

### relana<sup>®</sup> Communication Note 25-01

Ultra Low Levels of Pesticides in Organic Products (UPOP)

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c/o Lach & Bruns Partnerschaft Tempowerkring 6 21079 Hamburg Germany

relana@relana-online.com www.relana-online.com

#### 1. Introduction [1]

Since the current Organic Basic Regulation (EU) 2018/848 entered into force on 17 June 2018, the concept of the presence of unauthorised substances, products and processes as *a trigger threshold* for a reasonable suspicion and thus for suspensions and investigations has been the subject of repeated discussions.

The regulation has been in practical application for more than three years, thus there is extensive experience in dealing with the presence of unauthorised substances in organic products. Although products and processes are also covered by the scope of the regulation, in most cases it is the presence of substances, or more precisely the pesticide findings, where the scepticism - that presence alone is not a reliable basis - has proven to be true.

In the opinion of many stakeholders (such as the EU Commission, organic control bodies and authorities), the presence of unauthorised substances (in general: pesticides) is given, if a positive analysis result is available usually in the unit *mg/kg*. Factors that are decisive for the reliability of an analysis usually play no role: Sampling, analytical detection sensitivities, informative value for the entire batch, sampling errors, etc. As a result, it happens here and then that large import batches are notified via OFIS (Organic Farming Information System) and thus blocked on the basis of a single private-law analysis of a company in the marketing chain, although there are no other anomalies. And even if negative representative analyses have been carried out during dispatch and import, they are not considered for the evaluation of the organic integrity.

The reporting limit for the detected substances is moving further and further down to lower concentration ranges. Private service laboratories can also reliably detect most substances in the range below 10  $\mu$ g/kg (< 0,01 mg/kg), and the step into the lower dimension of nanograms/kg is technically feasible (but not on a routine level). Even if there is no detection in the range of 1 to 10  $\mu$ g/kg, it is highly likely that numerous pesticides and environmental pollutants will be detected at 10 to 1000 nanograms/kg (0,00001 – 0,0001 mg/kg). This confirms in practice that there is no such thing as *'non-existence'* in theory. It must be stressed that laboratories must spend significantly more efforts, time and thus money to measure such ultra-low concentrations. Consequently, such analyses are not feasible under routine conditions.

A further problem arises if one assumes, without checking, that a presence always originates from a direct or indirect application on the surface or on the product. In the case of organic products, a direct or indirect application means a violation of the Regulation (EU) 2018/848. It has been proven many times that contamination or impurities from agrochemical substances can be found everywhere in the environment, even far away from agricultural land. It is all a question of analytical sensitivity.



#### 2. Background

As discussed in the Introduction, the approach of the Organic Basic Regulation (EU) 2018/848 to define the integrity of organically produced products using the term "presence of unauthorised substances" is not reasonable. Depending on the efforts analytical laboratories apply, in nearly every food product – independent of its' origin and the process during cultivation – it is possible to detect chemical substances, especially chemically synthesised pesticides, which are not permitted to be used in organic agriculture.

The relana<sup>®</sup> circle (relana = **rel**iable **ana**lysis) of 11 most experienced private analytical laboratories in pesticide testing of food products decided to provide evidence to public on that topic.

Common practice in commercial and scientific based pesticide residue testing is to follow the analytical guidelines of the document "*Analytical Quality Control and Method Validation Procedures for Pesticide Analysis in Food and Feed*" (current version: SANTE/11312/2021 v2) [2]. In this document, the terms to be used for reporting of analytical results are described. The definition of the "Reporting Limit" (RL) is:

"The lowest level at which residues will be reported as absolute numbers. It is equal to or higher than the LOQ."

The LOQ = limit of quantification is defined by:

"The lowest concentration or mass of the analyte that has been validated with acceptable accuracy by applying the complete analytical method and identification criteria."

Pesticide testing laboratories typically make use of these terms and definitions when reporting analytical results. It is common practice to use a RL of 0,01 mg/kg for all pesticides, except the related MRL (Maximum Residue Limit) of Regulation (EC) 396/2005 is below 0,01 mg/kg. In such cases, the RL have to be lower, f.ex. at 0,001 mg/kg.

As the analytical methods for sample preparation and clean up are becoming more and more effective, and as the analytical instrumentation to detect and quantify pesticides are as well more and more sensitive and sophisticated, laboratories are able to detect and quantify pesticides at much lower levels than before. However, to achieve such ultra-low levels, a lot of additional precautions and quality control steps must be applied. This is possible in general, but need much more time, much more additional workload and at the end it would need a much higher price compared to common analytical routine approaches. Therefore, this project just likes to show, that it is possible to detect and quantify levels of pesticides even at concentration levels of ng/kg (< 0,0001 mg/kg).

#### 3. Analytical approaches

The participating laboratories of the relana<sup>®</sup> circle agreed in advance to apply a multiresidue-method-approach only. Thus, this project does not include pesticides, which need a specific analytical method to be analysed (f.ex. the group of polar pesticides or the group of Dithiocarbamates).

9 laboratories applied modified versions of the QuEChERS method EN15662, while modified versions of the QuEChERS official method AOAC 2007.01 were applied by two of the laboratories. All labs modified their analytical approaches related to a more extended sample clean up and higher enrichments of the final extracts. Of course, it was necessary to optimise the analytical measuring systems and to tune the instrument parameters for maximum measuring sensitivity. Doing this, the laboratories were able to achieve quantification limits (LOQ) between 0,00001 mg/kg (10 ng/kg) and 0,001 mg/kg (1000 ng/kg resp. 1  $\mu$ g/kg) depending on the type of pesticide and the type of food product analysed. The detection and quantification of pesticides using these special measures is therefore at least 10 times lower than the usual reporting limit of 0.01 mg/kg and up to a factor of 1000 lower if 0.00001 mg/kg can be achieved.

#### 4. Results

The participating laboratories applied their advanced multi-residue-method (MRM) approach, as described above. These methods cover between 700 and 850 different pesticides, depending on the specific approaches of the laboratories.

The laboratories were asked to report

- Results applying the common MRM approach (QuEChERS multi methods, EN or AOAC version) including the common reporting limit (RL) of 10 μg/kg (0,01 mg/kg)
- b. Results applying a modified more sensitive MRM approach (modified QuEChERS multi methods, EN or AOAC version) with correspondingly lower reporting limits of 10 ng/kg (0,00001 mg/kg) up to 1000 ng/kg (1 µg/kg resp. 0,001 mg/kg).

This means, that the sensitivity and the related reporting limit are lowered by a factor of 1000 compared to common pesticide testing approaches.

In total, results of 203 samples of organic agriculture were reported. The food products cover mostly all kind of fresh fruits and vegetables (from A as Apple to Z as Zucchini), so typical primary agriculture products. Thus, the big majority of the products were unprocessed without the risk of a contamination during possible processing of such products after harvesting. Just a small number of samples were processed (f.ex. strawberry purée, rice, tea, wheat) or are seeds (f.ex. sesame, sunflower, corn).



- Of all analysed 203 samples, 39 samples (19%) are still <u>without</u> any findings ≥ 10 ng/kg (0,00001 mg/kg).
- 164 samples (81%) are reported <u>with</u> findings  $\geq$  10 ng/kg (0,00001 mg/kg).
- Of the 164 samples with findings, 25 samples (12% of all 203 sample) are also reported with concentrations ≥ 0,01 mg/kg (10 µg/kg). In 15 of these 25 samples only pesticides are detected, which are allowed to be used in organic agriculture (f.ex. Azadirachtin, Spinosad, Pyrethrins).
  10 samples (4,9 % of all 203 samples) are reported with non-authorised substances ≥ 0,01 mg/kg (10 µg/kg).
- At least 160 samples (79% of 203 samples) are reported with pesticide findings between ≥ 10 ng/kg (0,00001 mg/kg) and < 10 µg/kg (0,01 mg/kg). This indicates that a substantial majority of samples of organic agriculture are affected resp. contaminated by pesticides.

The pie chart below illustrates the results formulated above. Note: The total number (224) is higher than the number of samples analysed (203). This is due to the fact that some samples appear in several categories.



The evaluations resp. summaries are attached at the end of this document (tables 1 and 2). The detailed results of all 203 samples are presented in a separate document (Table 3: "ALL\_UPOP\_Results.pdf).



#### 5. Summary and Conclusions

While applying advanced analytical techniques of multi-residue-method approaches to lower the sensitivity by a factor between 10 and 1000 compared to the common routine pesticide testing approaches, it is obviously, that in the huge majority of samples of organic food products, pesticides ("unauthorised substances") can be detected and quantified.

The laboratories were able to identify and quantify pesticides in 160 out of 203 food products at levels between 0,00001 mg/ kg (10 ng/kg) and 0,001 mg/kg (1  $\mu$ g/kg), which is equivalent to 79% of the analysed samples. When applying the common analytical routine multi-residue-method approaches, these 160 food products would have been reported without any pesticide findings.

25 samples (12 %) were reported with findings at or above the common LOQ of 0,01 mg/kg, and 10 of these 25 samples are reported having "unauthorised" substances (5%) above the common LOQ of 0,01 mg/kg. This does not straightforwardly conclude that these products do not meet the requirements of Regulation (EU) 2018/848.

Of the 203 analysed samples, 39 are without any positive findings even if applying the advanced analytical approaches. This is equivalent to 19% of the samples.

Taking a closer look on some of the most important fruits and vegetables (in terms of trading volume), it can be seen that either all samples (apples, carrots, grapes, strawberries) or the vast majority were reported with pesticide levels between 0,00001 mg/ kg (10 ng/kg) and 0,001 mg/kg (1  $\mu$ g/kg):

#### Sample results of selected important fruits and vegetables

Total number of samples: 97 Minimum number of samples analysed: 5

Commodity group	Total No. of Samples	No. of Samples ≥ <b>0,01 mg/kg</b> (≥ 10 µg/kg)	No. of Samples ≥ <b>0,00001mg/kg</b> (≥ 0,01 µg/kg)	No. of Samples without any detection *
Apples	8	0	8 (100%)	0
Bananas	26	3	24 (92%)	2
Carrots	9	0	9 (100%)	0
Grapes	5	1	5 (100%)	0
Nectarines	6	1	5 (83%)	1
Oranges	5	0	4 (80%)	1
Paprika =	5	0	4 (80%)	1
Sweet pepper				
Peaches	9	2	7 (78%)	2
Pears	7	0	6 (86%)	1
Strawberries	5	1	5 (100%)	0
Tomatoes	12	2	8 (67%)	4
TOTAL	97	10	85	12

\* Various reporting limits between 0,00001 mg/kg (10 ng/kg) and 0,001 mg/kg (1 μg/kg) depending on pesticides and commodities

85 of 97 samples (88%) of the selected main crops of the group of fruits and vegetables were reported with pesticide levels between 0,00001 mg/ kg (10 ng/kg) and 0,001 mg/kg (1  $\mu$ g/kg).

As a significant majority of 160 samples (79%) of the total number of 203 samples is contaminated with pesticides at such low levels (between 0,00001 mg/ kg (10 ng/kg) and 0,001 mg/kg (1  $\mu$ g/kg)), the descriptions in art. 28 paragraph 2 of reg. (EU) 2018/848 [3]:

"... the **presence of a** product or **substance** that is not authorised pursuant to the first subparagraph of Article 9(3) for use in organic production ..."

resp. the description in Art. 29 paragraph 1:

"... receives substantiated information about the **presence of** products or **substances** that are not authorised pursuant to the first subparagraph of Article 9(3) for use in organic production, or has been informed by an operator in accordance with point (d) of Article 28(2), or **detects such** products or **substances** in an organic or an in-conversion product ..." (bold letters by the authors),

are not suitable as a basis for justifying possible non-conformities with the requirements of this Regulation.

From the analytical point of view, it is to be noted, that

- Limits of detection and quantification are variable, depending on the applied analytical approach and the technical instruments available.
- Limits of detection and quantification are variable, depending on the aim of the requested analysis.
- Substances applied over a long period of time are omnipresent.
- A "Zero" concentration (level) does not exist.
- Substances are in principle also present below the analytical limits of detection resp. quantification.

It must be concluded, that depending on the

- o technical capabilities of pesticide testing laboratories,
- o additional efforts applied compared to routine approaches, and
- willingness of laboratories' clients to pay for these additional efforts

it is possible to identify and quantify "unauthorised" substances in mostly every food product, independent how this was produced resp. cultivated.

Therefore, it is recommended, not to focus on the "presence" or "detection" of unauthorised substances in organic products as a hint for possible non-conformities without any suspicion in advance, but instead to use pesticide analysis as an important tool to substantiate suspicious cases that have arisen during the production process of biologically produced products, monitored by the competent authorities or organic control bodies.

#### References

- [1] Martin Rombach: Lecture at BioFach fair, Nuremberg, Germany, 11. February 2025 "Vorhandensein und Verdacht – Ein Ausweg aus der Rückstandsfalle", translated: "Presence and suspicion - a way out of the residue trap"
- [2] "Analytical Quality Control and Method Validation Procedures for Pesticide Analysis in Food and Feed" (current version: SANTE/11312/2021 v2)
- [3] Regulation (EU) 2018/848

#### Participating laboratories in this project

Analytica Alimentaria GmbH, Sucursal en España, Almería, Spain

Analytica Alimentaria GmbH, Kleinmachnow, Germany

Analytisches Institut Bostel GmbH & Co.KG, Stuttgart, Germany

bilacon GmbH, Berlin, Germany

Eurofins Dr. Specht Express Testing & Inspection GmbH, Hamburg, Germany

Eurofins Dr. Specht Laboratorien GmbH, Hamburg, Germany

GALAB Laboratories GmbH, Hamburg, Germany

Greit s.r.l., Bologna, Italy

Labor Friedle GmbH, Tegernheim, Germany

PRIMORIS BELGIUM CV, Zwijnaarde, Belgium

VELTIA S. A., Thessaloniki Lab, Sindos, Greece

#### relana<sup>®</sup> communication note

This communication note is a literary property of relana<sup>®</sup>, based on the contribution and the knowledge of the members of the relana<sup>®</sup> laboratory quality circle. The aim of this publication is to increase knowledge and to provide expertise to all relevant and interested stakeholders to achieve best practices on analytical services related to food and feed testing. Everybody is invited to make use of this communication note and to circulate it wherever meaningful.



#### Table 1

**Sample results** (no individual samples but summarised in commodity groups) Total number of samples: 203

Commodity	Total No.	No. of Samples	No. of Samples	No. of
group	of	≥ 0,01 mg/kg	≥ 0,00001mg/kg	Samples
	Samples	(≥ 10 µg/kg)	(≥ 0,01 µg/kg)	without any
				detection *
Apples	8	0	8	0
Apricots	4	0	3	1
Artichokes	1	0	0	1
Asparagus	2	0	2	0
Aubergines	2	0	1	1
Avocados	3	0	3	0
Baby leaf	1	1	1	0
salad				
Bananas	26	3	24	2
Basil	1	1	1	0
Blackberries	3	0	1	2
Blueberries	4	0	3	1
Broccoli	2	1	2	0
Cabbage	1	0	0	1
Cashews	2	0	1	1
Carrots	9	0	9	0
Celery	1	0	1	0
Chard (Swiss)	3	0	3	0
Cherries	1	0	1	0
(sour)				
Cherries	4	1	4	0
(sweet)				-
Chillies	2	0	2	0
Chives	1	0	0	1
Corn (maize)	1	0	0	1
Cucumber	3	1	1	1
Elderberries	1	0	0	1
Fennel	1	0	1	0
Garlic	3	0	1	2
Ginger	2	0	2	0
Goji berries	1	1	1	0
Grapes	5	1	5	0
Kale	1	0	1	0
Lamb's lettuce	2	0	2	0
Lettuce	5	1	2	2
Lemon	3	1	3	0
Limes	1	0	1	0
Mandarins	2	1	1	0
Mango	3	0	3	0
Melon	1	0	1	0

Commodity group	Total No. of	No. of Samples ≥ 0,01 mg/kg	No. of Samples ≥ <b>0,00001mg/kg</b>	No. of Samples
	Samples	(≥ 10 µg/kg)	(≥ 0,01 µg/kg)	without any detection *
Nectarines	6	1	5	1
Onions	2	0	1	1
Oranges	5	0	4	1
Oregano	1	0	1	0
Pak Choi	1	0	1	0
Paprika	5	0	4	1
(Sweet				
pepper)				
Parsley	1	0	1	0
Peaches	9	2	7	2
Pears	7	0	6	1
Peas	1	0	0	1
Potatoes	4	0	2	2
Pumpkin	3	0	3	0
Rice	1	0	1	0
Rocked Salad	4	0	3	1
(Rucola)				
Sesame seed	1	0	0	1
Spinach	2	1	2	0
Spring onions	2	0	2	0
Strawberries	5	1	5	0
Strawberry	1	1	1	0
Sunflower	1	0	0	1
seeds		U	U	1
Теа	1	1	0	0
Tomatoes	12	2	8	4
Turmeric	1	1	1	0
Watermelon	3	0	3	0
Wheat	2	0	0	2
Zucchini	6	2	4	2
(Courgette)		_		_
TOTAL	203	25	160	39

 $^*$  Various reporting limits between 0,00001 mg/kg (10 ng/kg) and 0,001 mg/kg (1  $\mu g/kg)$  depending on pesticides and commodities

#### Table 2

#### Samples with results $\geq$ 0,01 mg/kg (10 µg/kg)

(no individual samples but summarised in commodity groups) Total number of samples: 203 Pesticides: GREEN background = permitted to be used in organic agriculture

RED background = NOT permitted to be used in organic agriculture

Commodity	Total No.	No. of	Pesticides	Level
group	of	Samples		(mg/kg)
	Samples	≥ 0,01 mg/kg		
Apples	8	0		
Apricots	4	0		
Artichokes	1	0		
Asparagus	2	0		
Aubergines	2	0		
Avocados	3	0		
Baby leaf salad	1	1	Spinosad	0,074
Bananas	26	3	<mark>Bifenthrin,</mark> Spinosad	0,013 resp. 0,011 (2x)
Basil	1	1	Azadirachtin, Pvrethrins	0,054 resp. 0.038
Blackberries	3	0		,
Blueberries	4	0		
Broccoli	2	1	Fludioxonil	0,020
Cabbage	1	0		
Cashews	2	0		
Carrots	9	0		
Celery	1	0		
Swiss Chard	3	0		
Cherries	1	0		
(sour)				
Cherries	4	1	Azadirachtin,	0,046 resp.
(sweet)			Spinosad	0,53
Chillies	2	0		
Chives	1	0		
Corn (maize)	1	0		
Cucumber	3	1	Sulfoxaflor	0,023
Elderberries	1	0		
Fennel	1	0		
Garlic	3	0		
Ginger	2	0		
Gojiberries	1	1	Acetamiprid	0,019
Grapes	5	1	Spinosad	0,016
Kale	1	0		
Lamb's lettuce	2	0		
Lettuce	5	1	Spinosad	0,064
Lemon	3	1	Propyzamide	0,014
Limes	1	0		

Commodity	Total No.	No. of	Pesticides	Level
group	of	Samples		(mg/kg)
	Samples	≥ 0,01 mg/kg		
Mandarins	2	1	Pyriproxyfen	0,064
Mango	3	0		
Melon	1	0		
Nectarines	6	1	Spinosad	0,011
Onions	2	0		
Oranges	5	0		
Oregano	1	0		
Pak Choi	1	0		
Paprika	5	0		
(Sweet				
pepper)				
Parsley	1	0		
Peaches	9	2	Pyrethrins,	0,011 resp.
			Spinosad	0,016
Pears	7	0		
Peas	1	0		
Potatoes	4	0		
Pumpkin	3	0		
Rice	1	0		
Rocked Salad	4	0		
(Rucola)				
Sesame seed	1	0		
Spinach	2	1	Spinosad	0,31
Spring onions	2	0		
Strawberries	5	1	Dimethomorph,	0,024 resp.
			Fenhexamid,	1,5 resp.
			Fluopyram	0,013
Strawberry	1	1	Spinosad	0,024
purée				
Sunflower	1	0		
seeds				
Теа	1	1	Anthraquinone	0,11
Tomatoes	12	2	Spinosad	0,018 /
				0,022
Turmeric	1	1	Chlorpyrifos	0,028
Watermelon	3	0		
Wheat	2	0		
Zucchini	6	2	Azoxystrobin.	0,050 resp.
(Courgette)	_		Spinosad	0,018
TOTAL	203	25		

### Table 3

Detailed results

See separate file "ALL\_UPOP\_Results.pdf"