IS70	**0,26**	10	NO
232	17,7	74,61	**-1,78**	5,0	NO
2192	583,2	92	**61,22**	0,01	NO
2213	34	85	**0,04**	10	YES

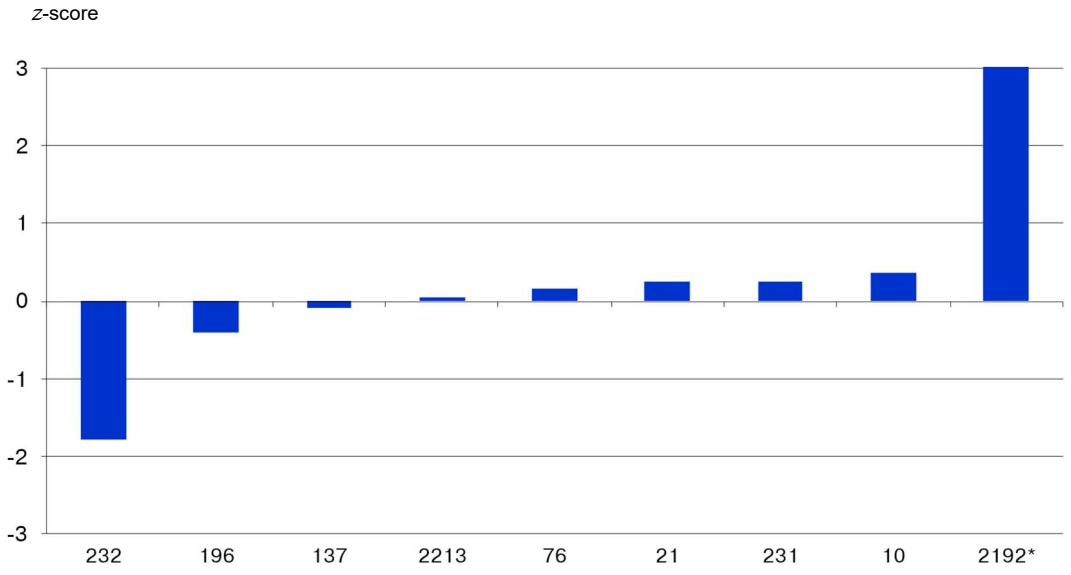
Test material N3704, pistachio

Concentration, arithmetic mean	n= 11	93,53	ug/Kg
Standard deviation	n= 11	183,72	ug/Kg
Coefficient of variation	n= 11	196,42	%

Table 57: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u\ (x_{pt})$	b	σ_{pt}
imidacloprid	8	33,68	3,44	10,21	1,22	0,27	8,98

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

Test material N3704, pistachio

Table 58: results of analyses performed on test material N3704: pesticides detection.

N3704 thiametoxam, assigned value: 15,15 µg/kg satisfactory range: 6,04 – 24,26 µg/kg					
Lab.code	**Result (µg/kg)**	**Recovery (µg/kg)**	***z*-score**	**LOQ (µg/kg)**	**Has the result been corrected by recovery?**
10	18	-	**0,63**	10	-
21	19	98	**0,85**	10	NO
76	22,00	85	**1,50**	10	NO
137	13,7	89	**-0,32**	<10	NO
196	14	98,4	**-0,25**	<5,0	NO
213	NS	-		-	-
231	15	IS70	**-0,03**	10	NO
232	9,1	92,1	**-1,33**	5,0	NO
2192	NS	-		-	-
2213	10,4	98	**-1,04**	10	YES

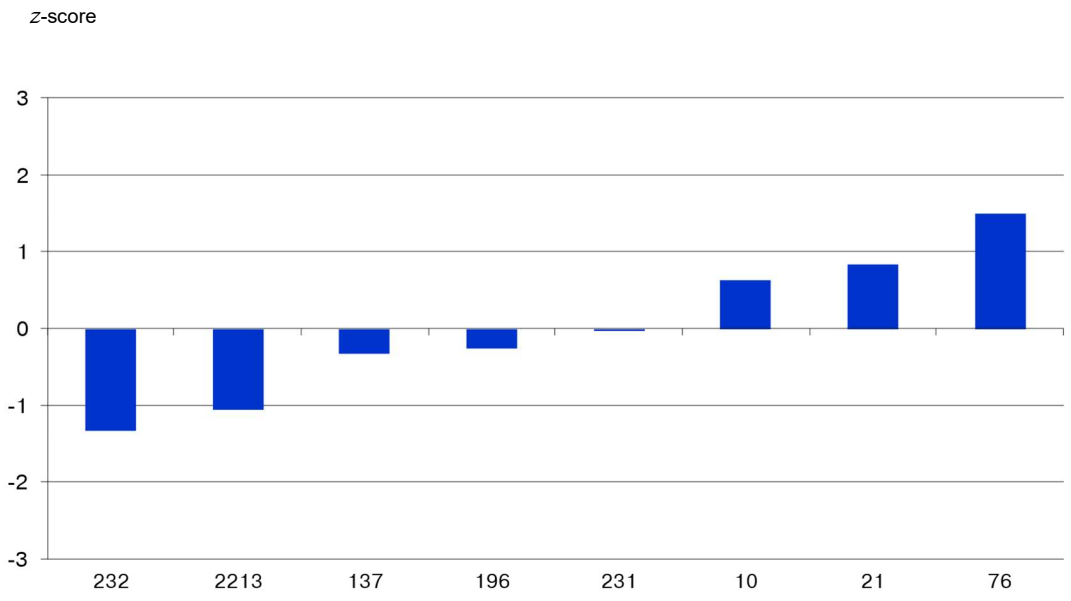
Test material N3704, pistachio

Concentration, arithmetic mean	n= 8	15,15	ug/Kg
Standard deviation	n= 8	4,35	ug/Kg
Coefficient of variation	n= 8	28,72	%

Table 59: assigned value and target standard deviations.

Analyte	Assigned value ug/Kg				Standard deviation for proficiency assessment		
	Data points, n	Mean from Algorithm A	SD	CV (%)	Standard uncertainty $u(x_{pt})$	b	σ_{pt}
thiametoxam	8	15,15	4,93	32,55	1,74	0,30	4,55

$[u(x_{pt})]^2 / \sigma_{pt}^2 \leq 0.1$



* outliers

ANNEX

ANNEX

ANALYTICAL METHODS USED

PESTICIDES IN LETTUCE, VF3700

Analytical methods used by participants:

pesticides	
Is the method already accredited?	**Laboratory Code**
NO	83
YES	09-16-26-136-153-169-176-177-180-187-188-190-213-2213

pesticides	
Analytical methods used:	**Laboratory Code**
GC-ECD, GC-NPD, HPLC-FL	180
GC-MS/MS, LC-MS/MS	16
GC/MS	09-136
GC/MS/MS	83-169
Gc/MS/MS	190
HPLC/MS/MS	26-153-169-177-187-190-213-2213
LC-M/MS and GC-MS/MS	188
LC-MS/MS and GC/MS/MS	176

pesticides	
Is the analytical method a routine one?	**Laboratory Code**
YES	16-26-83-136-153-169-176-177-180-187-188-190-213-2213

pesticides	
Do you participate regularly to pesticides proficiency test (PT)?	**Laboratory Code**
Once every four years	136
Yes: more than once per year	16-26-153-176-177-187-188-2213
Yes: one time per year	83-169-180-190-213

Quality:

pesticides	
Quality control implemented	**Laboratory Code**
Control chart	83-176-177-188-213-2213
Control chart, partecipation to PT, spiked samples, duplicate analysis, analysis of blank samples	136
Matrix Blank	190
Spiked sample	16-26-180-187-190

Number of replicates (1 replicate = 1 extraction):

pesticides	
Number of extraction	**Laboratory Code**
1	83-188-213-2213
2	16-26-177-187-190
3	136-176-180

pesticides	
Source of standards	**Laboratory Code**
Accustandard	190-213-2213
Agilent	136
LAb instrument	187
Lab instruments	16
RESTEK	26
Restek	83
Sigma-Aldrich	180
dr. Ehrenstorfer	176-177-188

	pesticides
Percentage of recovery measured in the same analytical run than test material?	**Laboratory Code**
NO	83-187
YES	16-26-136-176-177-180-188-190-213-2213

	pesticides
Blank sample used for spiking	**Laboratory Code**
Blank sample supplied by PT provider	16-26-83-176-177-188-190
Blank supplied by PT provider	136
Leccuce not apple	190
VF3700 Blank	213-2213
lettuce	180

	pesticides
Reference:	**Laboratory Code**
AOAC 2007.01 2013	136
BS Method	26
EN 15662:2008	176
Internal method	16-83-177-180-188-213-2213
Quechers	190

	pesticides
Amount of test sample for the extraction, after reconstitution (g):	**Laboratory Code**
5	177-190
10	16-26-176-180-188-213-2213
15	136

	pesticides
Extraction solvent	**Laboratory Code**
acetonitrile	16-26-83-136-176-177-180-187-188-190-213-2213

	pesticides
Clean up	**Laboratory Code**
QuEChERS	16-26-83-136-180-187-188-190
SPE	176-177-213-2213

	pesticides
pH adjusted	**Laboratory Code**
NO	16-83-136-176-180-187-188
YES	26-177-190

PESTICIDES IN SALMON, SF3701

Analytical methods used by participants:

	pesticides
Is the method already accredited?	**Laboratory Code**
NO	83-89-98-189
YES	02-18-21-103-124-146-147-168-200

	pesticides
Analytical methods used:	**Laboratory Code**
GC-ECD	168
GC/MS	146
GC/MS/MS	02-18-21-83-89-189
GC/MS/MS and LC/MS/MS	147
GC/MS/MS and UPLC/MS/MS	200
HPLC-MS/MS and GC-MS/MS	124
HPLC/MS/MS	98-103

	pesticides
Is the analytical method a routine one?	**Laboratory Code**
NO	98
YES	02-18-21-83-89-103-124-146-147-168-189-200

pesticides	
Do you participate regularly to pesticides proficiency test (PT)?	**Laboratory Code**
Not for this matrixgroup	98
Yes: more than once per year	21-89-103-124-146-189-200
Yes: one time per year	02-18-83-147
one time per four years	168

Quality:

pesticides	
Quality control implemented	**Laboratory Code**
Blank sample	21
Control chart	18-83-89-147-200
Spiked sample	02-98-103-124-146-168-189

Number of replicates (1 replicate = 1 extraction):

pesticides	
Number of extraction	**Laboratory Code**
OTHER	189
1	02-18-83-89-103-124
2	21-146-147-168-200
4	189

pesticides	
Source of standards	**Laboratory Code**
LGC	124-200
Lab Service	02
Restek	83
all suppliers mentioned	103
dr. Ehrenstorfer	18-21-89-168

pesticides	
Percentage of recovery measured in the same analytical run than test material?	**Laboratory Code**
NO	83-200
YES	02-18-21-89-103-168-189

pesticides	
Blank sample used for spiking	**Laboratory Code**
Blank sample supplied by PT provider	18-21-83-103-189
Internal blank	200
Salmon SF3701 blanc	89
routine sample	168

pesticides	
Reference:	**Laboratory Code**
In house method	21
Internal method	02-18-83-89-103-168-200

pesticides	
Amount of test sample for the extraction, after reconstitution (g):	**Laboratory Code**
OTHER	21
1	21
5	02-18-103-168-189-200
10	83-89

pesticides	
Extraction solvent	**Laboratory Code**
acetone, dichloromethane en petroleumether	21
acetonitrile	02-18-83-89-103-168-189-200

pesticides	
Clean up	**Laboratory Code**
QuEChERS	83-89-103-168-189-200
SPE	02-18
Thermal clean-up and florisil	21

pesticides	
pH adjusted	**Laboratory Code**
NO	18-21-83-89-103-168-200
YES	02-189

FIPRONIL AND OTHER PESTICIDES IN EGGS, E3702

Analytical methods used by participants:

fipronil and other pesticides	
Is the method already accredited?	**Laboratory Code**
NO	09-83-91
YES	02-21-32-57-78-82-85-146-147-162-197-257-332

fipronil and other pesticides	
Analytical methods used:	**Laboratory Code**
GC-MSMS LC-MSMS	91
GC/MS	82-83
GC/MS& LC/MS/MS	146
GC/MS/MS	02-09-134-332
GC/MS/MS and LC/MS/MS	21-147
HPLC and GC-MS/MS	57-257
HPLC/MS	32
HPLC/MS/MS	78-85-134-162-197

fipronil and other pesticides	
Is the analytical method a routine one?	**Laboratory Code**
YES	21-32-57-78-82-83-85-91-146-162-197-257-332

fipronil and other pesticides	
Do you participate regularly to pesticides proficiency test (PT)?	**Laboratory Code**
Yes: more than once per year	02-21-82-91-146-332
Yes: one time per year	32-57-78-83-85-147-162-197-257

Quality:

fipronil and other pesticides	
Quality control implemented	**Laboratory Code**
Blank sample	21
Control chart	32-83-147-332
Spiked sample	02-57-78-82-85-146-162-197-257
and RM	162

Number of replicates (1 replicate = 1 extraction):

fipronil and other pesticides	
Number of extraction	**Laboratory Code**
1	02-57-83-85-197-257
2	21-32-82-146-147
3	162-332
5	78

fipronil and other pesticides	
Source of standards	**Laboratory Code**
2AS	02
LGC	85
Restek	83
Sigma-Aldrich	32-82
dr. Ehrenstorfer	21-57-78-162-257-332

fipronil and other pesticides	
Percentage of recovery measured in the same analytical run than test material?	**Laboratory Code**
NO	32-83-332
YES	02-21-57-78-85-162-197-257

fipronil and other pesticides	
Blank sample used for spiking	**Laboratory Code**
Blank provided	21
Blank sample supplied by PT provider	32-78-83-332
egg	02
in house	57-257
organic egg	162

fipronil and other pesticides	
Reference:	**Laboratory Code**
Internal method	02-21-32-57-78-83-162-197-257-332
OTHER	85
QuEChERS METHI European EN method	82

fipronil and other pesticides	
Amount of test sample for the extraction, after reconstitution (g):	**Laboratory Code**
5	02-32-78-162-332
10	83-85

fipronil and other pesticides	
Extraction solvent	**Laboratory Code**
Acetone/Dichlormethane/Petroleum ether	21
acetonitrile	02-32-78-83-85-162-197
diethyl ether:hexane	57-257
ethyl acetate + cyclohexane 1:1	332

fipronil and other pesticides	
Clean up	**Laboratory Code**
GPC	332
QuEChERS	02-32-78-83-85-162
SPE	57-82-197-257
Thermal cleun up, florisil	21

fipronil and other pesticides	
pH adjusted	**Laboratory Code**
NO	21-32-57-78-83-85-162-197-257-332
YES	02-82

PESTICIDES IN HONEY, H3703

Analytical methods used by participants:

pesticides	
Is the method already accredited?	**Laboratory Code**
NO	26-121-149-175-210-3175
YES	09-35-82-108-147

pesticides	
Analytical methods used:	**Laboratory Code**
GC-MS/MS + LC-MS/MS	3175 – 175 - 147
GC/MS	82
GC/MS/MS	09-35
HPLC/MS/MS	26-35-108-121-149
in house method	210

pesticides	
Is the analytical method a routine one?	**Laboratory Code**
NO	210
YES	26-35-82-108-121-147-149-175-3175

	pesticides
Do you participate regularly to pesticides proficiency test (PT)?	**Laboratory Code**
Yes: more than once per year	82-147-210-3175
Yes: one time per year	26-35-121-149-175
once per 4 years	108

Quality:

	pesticides
Quality control implemented	**Laboratory Code**
Control chart	147-175-3175
Duplicate analyses	35
Spiked sample	26-82-108-149-210

Number of replicates (1 replicate = 1 extraction):

	pesticides
Number of extraction	**Laboratory Code**
1	121-149
2	26-35-82-108-147-175-210-3175

	pesticides
Source of standards	**Laboratory Code**
LGC	3175
LGC, Sigma Aldrich, Dr. Ehrenstorfer	175
OTHER	108-147
RESTEK	26 - 210
Sigma-Aldrich	82
dr. Ehrenstorfer	35-149

	pesticides
Percentage of recovery measured in the same analytical run than test material?	**Laboratory Code**
YES	26-108-121-147-149-175-210

	pesticides
Blank sample used for spiking	**Laboratory Code**
Blank sample supplied by PT provider	26-108-149-175-3175
HONEY	35
In-house blank honey	147
Local	121
OTHER	82
The blank provided	210

	pesticides
Reference:	**Laboratory Code**
QuEChERS European EN method	82
BS Method	26
Internal method	35-121-147-149-175-210-3175
QUECHERS	108

	pesticides
Amount of test sample for the extraction, after reconstitution (g):	**Laboratory Code**
5	26-35-108-121-147-149-175-210-3175
10	82

	pesticides
Extraction solvent	**Laboratory Code**
acetonitrile	26-35-82-108-121-147-149-175-210-3175

	pesticides
Clean up	**Laboratory Code**
QuEChERS	26-35-82-108-147-149-175-210-3175

	pesticides
pH adjusted	**Laboratory Code**
NO	35-108-121-147-149-175-210-3175
YES	26-82

PESTICIDES IN PISTACHIO, N3704

Analytical methods used by participants:

	pesticides
Is the method already accredited?	**Laboratory Code**
NO	10-196
YES	21-76-137-192-213-231-232-2192-2213

	pesticides
Analytical methods used:	**Laboratory Code**
GC/MS/MS	137
HPLC/MS	196
HPLC/MS/MS	10-21-137-192-213-231-232-2192-2213
LC-QTOF	76

	pesticides
Is the analytical method a routine one?	**Laboratory Code**
NO	196
YES	10-21-76-137-192-213-231-232-2192-2213

	pesticides
Do you participate regularly to pesticides proficiency test (PT)?	**Laboratory Code**
EUPT from EURL	137
No: not regularly	10
Yes: more than once per year	21-137-192-232-2213
Yes: one time per year	76-196-213-231-2192

Quality:

	pesticides
Quality control implemented	**Laboratory Code**
Blank sample	21
Control chart	192-213-2192-2213
Control chart with Certified Reference Material (CRM)	232
Regular partecipation to proficency test (PT)	196
Spiked sample	76-137-231

Number of replicates (1 replicate = 1 extraction):

	pesticides
Number of extraction	**Laboratory Code**
1	137-192-213-231-2192-2213
2	21-196-232
3	76

	pesticides
Source of standards	**Laboratory Code**
Accustandard	213-2213
CPA Chem	196
HPC/Germany	232
LGC	2192
LGC STANDARDS	137
OTHER	192
Restek LC multi residue pesticide kit	231
Sigma-Aldrich	137
dr. Ehrenstorfer	21-76

	pesticides
Percentage of recovery measured in the same analytical run than test material?	**Laboratory Code**
NO	2192
YES	21-76-137-192-196-213-231-232-2213

pesticides	
Blank sample used for spiking	**Laboratory Code**
Blank pistachio	232
Blank provided	21
Blank sample supplied by PT provider	76
N3704 Blank	213-2213
OTHER	192-231-2192
PISTACHIO	137
Pistacchi biologici	196

pesticides	
Reference:	**Laboratory Code**
BS EN 15662	231
Internal method	21-76-137-192-213-232-2213
OTHER	2192
UNI EN 15662:2018	196

pesticides	
Amount of test sample for the extraction, after reconstitution (g):	**Laboratory Code**
1	21
2	213-2213
5	76-192-196-231-2192
7,5	232
15	137

pesticides	
Extraction solvent	**Laboratory Code**
Acetone, Dichlormethane, Petroleum ether	21
acetonitril with 1% acetic acide	232
acetonitrile	76-137-192-196-213-231-2192-2213

pesticides	
Clean up	**Laboratory Code**
QuEChERS	76-137-192-196-231-232-2192
SPE	213-2213
Thermal cleanup	21

pesticides	
pH adjusted	**Laboratory Code**
NO	21-137-192-196-232-2192
YES	76-231

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