

## Pesticide residues in (organic) honey and their judgement

We currently have many customer requests on the subject of pesticide residues in organic honey. The currently observed relevant residues are mostly glyphosate and thiacloprid (neonicotinoid) as well as the various bee medicines / varroacides such as in particular amitraz, coumaphos and tau-fluvalinate. We would like to explain the topic to our customers in more detail in this newsletter.

### Absence of residues = Organic?

As part of the food business operator's quality control, a risk-based check should be carried out, in particular for organic goods, to ensure that the legal provisions of the EU with regard to pesticide residues have been complied with. However, the increasingly sensitive analytical methods raise the question under which conditions a sample can still be safely traded as "organic" and how possible positive residue results might be judged from the point of view of EU food surveillance authorities. In general, it must be emphasized that the declared name of the food (label) "organic", "ecological", "eco" or "bio" is not defined by the "absence of residues", which is often misunderstood.

An "absence of residues" is also a question of definition. It depends on the analytical sensitivity (limit of quantification = LOQ) with which "no residue" was found.

### EU Organic Control Bodies - Are there actually limits for (organic) honey?

The EU Organic Control Bodies are responsible for monitoring compliance with the provisions of the EU regulation on organic farming (EC) No. 834/2007 and the Implementing Regulation (EC) No. 889/2008. In the case of residues found in organic products, the question arises as to whether there have actually been violations of the legal provisions or whether the principles of organic production have been followed and it may be an unavoidable contamination from the environment.

The determination of the root cause and possible measures are also the responsibility of the inspection bodies/authorities and not of the food business operator or laboratory, which carries out the quality control analytically and usually also interprets the results for its customers.

Problematic in the evaluation by the inspection bodies is the level and number of proven positive residue results in organic goods, as there are no legally binding reference or orientation values for measures in organic goods in the various EU member states to date.

So far, for honey only maximum residue limits (MRL) for the two bee medicines / varroacides amitraz and coumaphos used in conventional beekeeping have been set with 0.2 mg/kg and 0.1 mg/kg (37/2010/EU and 396/2005/EC) - in addition to the pesticides generally regulated for most foods. However, as these varroacides are generally not allowed to be used in organic beekeeping, "no" residues of these synthetic pesticides should actually be detectable in organic goods. You can find more about this topic later in the section "Special case bee medicines / varroacides in organic honey".

The question therefore arises as to what, in addition to the MRLs (if available/set), can be used as a basis for assessing pesticide residues in organic goods, both from the point of view of the competent EU authorities and from the point of view of the trade.

# Orientation value for organic goods 0.010 mg/kg (BNN) - measurement uncertainty to be taken into account 50%

The approach promoted by the Association of Organic Processors, Wholesalers and Retailers (BNN) in Germany, for example, uses an orientation value of 0.010 mg/kg. <sup>1</sup> The BNN argues that even in compliance with all legal regulations for organic farming, inadmissible substances can be detected in organic products. After all, organic farming does not take place under a glass bell. However, according to BNN, this orientation value is not a legal limit value and should therefore not be interpreted as a "Bio-MRL" or a "critical limit". <sup>2</sup>

In the legal assessment of pesticide residues, an expanded measurement uncertainty or analytical variance of 50% (DG SANTE) should generally be taken into account, "in favour of the accused". Thus, for the BNN, a product containing up to 0.020 mg/kg of a pesticide would still be marketable as "bio" by the members without restriction, taking into account the measurement uncertainty (if, in addition, a maximum of 2 pesticides were contained in this amount and there were no other indications that legal provisions had been violated). Only starting from 0.021 mg/kg would the BNN orientation value be exceeded, taking into account the measurement uncertainty of 50%. <sup>3</sup> However, the scope of BNN does not include animal products like honey. It is therefore at the discretion of the food business operator or the control body whether this orientation value is also used for honey if there are no other guideline values at present.

#### Current judgement of low positive findings in organic products by EU organic control bodies

According to the latest information from the German Eco-Control Authorities, minor findings in the range of the common LOQ of 0.01 mg/kg in organic goods are currently not being followed up, as these are reported in very large quantities and can be detected in almost every organic product. If measures were taken in all these cases, all inspection bodies and thus the entire German organic market would be virtually paralyzed. In our experience, inspection bodies from other EU countries (e.g. Spain, Bulgaria) proceed in a similar way, so that in the case of low findings there is currently no need for action from the point of view of the control bodies.

Last week, QSI also issued a statement to the control bodies that, in particular, findings of pesticides which are not plausibly used in beekeeping could potentially lead to unavoidable contamination, especially with low findings of the substance glyphosate. Glyphosate is used worldwide and can be introduced into the honey via the environment (soil, water, wind, downforce, dust) without the organic beekeeper having any special influence on it (only the location of the hives can be influenced, but even a flight radius of the bees of approx. 3 km can hardly be controlled).

Nevertheless, in the organic products tested for glyphosate at QSI in the years 2017-2018, only 8 % of the cases showed residues above the LOQ of 0.01 mg/kg (82 out of 1129 samples). In 27 % of the investigated organic honeys traces below the LOQ were detectable, in 67 % of the organic samples no measurable residues of glyphosate were found at all, even significantly below the LOQ.

In comparison to all investigated honeys (n=16870) during the same period, 43 % of the samples were positive above the LOQ of 0.01 mg/kg and 11 % of the samples showed glyphosate contents above the MRL of 0.05 mg/kg. The data show that it is certainly possible to produce organic honey without measurable glyphosate residues (< LOQ).

<sup>&</sup>lt;sup>1</sup> https://n-bnn.de/sites/default/dateien/pdfs/BNN-Orientierungswert EN 04022014.pdf

<sup>&</sup>lt;sup>2</sup> https://www.ifoam-eu.org/sites/defaul<u>t/files/event/files/ifoameu\_event\_biofach\_kirsten\_arp\_201302.pdf</u>

https://n-bnn.de/sites/default/dateien/bilder/Downloads/BeispieleOrientierungswert\_EN\_04022014.pdf

Pesticides in conventional goods (MRL) from an official point of view - Recommendation for trade Similarly, EU authorities must take into account the 50% expanded measurement uncertainty (SANTE/11813/2017 <sup>4</sup>) when assessing MRL exceedances. With a fixed maximum residue limit (MRL) of 0.01 mg/kg, for example, and a determined residue content below 0.021 mg/kg, an exceeding of the MRL cannot be assumed with a probability bordering on certainty (95 % confidence interval). On the other hand, for importers, producers and suppliers, this means that the MRL should not be exhausted in the context of self-monitoring. To ensure that the goods comply with the legal food requirements with a probability bordering on certainty (95 % confidence interval), the residue content in the sample should in the worst case (measured value plus 50 %) still be below the MRL, taking into account the measurement uncertainty. In the above-mentioned (worst) case, this would be a maximum residue value of 0.013 mg/kg for a substance with an MRL of 0.01 mg/kg, because 0.013 mg/kg plus 50% (=0.0065 mg/kg) would be rounded accordingly to 0.020 mg/kg, which would mean that the MRL would still not be exhausted even taking into account the measurement uncertainty. <sup>5</sup>

### Special case bee medicines / varroacides in organic honey

Residues of varroacides (amitraz, coumaphos etc.) are particularly problematic for organic honey, since only the substances formic acid, lactic acid, acetic acid and oxalic acid as well as menthol, thymol, eucalyptol or camphor mentioned in Regulation (EC) No. 889/2008, Article 25 (6) may be used for a varroa treatment. A positive result of a synthetic varroacide in organic honey could therefore plausibly result from an unauthorized use. But possibly also a contamination could be present via recycled beeswax (foundations), which should have also organic quality in organic beekeeping, if possible. However, there are exceptions if no organic wax is available on the market. Another possible entry source would be, for example, a contamination via the use of amitraz in fruit-growing (as an insecticide), which would also have to be checked by the control body.

### Update Amitraz (see also QSI-Newsletter March 2018)

Amitraz represents a special problem case, since the new analytical methods, in which the three amitraz metabolites DMPF, DMF and DMA are determined directly by LC-MS/MS (or GC-MS/MS), find about 3 times higher residue contents of the sum amitraz in honey than the meanwhile outdated hydrolysis method, which was used by QSI before 2018. The true residue content in a real sample is of course not known. This found residue ratio of about 3:1 (new: old method) has been confirmed by more than 100 comparative studies with both methods at QSI. The number of positive results in organic honey has increased significantly in 2018 due to the more sensitive, new methodology, since low amitraz residues, which were previously below the respective LOQ (0.005 mg/kg, 0.010 mg/kg, 0.020 mg/kg), are now significantly above the LOQ with the new methodology. In March 2018 we published a newsletter on the change of the amitraz method. In April, May and November 2018 BIPEA offered three proficiency tests on amitraz in honey, which according to our findings were all only passed using the new method and the applied formula (stoichiometry) QSI uses for the calculation of the sum amitraz from the determined metabolites. With the old hydrolysis method one would have been outside the acceptable result range, i.e. one would have found too little amitraz and failed the PT's. QSI has successfully passed all 3 ring trials with the new methodology. A successful participation is beside the validation a requirement from the ISO 17025 for the accreditation of the method.

<sup>&</sup>lt;sup>4</sup> https://ec.europa.eu/food/sites/food/files/plant/docs/pesticides mrl guidelines wrkdoc 2017-11813.pdf

<sup>&</sup>lt;sup>5</sup> http://www.cvuas.de/pub/beitrag.asp?subid=1&Thema\_ID=5&ID=2204&Pdf=No&lang=DE

The EU Reference Laboratory for pesticides (CVUA Freiburg) and also the highest responsible German authority, the Federal Office of Consumer Protection and Food Safety (BVL Berlin) have confirmed that the monitoring laboratories will probably measure amitraz mostly using the new methodology, but will still determine the sum amitraz using the old calculation formula. With the old calculation (stoichiometry of 1:1), the authorities probably overestimate the "real" amitraz residues by a factor of 2. The National Reference Laboratories (NRL) for pesticides have comparably little experience with the calculation of amitraz findings in honey, but rather with the matrix fruit (application as insecticide in fruit growing). For pears, on the other hand, the old calculation formula yields plausible results, since only one metabolite is formed from one amitraz molecule in this matrix.<sup>6</sup> However, in our experience (several thousand amitraz measurements in 2018) at least 2 metabolites are always built in honey. QSI therefore does not share the opinion of the BVL that only one amitraz metabolite is built in honey. The EU-RL in Freiburg discussed at least the new calculation proposed by QSI for the new method, but has not returned with any news since March 2018.

In the meantime it could be verified that the amitraz metabolite DMPF commonly observed in honey generally cannot be detected with the old acid hydrolysis method, which can plausibly explain the one factor 3 lower findings with the outdated hydrolysis method. The following table illustrates the problem of analysis and calculation:

Table 1: Example for amitraz residue in honey, detected content depending on method and calculation model/stoichiometry of metabolism (estimation QSI)

"True" Residue Content	Content outdated hydrolysis method (1:1 stoichiometry)	Content new direct method (e.g. QSI, new calculation, 1:2 stoichiometry)	(e.g. authority labs, EU-RL,
unknown	0,015 mg/kg	0,045 mg/kg	0,090 mg/kg
unknown	0,070 mg/kg	0,21 mg/kg	0,42 mg/kg

For reasons of better clarity, QSI now only reports the legally regulated sum amitraz in the official test report, which is calculated from the three individual amitraz metabolites.

The lowest LOQ offered by QSI for the varroacides is still 0.005 mg/kg, since October 2018 also within the new pesticide screening method for organic honey (code 88550, 600+ substances, LC-MS/MS and GC-MS/MS), which is offered beside the common pesticide combination method (code 88500, report limits=0.01 mg/kg). With the combination methods, you also receive a current pesticide list with all tested substances together with their report limit / LOQ with each test report.

All pesticide / varroacides methods and offered LOQ are accredited under the terms of ISO 17025. The pesticide combination method is carried out subcontracted by our Tentamus partner laboratory bilacon GmbH in Berlin and is accredited under the code PV-SA-085 2017-08 by the DAkkS. Findings below the LOQ are automatically communicated to the customers by QSI from ½ LOQ within an "Additional Information" (pdf-file) next to the official test report (not for code 88500, only Bio-Code 88550 and single pesticides/packages varroacides from QSI). As customer you have the possibility to cancel the "Additional Information" if you do not want to be informed about small findings below the LOQs. However, this setting also applies to all other analysis methods from our company, including antibiotics. We also offer all pesticide analyses with express service.

Please do not hesitate to contact us if you have any further questions on this subject.

<sup>&</sup>lt;sup>6</sup> https://www.ncbi.nlm.nih.gov/pubmed/25053051