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relana®-Method-Ringtest
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Pesticides on Leaves

Part 1: Vine Leaves

(Part 2: Apple Leaves)

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1 Introduction

The analysis of leaves for pesticide residues in/on several fruit crops like apples, pears or grapes is often part of monitoring projects in order to verify good agriculture practice related to the specific requirements of the particular rules (f. ex. integrated cultivation, national or regional quality labels etc.).

Within the 2016 working programme of the laboratory quality circle relana® this topic is on the agenda in order to get a first overview about the laboratory performances and the comparability of pesticide residue analyses of such leave samples after common treatments with pesticide formulations.

2 Test material

In order to provide appropriate test material, it was agreed to make use of leaves, which have been treated by the growers in a common way during the current season. Vine leaves and apple leaves were identified as appropriate test materials for the method ring test, because these types of leaves are the most important ones analysed by the laboratories. This report is related to the analysis of vine leaves (Part 1), while a separate report refers to the results of the apple leaves (Part 2).

The vine leaves were picked at a private vineyard located in Alto Adige / Südtirol (Italy), near to Bolzano (Bozen). The variety of the leaves is called “Lagrein”, which is an autochthonous (indigenous) grape variety of Alto Adige.

The private grower applied all pesticide formulations he was recommended by the local vine growing association. Therefore, several pesticide applications were performed during the weeks before picking the leave samples. The total test material of leaves (ca. 3 kg) was mixed thoroughly and divided into 11 portions of 250 g each by Lach&Bruns.

Picking of the leaves took place the 28. June 2016. They were sent to Lach & Bruns and arrived at Hamburg the 29. June 2016. The samples were sent out to the relana® laboratories the 4. July 2016. To keep the leaves in an appropriate condition, the (already frozen) samples were packed and sent by making use of dry ice.

3 Results and Discussion

The samples were sent to the laboratories including an order sheet asking for the analysis of current pesticides. As the vine leaves contain incurred residues only, the evaluation of the results is done using the comparability criterion (z-score model). Every lab is given an individual lab code.

All in all, the laboratories have reported 18 different pesticides with significant levels above the common reporting limit (RL) of 0,01 mg/kg. Anyhow, as the results of Bupirimat (assigned value at 0,014 mg/kg) and Difenoconazole (assigned value at 0,013 mg/kg) are close to the common RL, no z-score evaluation is performed because some labs reported results of "detected, below RL". Therefore, 16 pesticides were evaluated using the z-score model.

Discussion of the results related to the particular pesticides

A) Pesticides showing a typical distribution of the results (12 pesticides)

The results related to

Buprofezin, Cyazofamid, Cyflufenamid, Dithiocarbamates, Dodine, Ethirimol, Fluopyram, Indoxacarb, Metrafenone, Penconazole, Spiroxamin, and Triadimenol

are well comparable with typical result distributions (bell-shaped-curve, Gaussian curve).

Related to these pesticides, only some few significant outliers are observed: Cyazofamid, Dithiocarbamates, Ethirimol, Indoxacarb. The inhomogeneity of the test material might be an option but also other reasons should be investigated.

Three labs did not analyse for Dithiocarbamates, although it was requested in the letter of instruction: "*Please analyse the vine leaves applying the typical analytical method(s) you would recommend to your clients related to such a sample of leaves*". As the use of Dithiocarbamate fungicide formulations are common in vineyards (which is confirmed by the assigned value in the vine leaves: 89 mg/kg), the analysis for Dithiocarbamtes should be offered resp. recommended related to such a sample. The three labs should take this into consideration.

B) Pesticides showing an atypical distribution of the results (4 pesticides)

Dimethomorph

The results of Dimethomorph show two significant **clusters** of results:

- Five labs reported levels between 30 µg/kg and 39 µg /kg, which is a very homogenous distribution.
- Six labs reported levels between 110 µg /kg and 160 µg /kg, which is also a very homogenous distribution.

This is a very surprising situation, which cannot be explained at the moment.

Folpet

The results of Folpet differ significantly from each other. Once again, there are two clusters of results: six labs with levels between 40 mg/kg and 48,7 mg/kg and three labs with results between 63 and 69,9 mg/kg. Two outliers are present as well: 6,9 mg/kg and 91,5 mg/kg. Nevertheless, the results of 9 out of 11 labs are close together taking into account, that non-homogenised leaves with incurred residues were analysed. This is a good result related to such a demanding parameter like Folpet.

Tetraconazole

The results of Tetraconazole differ significantly from each other, covering a concentration range from 12 µg/kg to 110 µg/kg, which is a factor of 10. Homogenous clusters of results cannot be identified. The robust standard deviation is significantly exceeding the Horwitz deviation (thus the expected deviation).

Zoxamide

The results of Zoxamide differ significantly from each other, covering a concentration range from 30 µg/kg to 350 µg/kg, which is again a factor of ca. 10. Homogenous clusters of results cannot be identified. The robust standard deviation is significantly exceeding the Horwitz deviation (thus the expected deviation).

Conclusion

All in all, the participating laboratories present satisfying performances of pesticide residue analyses in/on vine leaves. The described deviations related to particular pesticides will be discussed and questioned individually with the particular laboratories.