relana®

Minutes of the

relana® meeting 2017 - Gent, Belgium

Date:
Session 1: Tuesday 13.06.2017 “14:00 p.m to 18:00 p.m.”
Session 2: Wednesday 14.06.2017 “9:00 a.m. to 13:30 p.m.”

Place: Hotel de Flandre
Poel 1-2
B - 9000 - Ghent
BELGIUM
www.hoteldeflandre.be

Participants:

Analytica Alimentaria, Espana
Mr **Fran**cisco Ferrer

Analytica Alimentaria, Germany
Ms **Cecilia** Boned

Bilacon, Germany
Mr **Roy** Sperling

eurofins Dr. Specht, Germany
Mr **Marco** Miebach

eurofins LZV, The Netherlands
Mr **Marvin** Overbeeke
Mr **Sebastian** Hahn
Mr **Khalid** Bensbaho

Galab, Germany
Mr **Eckard** Jantzen
Mr **Frank** Schreiber

Greit, Italy
Mr **Lorenzo** Petrini

Institut Bostel, Germany
Ms **Anja** Bostel
Mr **Philipp** Silberberger

LVA, Austria
Mr **Zoltan** Balla
Mr **Andreas** Gschaider

PRIMORIS, Belgium
Mr **Hans** Braeckman
Mr **Bart** Willaert
Mr **Wim** de Meyer
Ms **An** Gea

Lach & Bruns, Germany
Ms **Silke** Bruns
Mr **Günter** Lach

Excused absence
Labor Friedle, Germany

Preparation of the minutes-protocol

Hamburg, 22nd June 2017

Dr. Silke Bruns

Dr. Günter Lach

Contents

Page

1. General

Welcome / Opening words / Agenda

2. Stock solutions project

Internal relana®-project: Remarks of the participants; discussions about the observations related to the particular pesticides; discussion about next steps.

3. relana® method ring tests

Pyrethroids, Metabolites, Leaves: discussion and remarks (selection).

In general very good results were achieved. Several aspects have been highlighted and discussed in order to achieve good recoveries and at least good results in routine analyses: Decreasing of sample weight, reducing injection volume, dilution of the final extract, quantification using standard addition or matrix-matched calibration.

4. Sample pre-preparation project

Discussion and remarks of the participants (selection):

- This project gives a realistic picture of where we are in the routine.
- Onions: When do the roots have to be cut off - or not? Is this depending on the variety (bulb onions, spring onions, etc.)? Depending on the kind of applications (of pesticides), the pesticides might be not equally distributed across the onions and the products in general.
- Risk assessment versus annex 1 of Regulation 396/2005 → two preparations are sometimes necessary, depending on the product.
- One problem: no control measures are included so far at the sample preparation level.
- Recommendation: Start with the most important samples analysed by your laboratory and establish clear sample preparation procedures for them. A relana®-working group related to sample preparation in general and the sample preparation project of relana® in particular might be a helpful tool.

5. Interpretation of pesticide findings in vegetable oils

Discussion and remarks of the participants (selection):

The source of the processing factor must be mentioned in the test report. Why should the laboratory take the responsibility to judge which processing factor is the right one?

BNN-guideline value (0,01 mg/kg) is now used as a strict limit, which was not the idea. The use of processing factors is going into the same direction.

6. relana® Revision Project

Internal relana®-project!

7. Analytical challenges

7.1. Pesticides

Folpet / Phthalimid and Captan / THPI: new developments and results; consequences

Discussion and remarks of the participants (selection):

In general, the influence of the injector temperature is of high importance. Starting the injection at 60° C and increasing the injector temperature up to 200° C is one opportunity. The **relana® position papers** are very important and helpful to show what is possible and what is not possible from an analytical point of view.
EU-reference labs actually discuss, whether to change the residue definition or not. The reporting of residues still remains a problem (exceeding of MRL or specification limits although no Folpet was identified).
One relana®-laboratory explained that it cannot provide a reliable statement as the analyses does not allow to judge where the PI comes really from. The laboratory does not follow the residue definition as required by the MRL, while for Captan / THPI the laboratory follows the residue definition.

relana® should repeatedly communicate the situation about the analytical limitations of Folpet/PI analyses.

Acidic herbicides

The method ring test is scheduled for autumn 2017: The participating laboratories of the meeting were asked, which kind of products and acidic herbicides should be used for the acidic herbicides method ring test. The discussion resulted in a final design, including the active compounds (f.ex. 2,4-D, Haloxyfop etc.) and main metabolites and also some known conjugates of the selected acidic herbicides. Spiked samples as well as real samples with incurred residues will be part of the test. The main objective will be to verify the hydrolysis step and to compare the results of the MRM approach with the results of the SRM including the alkaline hydrolysis.

Paraquat / Diquat

So far, the analyses of samples for Paraquat resp. Diquat do not deliver stable / comparable results. Important: Less matrix leads to better signals. Carryover is no problem anymore, when applying a “QA”-sequence. A hot extraction is only applied for the dried products.

Experience by one relana®-lab: After changing to a different kind of HPLC-column, the analysis is very stable even after four months.

Maleic hydrazide

Overall question: Can maleic hydrazide be included into the multi-method scope?

For maleic hydrazide analyses, a high dilution of the sample extract is necessary. Maleic hydrazide is eluting in the dead volume, so it is in general difficult to be analysed and a high matrix suppression is observed. Therefore, a single method is necessary. The inclusion of maleic hydrazide into the multi-method scope is misleading,

TFNA: low recoveries in ring tests.

Discussion and remarks of the participants (selection):

The overall question discussed was, whereas the z-score model is more appropriate to evaluate the analytical results of TFNA, or if the trueness criterion really fits for the evaluation.

When analysing for TFNA, the matrix-effect strongly varies between different products.

Phosphonic acid: Where is the actual RL?

Discussion and remarks of the participants (selection):
To achieve a lower Reporting Limit (RL) it is necessary to use a most sensitive Triple Quad device and an analytical column, which fits best for Phosphonic acid.

The collection of data of "blanks" is of high relevance (blanks =samples showing no Fosetyl-Al but levels of Phosphonic acid) as this is always discussed (background levels of Phosphonic acid). So far, no published knowledge exists about "unavoidable" background levels of Phosphonic acid.

Phosphine

Short introduction of the aspect, that even very low levels of Phosphine (1 ppb) detected on organic products are used to question the organic status of the related food product. Announcement of a project on Phosphine by FIBL, Switzerland.

7.2. Contaminants

Nitrate analysis

Sample preparation approaches and corresponding discussions related to the aspect, that always a whole sample unit must be taken for homogenisation. This approach is not realistic and is not established by the laboratories under routine conditions. Typically, sub samples of the total laboratory sample unit are used for homogenisation.

MOSH / MOAH analyses

Exchange of experiences

General conclusion: The most demanding issue is the risk of contamination during analysis.

Each relana® laboratory was asked about the level of knowledge and corresponding interest in sharing knowledge with other relana® members. According to the information provided, relana® will set up working groups for sharing experiences within these groups according to the knowledge of the laboratories.