BioFach 2018

lach : bruns

Taking a closer look.

Pesticides and contaminants in organic products

Dr. Silke Bruns, 15. February 2018

This presentation is about analytical performances of laboratories

AGENDA

Ring tests “basics”

Un-announced Ring tests
But: VISIBLE test character

Undercover Ring tests

Ring tests “Basics”

What is a ring test?

A ring test (also called as proficiency test) is an **inter-laboratory test** that allows to **evaluate the performance of testing laboratories**, and is based on analysis of **similar homogeneous samples**. […] to enable laboratories to assess and improve their […] analysis performance.

Why should labs participate in ring tests?

A ring test offers to a laboratory a **possibility of external quality assessment** of the analytical results it produces.

Source: Food and Agricultural Organisation of the United Nations

Ring tests “Basics”

How ring tests are usually designed

Normal Ringtest Design

- the laboratory is informed about the sample arrival in advance, as the lab applied for taking part in the test in advance.

- the samples are usually analysed with special care (≠ routine)

- the scope of the spiked pesticides is limited and a list of possibly spiked pesticides is known to the lab

- turn-around-time varies between several weeks

Ring tests “Basics”

Ring test samples versus Routine Samples

- unannounced arrival

- short turn-around times (48 hours or even less)

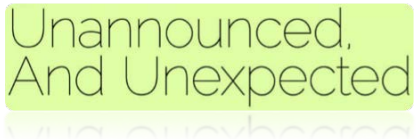
- the kind and level of pesticide residues are not known

- Ring test samples are **always homogenates**.
  Thus the first and crucial steps (sample preparation and homogenisation) are not checked in ring tests.

Puree – instead of non-chopped samples

Un-announced Ring tests
But: VISIBLE test character

Closing the gap between Ring test samples and Routine Samples



- Un-announced arrival: The entire ring test is not known at all to the laboratories. It does not know if, when and what kind of test will take place / or not.

- VISIBLE test character: The test sample is sent together with an instruction letter to the laboratory → The lab then is informed: "*This is a test sample.*"

- Short turn-around times: The laboratory is instructed to deliver results within a few days (< 1 week).

- The kind and level of pesticide residues are not known.

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BNN Un-announced Ring tests (visible test character):

Apple matrix

= some kind of “all around matrix”, common product for pesticide laboratories

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BNN Un-announce ring test: Apple Chips (Sept. 2016)

26 labs participated

10 pesticides spiked in total

Pesticide	spiked level (mg/kg)
Carbendazim	0,023
Carbofuran	0,015
Cyflufenamid	0,055
Folpet	0,045
Phthalimid	0,030
Spirodiclofen	0,048
Tebuconazol	0,015
THPI	0,052
Ethephon	0,078
Phosphonic	0,16

At conc. levels between 0,015 mg/kg and 0,16 mg/kg

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BNN Un-announced ring test: Apple Chips (Sept. 2016)

7 out of 26 participating laboratories identified and quantified all analytes correctly (27%)

Hmmm ...

Brilliant None of the laboratories reported false positive results.

False negative results were reported for Folpet (one lab), THPI (one lab) and Phosphonic acid (one lab).

Could be worse

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BNN Un-announced Ring tests (visible test character):

Tea matrix

= challenging matrix, only established in specialised

laboratories for tea analyses

BNN Un-announcded Ring test: Tea (May 2013)

16 labs participated

8 pesticides spiked in total

Pesticides	Spiking level mg/kg
Acetamiprid	**0,042**
Bifenthrin	**0,058**
Carbendazim	**0,033**
Chlorfenapyr	**0,145**
Clothianidin	**0,145**
Endosulfan-Sulfat	**0,027**
pp-DDE	**0,025**
pp-DDT	**0,095**

At conc. levels between 0,025 mg/kg and 0,145 mg/kg

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BNN Un-announced ring test: Tea (May 2013)

2 out of 16 participating laboratories identified and quantified all analytes correctly (13%)

False positive results: p,p'-DDD (9 times) and o,p'-DDT (3 times)

False negative results were reported for Chlorfenapyr (two labs), Endosulfan-sulfate (one lab) and Anthraquinone (eight labs).

Undercover Ring tests
Test character NOT known

Closing the gap between Ring test samples and Routine Samples

- Unannounced arrival: The date for sending the test sample to the laboratory is NOT known in advance.
- Test character NOT visible: The test sample is sent by official clients to the laboratory (thus NOT by BNN)→ The lab is NOT informed about the test character.
- routine turn-around times: The laboratory is instructed to deliver results as agreed with the client (usually < 1 week).
- the kind and level of pesticide residues are not known to the laboratory.

UNDERCOVER

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BNN UNDERCOVER ring test: Matcha tea (March 2017)

7 labs participated unknowingly

7 pesti-cides in total

Pesticide	spiking level (mg/kg)
Anthraquinone	0.016
Bifenthrin	0.095
Lindane	0.023
Phthalimide	0.071
Trifluralin	0.019
Nicotine	incurred (AV: 0,073)
Perchlorate	incurred (AV: 0,270)

At conc. levels between 0,016 and 0,27 mg/kg

Undercover Ring tests

BNN UNDERCOVER ring test: Matcha tea (March 2017)

2 out of 7 participating laboratories identified and quantified all parameters correctly (29 %).

Hmmm ....

Damn ....

One laboratories reported a result, which was considered false positive (Chlorate instead of Perchlorate).

False negative results were reported: Anthraquinone (3 labs), Lindane (2 labs), Trifluralin (2 labs), Bifenthrin (1 lab)

Could be worse ....

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SYNABIO

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BNN / Synabio Un-announced Ring tests (visible test character):

Flour (cereal) matrix

= challenging matrix, only established in specialised

laboratories for cereal analyses

BNN / Synabio Un-announced ring test: Cereal flour (Dec. 2017)

20 labs participated

8 pesticides spiked in total

Pesticide	Spiked level (mg/kg)
AMPA	*0,048*
Glyphosat	*0,037*
Trimethylsulfonium iodide	*0,063*
beta-Cyfluthrin	*0,037*
Diflufenican	*0,033*
Piperonylbutoxid	*0,1*
Pirimiphos-methyl	*0,023*
Sulfoxaflor	*0,067*

At conc. levels between 0,023 mg/kg and 0,1 mg/kg

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SYNABIO

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BNN / Synabio Un-announced ring test: Cereal flour (Dec. 2017)

7 out of 20 participating laboratories quantified all eight parameters correctly (35%)

Brilliant None of the laboratories reported false positive results.

Only one false negative result was reported for Glyphosate. Looks Good!

Conclusions

- BNN **un-announced** ring tests: Unannounced, And Unexpected
  Closing the gap between the analytical challenge of (announced) Ring test samples versus Routine Samples
- BNN **undercover** ring tests: UNDERCOVER
  Real life challenge, as the test character is not known at all to the (unknowingly participating) laboratories
- Both approaches (un-announced / undercover) better reflect the routine analytical performances of laboratories, also related to “all around products” like apples.

- Analytical performances are also strongly related to the challenge caused by the product-matrix itself:

tea: 

flour (cereals): Better

Thank you

Merci

Grazie

Gracias

Danke

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