Presence and Suspicion

Can the lower analytical limit of quantification under reg. (EU) 396/2005 provide more legal certainty?

Dr. Günter Lach (Lach & Bruns Partnerschaft)
Martin Rombach (Prüfgesellschaft mbH)

Reg. (EC) No. 396/2005
article 3 section 2 - Definitions

***Maximum residue level (MRL)**:*

*The **maximum residue level** of a pesticide residue in or on food or feed is set in accordance with this Regulation on the basis of **good agricultural practice** and the **lowest level of human exposure** necessary to protect vulnerable consumers.*

The value is a **balance of interests** between the need for application in agricultural practice, and the protection of consumers.

Aspects for the development of limit values for organic products

- **Protecting consumers from deception**
- **Technical detection limits** for the active substance/product combination
- **Background level(s)** of the harvest at the place of production
- **Unavoidable contamination** during harvest, transport, storage, and processing
- **Degradation, dilution or concentration** of the active substance during processing

Threshold value and official investigation

Presence as trigger limit for the official investigation

→ Implementing Regulation (EU) 2021/279 Art. 2 sect. 3:

Main results of an official investigation are

- determination of the **integrity** of the organic product
- determination of **source** (origin) and **cause of presence**
- answers rel. to EU Reg. 2018/848 Art. 29 sect. 2: **Application? Precautionary measures? Response to previous controls?**

What does “Presence” mean?

The presence of **0.01 mg** glyphosate in (one)**1 kg** of wheat corresponds to a number of

35 quadrillion molecules in this kilogramme,

i.e. **35 x 10^{15} molecules**.

In digits: **35.000.000.000.000.000** molecules.

Non-authorised substances

lach : bruns

Pesticides Database - Active Substances
(File created on 07/02/2025)

ID	Substance	CAS Number	Status under Reg. (EC) No 1107/2009	Date of approval
472	Boscalid (formerly nicobifen)	188425-85-6	Approved	01/08/2008
473	Brandol (hydroxynonyl-2,6-dinitrobenzene)	No CAS allocated	Not approved	
474	Brodifacoum	56073-10-0	Not approved	
475	Bromacil	314-40-9	Not approved	
476	Bromadiolone	28772-56-7	Not approved	01/06/2011
477	Bromethalin	63333-35-7	Not approved	
478	Bromocyclen	1715-40-8	Not approved	
479	Bromofenoxim	13181-17-4	Not approved	
480	Bromophos	2104-96-3	Not approved	
481	Bromophos-ethyl	4824-78-6	Not approved	
482	Bromopropylate	18181-80-1	Not approved	
483	Bromoxynil	1689-84-5	Not approved	01/03/2005
484	Bromuconazole	116255-48-2	Approved	01/02/2011
1381	Bronopol	52-51-7	Not approved	
485	Bupirimate	41483-43-6	Approved	01/06/2011
486	Buprofezin	69327-76-0	Approved	01/02/2011

Non-authorised substances

Pesticides Database - Active Substances
(File created on 07/02/2025)

Active Substance ID	Substance	CAS Number	Status under Reg. (EC) No 1107/2009	Date of approval
505	Captan	133-06-02	Approved	01/11/2024
508	Carbofuran	1563-66-2	Not approved	

Non-authorised substances

Code	Products to which MRLs apply	Carbofuran (sum of carbofuran (including any carbofuran generated from carbosulfan, benfuracarb or furathiocarb) and 3-OH carbofuran expressed as carbofuran)(R) ℹ Regulation (EU) 2015/399 applicable Annex II, IIIB
0100000	FRUITS, FRESH or FROZEN; TREE NUTS	
0110000	Citrus fruits	**0.01***
0110010	Grapefruits	0.01*
0110020	Oranges	**0.01***
0110030	Lemons	0.01*
0110040	Limes	0.01*
0110050	Mandarins	**0.01***
0110990	Others (2)	0.01*
0130000	Pome fruits	**0.001***
0130010	Apples	**0.001***
0130020	Pears	**0.001***
0130030	Quinces	**0.001***
0130040	Medlars	**0.001***
0130050	Loquats/Japanese medlars	**0.001***
0130990	Others (2)	**0.001***
0401000	Oilseeds	
0401090	Cotton seeds	0.1
0401100	Pumpkin seeds	0.02*

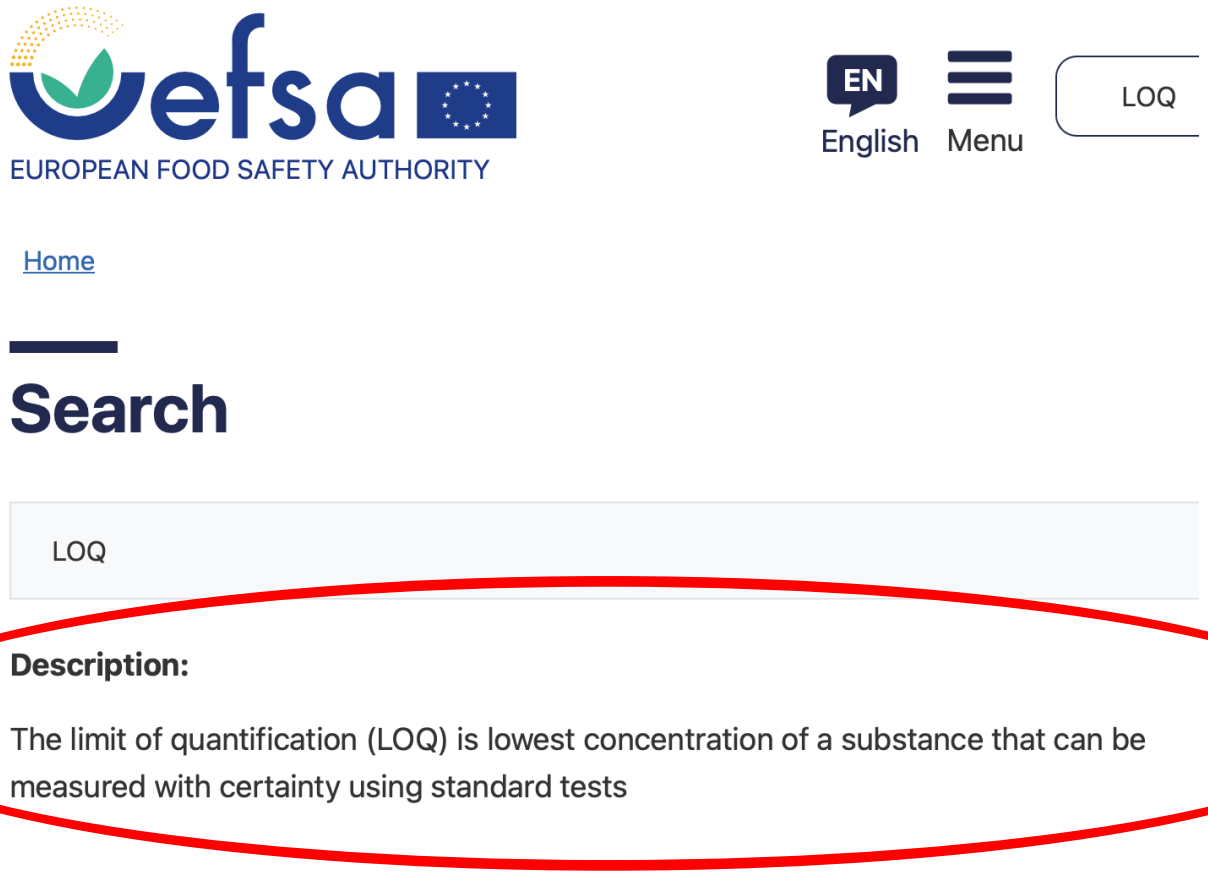
Non-authorised substances

- There is also a **signidicant number** of **unauthorised substances** for **conventional products**.

- ***MRL = LOQ (Limit of Quantification)**

  Non-authorised active substances may not be present in principle, but in concentrations **below the *MRL** they **cannot be validly quantified** and therefore should **not be taken into account.**

Definitions



Definitions

▼B

REGULATION (EC) No 396/2005 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL

of 23 February 2005

on maximum residue levels of pesticides in or on food and feed of plant and animal origin and amending Council Directive 91/414/EEC

(Text with EEA relevance)

Reg. (EC) No. 396/2005. Article 3

Definitions

(f) *'**limit of determination**' (**LOD**) means the validated lowest residue concentration which can be quantified and reported by routine monitoring with validated control methods.*

→ **LOD thus corresponds with LOQ!**

asterix-MRLs (*MRL)

Reg. (EC) No. 396/2005

EU Pesticides Database (v3.2)

This site is managed by: Directorate-General for Health and Food Safety

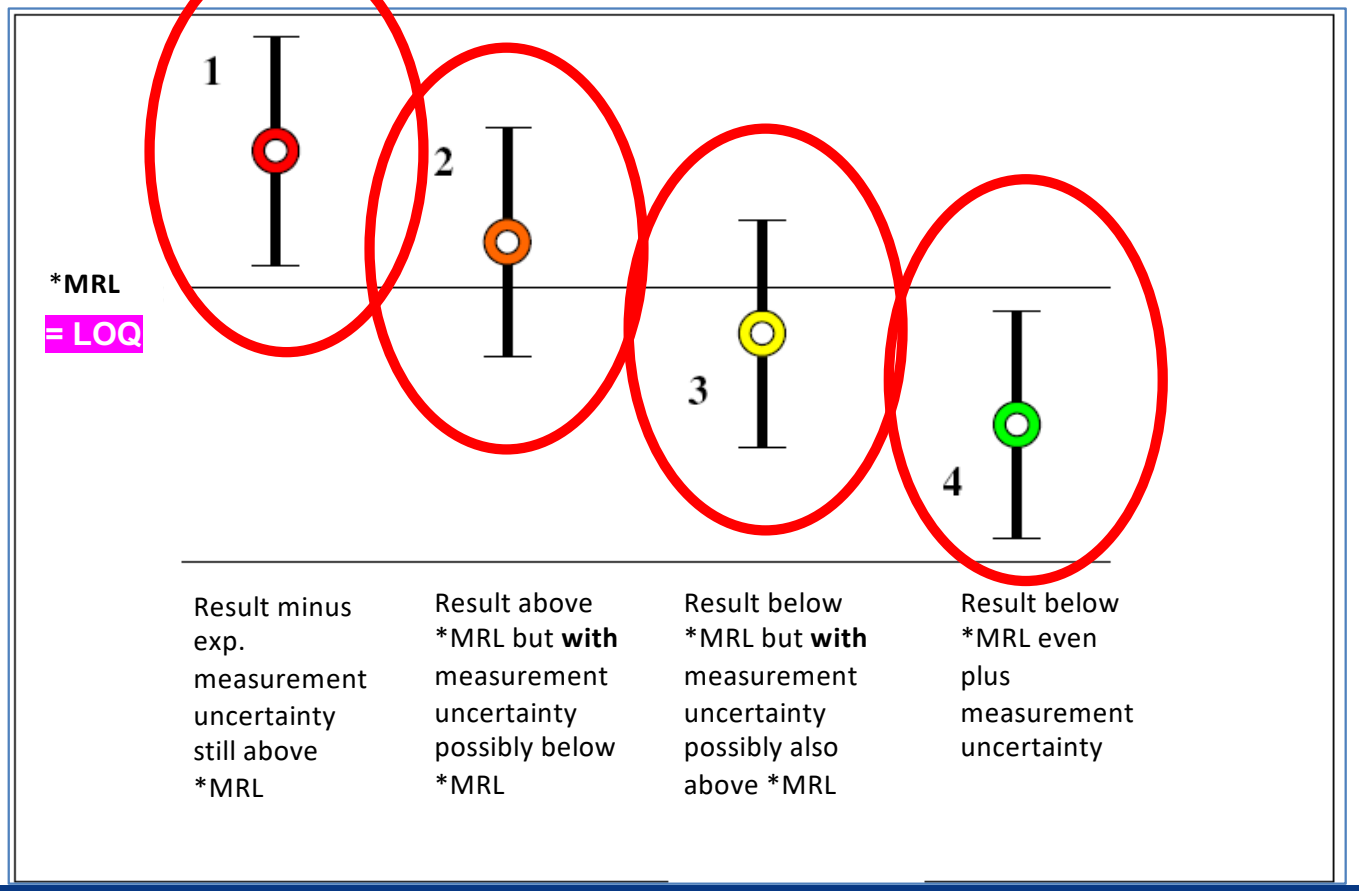
Code	Products to which MRLs apply	Anthraquinone (F) Reg. (EU) No 1146/2014 applicable
0254000	(d) watercresses	0.01*
0631030	Rose	0.02*
0632010	Strawberry	0.02*
0850010	Cloves	0.02*

0.02*

0,02* mg/kg is both the maximum content **AND** the analytical limit of quantification

(*) **Limit of analytical determination**

Application of the expanded measurement uncertainty of +/- 50 %



DG AGRI „non-compliance check if > LOQ“

Ref. Ares(2024)665554 - 29/01/2024

EUROPEAN COMMISSION
DIRECTORATE-GENERAL FOR AGRICULTURE AND RURAL DEVELOPMENT

Directorate B – Sustainability
B.4 – Organics

Brussels
AGRI.B.4/HD/MS/AGRI.B.4(2023)12415446

Dear Sir, / Dear Madam,

If for food/feed products in such combinations, a contamination is found at a level above the LOQ (taking account of measurement uncertainty), the competent authority is legally required to follow up on the finding. If a contamination is found at a level below the LOQ (taking account of measurement uncertainty), it is the responsibility of the competent authority to assess and to decide whether to follow up on the finding.

All one has to do is enter into the search field the name of the active substance, select the correct entry from amongst the entries then appearing, and click on “Display selected items”. MRLs then appear for different food/feed products. For some of those products, the MRL is marked with an asterisk. For these products, the MRL constitutes the EU harmonised LOQ.

If for food/feed products in such combinations, a contamination is found at a level above the LOQ (taking account of measurement uncertainty), the competent authority is legally required to follow up on the finding. If a contamination is found at a level below the LOQ (taking account of measurement uncertainty), it is the responsibility of the competent authority to assess and to decide whether to follow up on the finding.

Members of working group on non-compliances

Commission européenne/Europese Commissie, 1049 Bruxelles/Brussel, BELGIQUE/BELGIË – Tel. +32 22991111
Office: L130 09/073 – Tel. direct line +32 229-86052

Henri.DELANGHE@ec.europa.eu

DG AGRI „non-compliance check if > LOQ“

Products to which MRLs apply	Glyphosate Reg. (EU) No 293/2013 applicable
FRUITS, FRESH or FROZEN; TREE NUTS	
Citrus fruits	
Grapefruits	0.1*
Oranges	0.5
Lemons	0.1*
Limes	0.1*
Mandarins	0.5
Others (2)	0.1*

Products to which MRLs apply	Glyphosate Reg. (EU) No 293/2013 applicable
Seed spices	0.1*
Anise/aniseed	0.1*
Black caraway/black cumin	0.1*
Celery	0.1*
Coriander	0.1*
Cumin	0.1*
Dill	0.1*
Fennel	0.1*
Fenugreek	0.1*
Nutmeg	0.1*

DG AGRI „non-compliance check if > LOQ“

Code	Products to which MRLs apply	Boscalid (R),(F) Reg. (EU) 2022/1324 applicable
0100000	FRUITS, FRESH or FROZEN; TREE NUTS	
0110000	Citrus fruits	2
0110010	Grapefruits	2
0110020	Oranges	2
0110030	Lemons	2
0110040	Limes	2
0110050	Mandarins	2
0110990	Others (2)	2

Code	Products to which MRLs apply	Boscalid (R),(F) Reg. (EU) 2022/1324 applicable
0130000	Pome fruits	
0130010	Apples	2
0130020	Pears	1.5
0130030	Quinces	1.5
0130040	Medlars	0.01*
0130050	Loquats/Japanese medlars	0.01*
0130990	Others (2)	0.01*

Contamination below LOQ

If for food/feed products in such combinations, a contamination is found at a level above the LOQ (taking account of measurement uncertainty), the competent authority is legally required to follow up on the finding. If a contamination is found at a level below the LOQ (taking account of measurement uncertainty), it is the responsibility of the competent authority to assess and to decide whether to follow up on the finding.

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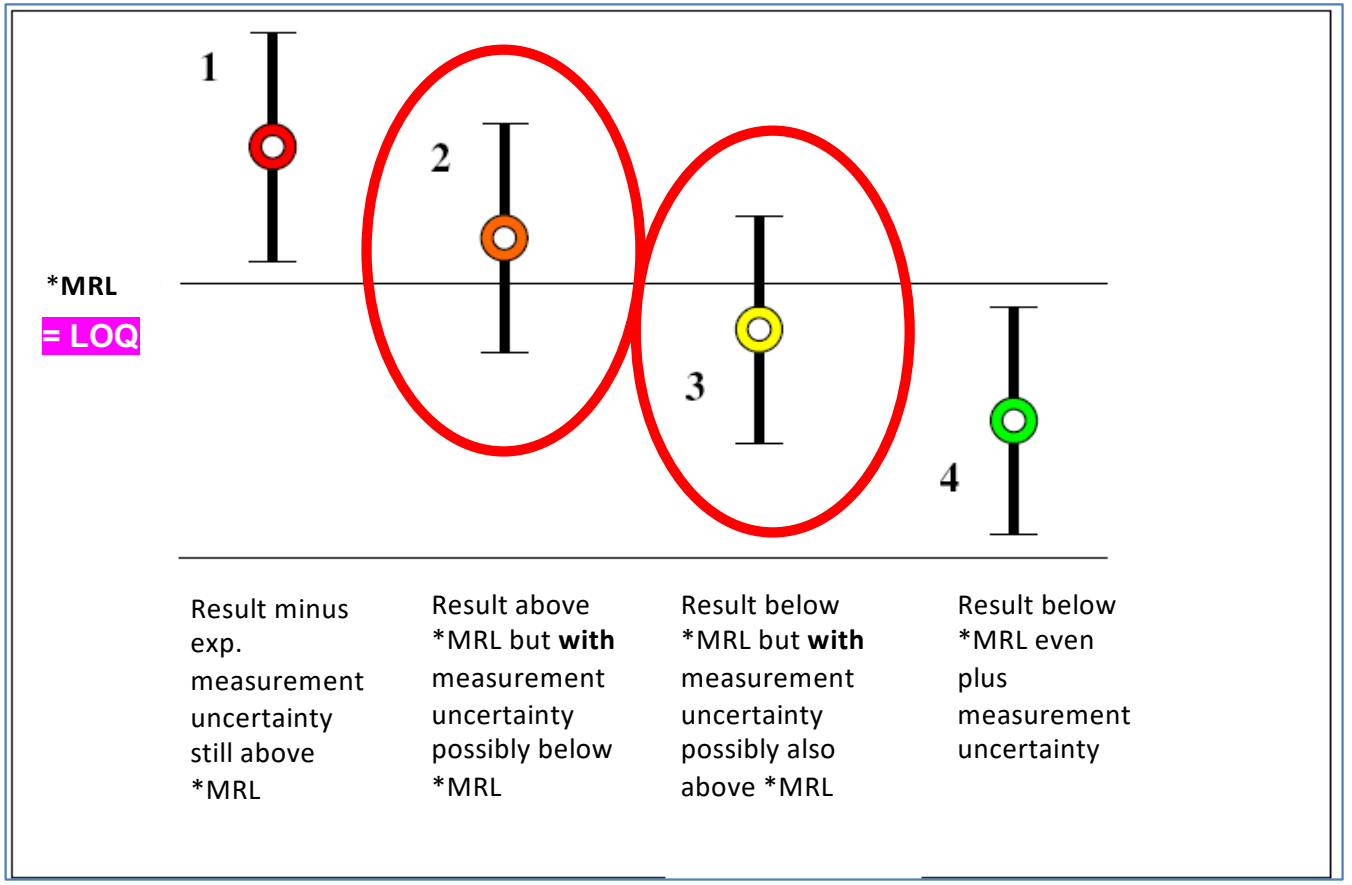
Application of the expanded measurement uncertainty of +/- 50 %



Contamination below LOQ

Request of Georg Eckert to Henri Delange (analogous):

Is it correct, that in the event of a contamination at a level below LOQ, there is no need for an investigation?

Henri Delange to Georg Eckert:

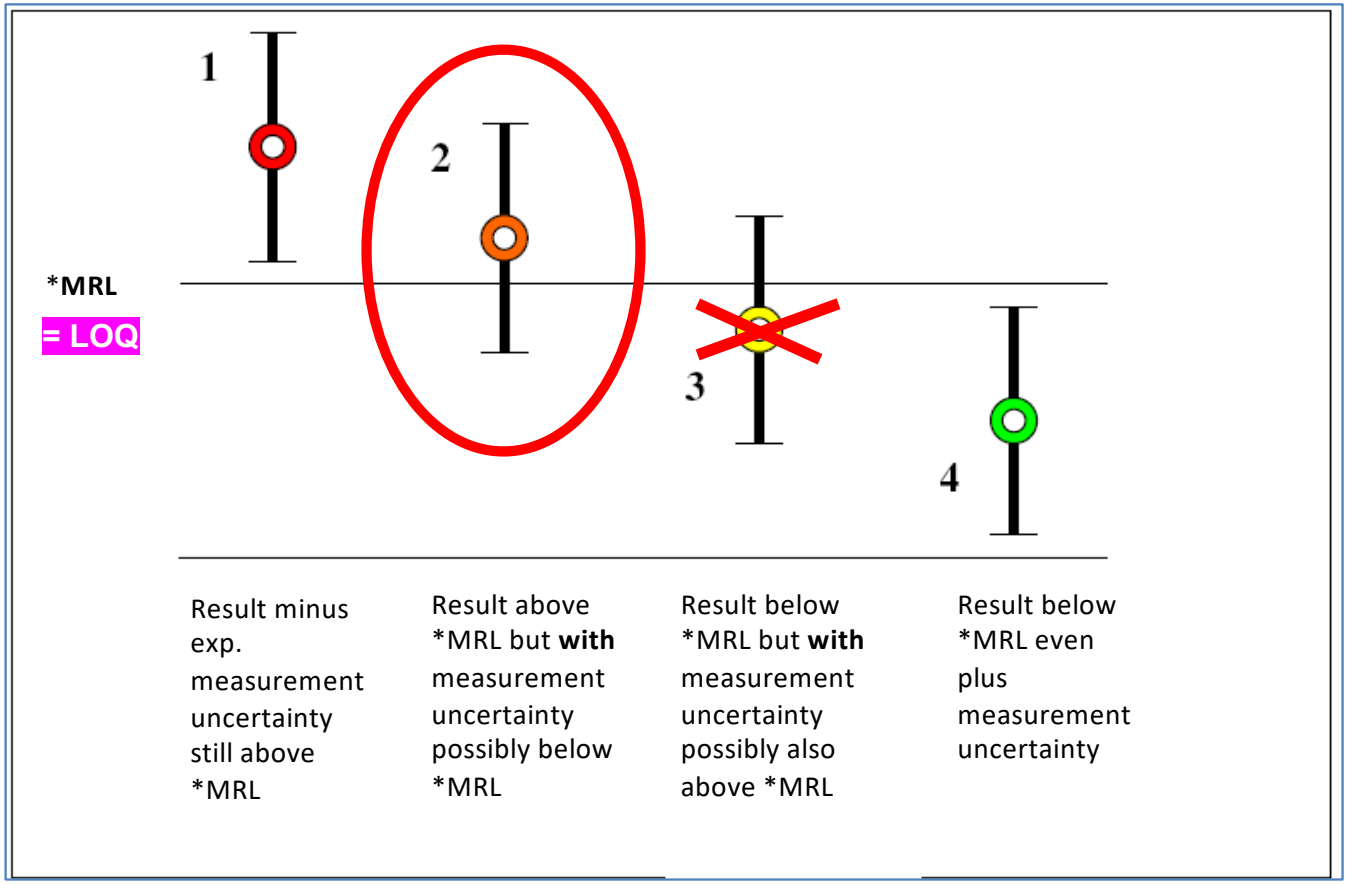
"In the event of a contamination at a level below LOQ, there is no need for an investigation is not what the letter says.

The letter says:

*If a contamination is found **at a level below the LOQ** (taking account of measurement uncertainty), it is the responsibility of the **competent authority** to assess and **to decide whether to follow up** on the finding."*

*… at a level below the LOQ **(taking account of measurement uncertainty)**, …*

… *at a level below the LOQ* (***taking account of measurement uncertainty***), …



Contamination below LOQ

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Example (by Georg Eckert to Henri Delange):

Cloves** – **Anthraquinone**: **MRL 0,02 mg/kg = LOQ (*MRL)*

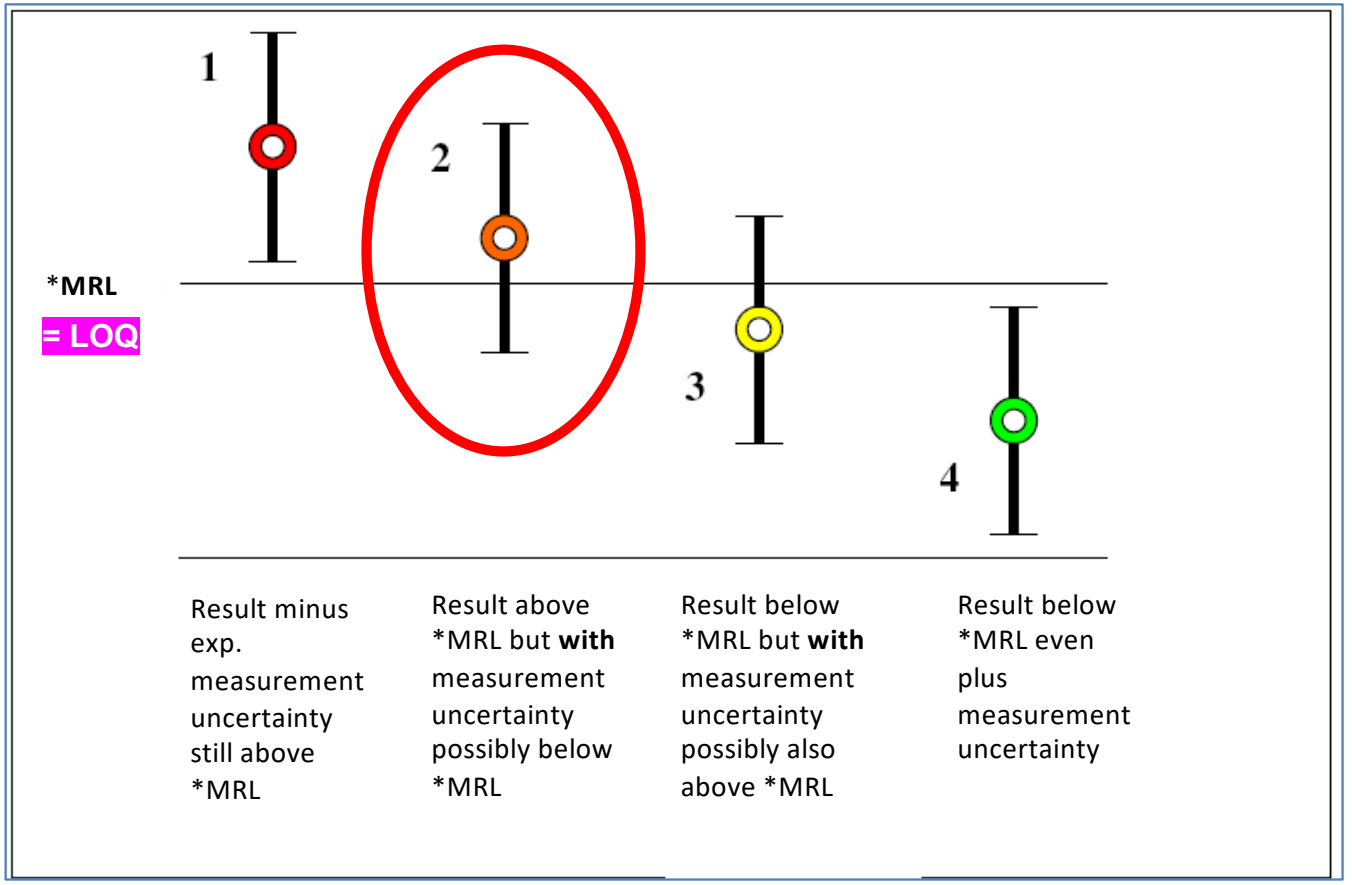
Case A) ***Result** pesticide analysis:*

***0,03 mg/kg** minus. 0,015 mg/kg (expand. measurement uncertainty of 50%):*
0,015 mg/kg

*==> Result is below the *MRL and at the same time the LOQ, but only taking into account the expanded measurement uncertainty*

*The **result of 0.03 mg/kg** stated in the test report is **validated** and therefore also **legally 'safe'**, as it is above the 'official' limit of quantification (LOQ)!*

Application of the expanded measurement uncertainty of +/- 50 %



Contamination below LOQ

lach bruns

Example (by Georg Eckert to Henri Delange):

Cloves** – **Anthraquinone**: **MRL 0,02 mg/kg = LOQ (*MRL)*

Case A) ***Result** pesticide analysis:*

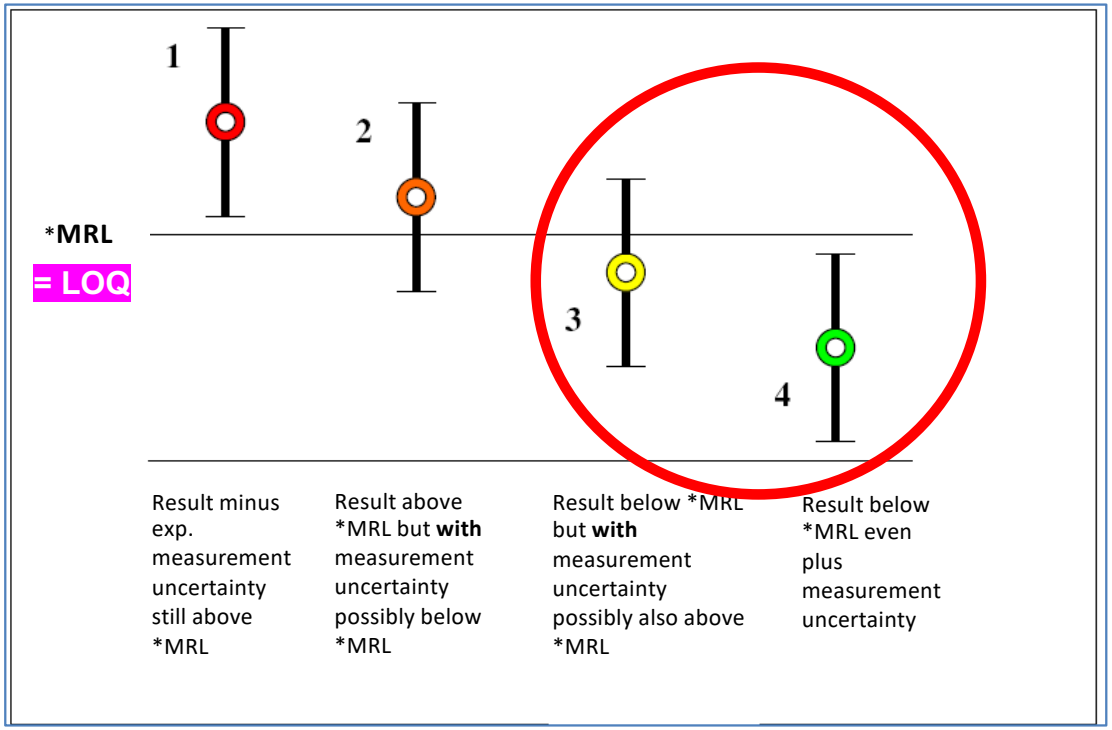
***0,015** mg/kg*

*The **result itself** is already **below the *MRL** and at the same time **the LOQ** and therefore **NOT valid** and **NOT 'safe',** as the value is **below** the '**official**' limit of quantification (**LOQ**)!*

~~*If a contamination is found at a level below the LOQ **(taking account of measurement uncertainty)**, it is the responsibility of the competent authority to assess and to decide whether to follow up on the finding.*~~

Contamination below LOQ

*The result itself is already **below the *MRL** and at the same time **below the LOQ** and therefore **NOT valid** and **NOT 'safe',** as the value is **below** the '**official**' limit of quantification (**LOQ**)!*



Product groups acc. reg. (EC) 396/2005

- **Product groups are listed in the Pesticide MRL Regulation 396/2005 according to their usual supply form with the corresponding MRLs!**
- **This means that the *MRLs (= LOQ) can often be applied directly!**
- **If not: processing factors!**

Product groups acc. reg. (EC) 396/2005

Code	Products to which MRLs apply	Glyphosate Reg. (EU) No 293/2013 Applicable Annex	Boscalid (R),(F) Reg. (EU) 2022/1324 Applicable Annex	Fenoxycarb Reg. (EU) 2024/341 Applicable Annex
600000	TEAS, COFFEE, HERBAL INFUSIONS, COCOA AND CAROBS			0.05*
610000	Teas	2	40	0.05*
640000	Cocoa beans	0.1*	0.01*	0.05*

Processing factors

grapes - raisins

VF **> 1** = **Enrichment**
VF **< 1** = **Depletion**

within the processed product

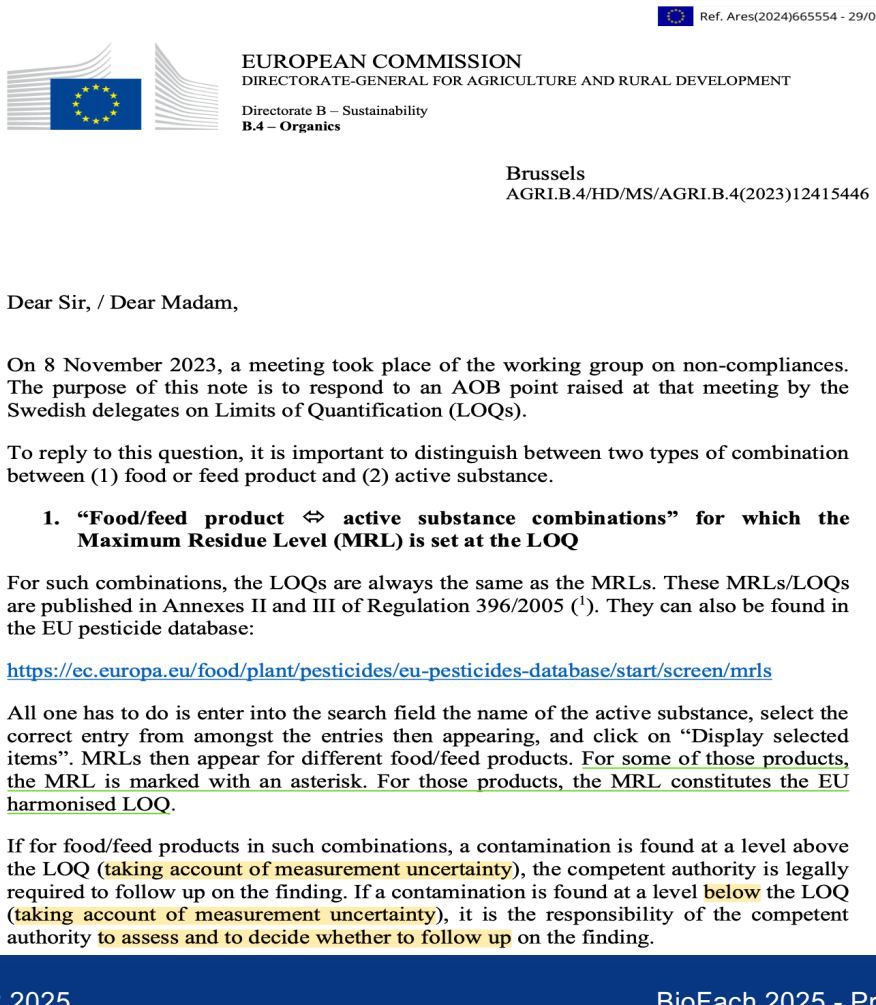
residue definition (for monitoring)	commodity	processed matrix	median *Pf*	number of trials	acceptability of study
carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	grapes, red	raisin	3.07	2	indicative
carbendazim and benomyl (sum of benomyl and carbendazim expressed as carbendazim)	grapes, white	raisin	2.53	1	indicative
chlorantraniliprole	grapes, white	raisin	4.92	2	yes
chlorantraniliprole	grapes, red	raisin	3.77	2	yes
chlorothalonil	grapes	raisin	0.47	2	yes
chlorpyrifos	grapes	raisin	0.17	1	indicative
chlorpyrifos	grapes, white	raisin	0.95	1	indicative
chlorpyrifos-methyl	grapes, white	raisin	not applicable	1	no
chlorpyrifos-methyl	grapes, red	raisin	< 0.09	1	indicative
clofentezine	grapes	raisin	0.29	6	yes
clofentezine	grapes	raisin	< 0.67	1	indicative
clofentezine	grapes	raisin	1.00	1	no

Processing factors

- If possible, use analytical data from the raw products themselves for an assessment.
- Pesticide levels in processed foods can only serve as an indication for further investigations.
- In any case, it is advisable to refer to the analysis of the raw material, as the influence of processing on the respective pesticides cannot be assessed (in a legally binding manner).

European database of processing factors for pesticides in food:
https://zenodo.org/record/1488653#.XjrjSy2bpR6

Procedure for pesticide findings



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EUROPEAN COMMISSION
DIRECTORATE-GENERAL FOR AGRICULTURE AND RURAL DEVELOPMENT

Directorate B – Sustainability
B.4 – Organics

Brussels
AGRI.B.4/HD/MS/AGRI.B.4(2023)12415446

Dear Sir, / Dear Madam,

On 8 November 2023, a meeting took place of the working group on non-compliances. The purpose of this note is to respond to an AOB point raised at that meeting by the Swedish delegates on Limits of Quantification (LOQs).

To reply to this question, it is important to distinguish between two types of combination between (1) food or feed product and (2) active substance.

1. **"Food/feed product ⇔ active substance combinations" for which the Maximum Residue Level (MRL) is set at the LOQ**

For such combinations, the LOQs are always the same as the MRLs. These MRLs/LOQs are published in Annexes II and III of Regulation 396/2005 ([1]). They can also be found in the EU pesticide database:

https://ec.europa.eu/food/plant/pesticides/eu-pesticides-database/start/screen/mrls

All one has to do is enter into the search field the name of the active substance, select the correct entry from amongst the entries then appearing, and click on "Display selected items". MRLs then appear for different food/feed products. For some of those products, the MRL is marked with an asterisk. For those products, the MRL constitutes the EU harmonised LOQ.

If for food/feed products in such combinations, a contamination is found at a level above the LOQ (taking account of measurement uncertainty), the competent authority is legally required to follow up on the finding. If a contamination is found at a level below the LOQ (taking account of measurement uncertainty), it is the responsibility of the competent authority to assess and to decide whether to follow up on the finding.

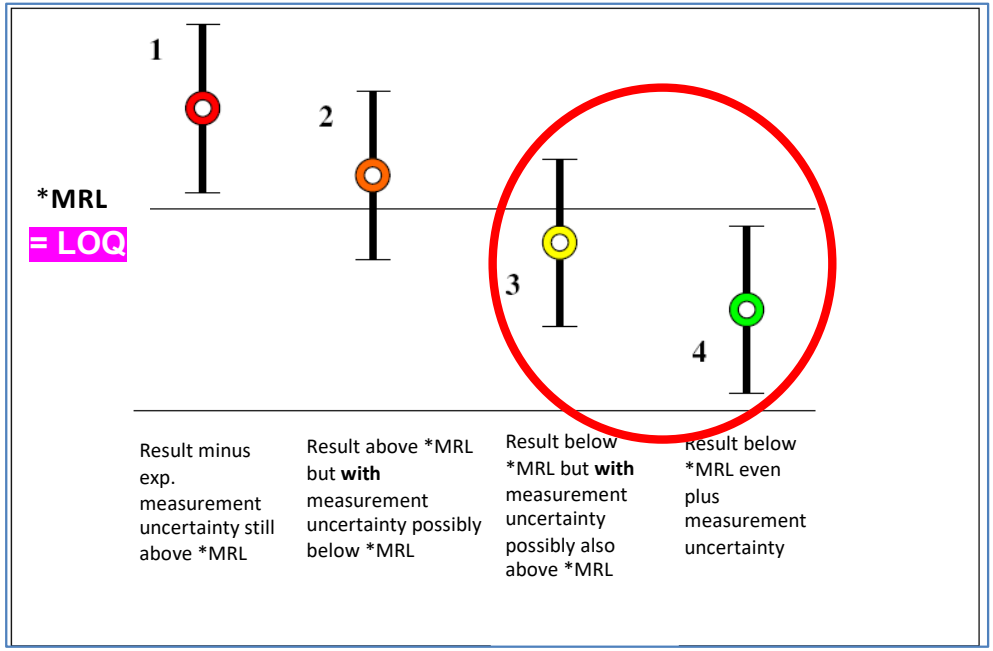
In the case of **pesticide** findings **below** the (asterisk) ***MRL**, it must be assumed that these **do not give rise to any suspicion** with regard to the presence of unauthorised substances and products.

Results **below** the ***MRL = LOQ** are **not validated**!

Procedure for pesticide findings

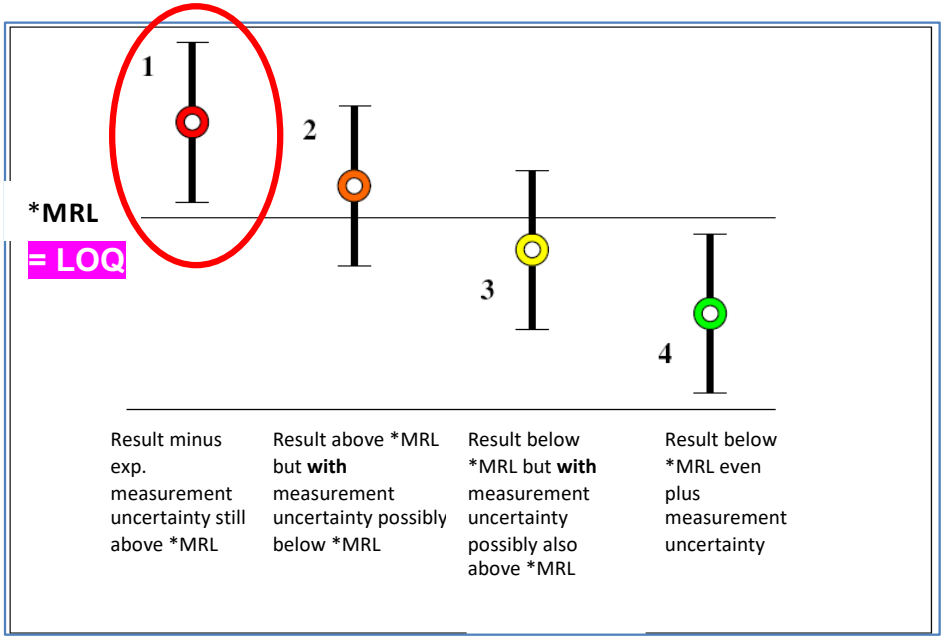
For pesticide results **below** the (asterisk) ***MRL**, it can be assumed that this **does not give rise to any suspicion** regarding the presence of unauthorised substances and products.

Results **below** the ***MRL = LOQ** are **not validated**!



Procedure for pesticide findings

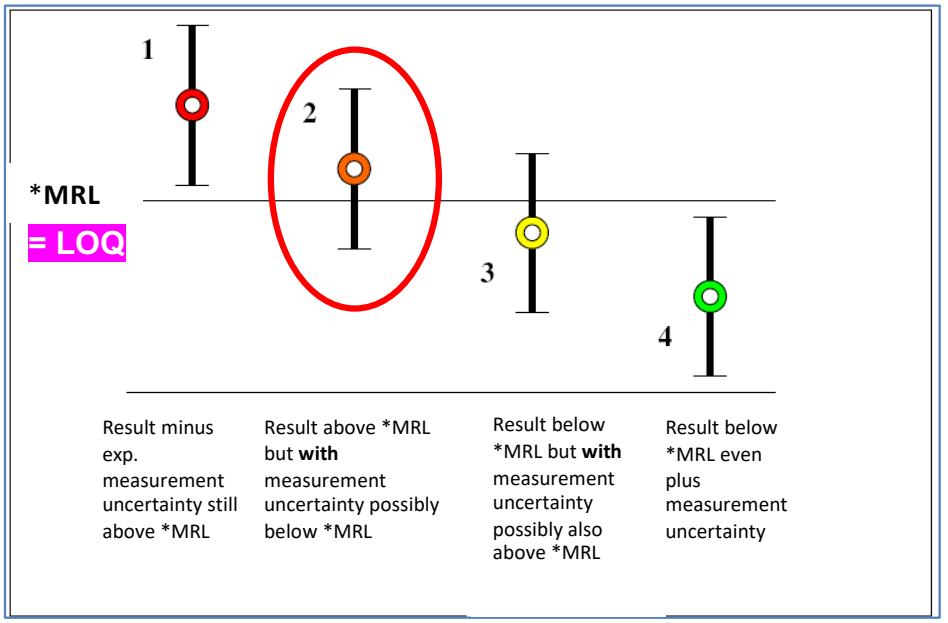
Pesticide results that are **above** the (asterisk) *RHG, **taking into account** the **measurement uncertainty** (50%), give **rise to suspicion** regarding the presence of **unauthorised substances** and products.



Procedure for pesticide findings

lach : bruns

For pesticide results that are **below** the (asterisk) *MRL **including** the **measurement uncertainty** (50%), it is the **responsibility** of the **control authorities** or **control bodies** to assess and decide whether follow-up measures are necessary.



Thank You!