

QA/QC IN BEEHIVE PESTICIDE MULTIRESIDUE ANALYSIS FOR ENVIRONMENTAL BIOMONITORING

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INTRODUCTION

Beehives are currently being used as environmental biomonitors of pesticide residues. The quality assurance and quality control of their analysis must be performed in order to obtain reliable results for environmental biomonitoring purposes. The aim of the present work was to explore the usefulness of applying the concepts of SANTE guidelines (SANTE/12682/2019) and Shewhart charts for these particular analysis. Following the on-going quality control recommendations of SANTE guidelines a single recovery experiment of all the studied analytes was introduced in each analysis batch (15-20 depending on the matrix) since the year 2014. When the extension of the scope was performed, new analytes were also included previously conducting the initial validation.

EXPERIMENTAL

Seasonal samplings were carried out (autumn, winter, spring, summer)

A recovery test and a blank (sample containing no residues) are included with each set of actual samples to be analyzed. Thus, to date there are n = 14 (bees), n = 15 (capped honey), n = 20 (beeswax) and n = 15 (pollen)

Percentage recoveries of these experiments were used to graph the Shewhart chart for each matrix and to calculate the within laboratory reproducibility, RSDwR.

The sample preparation method is based on the QuEChERS method

Extraction: MeCN, Buffer citrato
Clean up: Freeze-out + dSPE (PSA+C18+GCB)

Extraction: MeCN
Clean up: Freeze-out + Hexane LLE + dSPE (PSA + C18)

Extraction: MeCN, Buffer citrato
Clean up: MgSO₄ + PSA

Extraction: MeCN, Buffer citrato
Clean up: Freeze-out + dSPE (MgSO₄+PSA+C18)

DISCUSSION AND CONCLUSIONS

Most of the pesticide recoveries in each batch ranged between 70 and 120% with a RSDwR below 20%.

The exceptions were carbendazim in bees, beeswax and capped honey; imazalil in beeswax; clothianidin and imidacloprid in pollen; boscalid and atrazine in capped honey.

Deviations in punctual batches indicated specific instrumental problems which needed to be addressed and fixed and the samples re-injected.

For positive findings it is important to look at the chart and verify that the pesticide in that batch is under control or it needs to be re-analyzed.

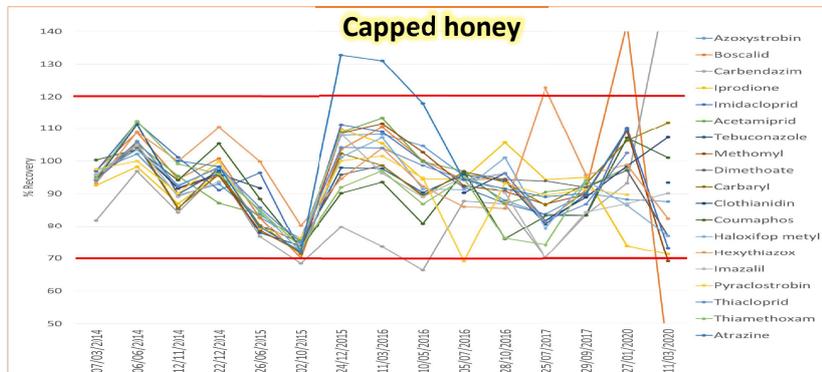
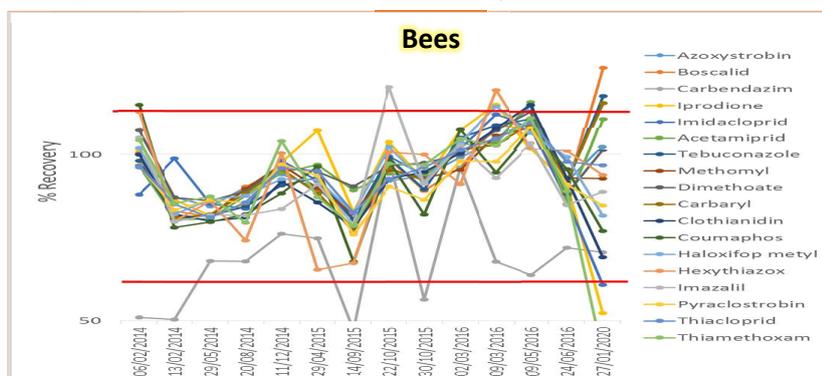
The on going control of the analytical process gives confidence to the results of our monitoring programs.

The statistical tool and SANTE guidelines concepts were applicable for the QA/QC of beehives' pesticide multiresidue methods for environmental biomonitorings.

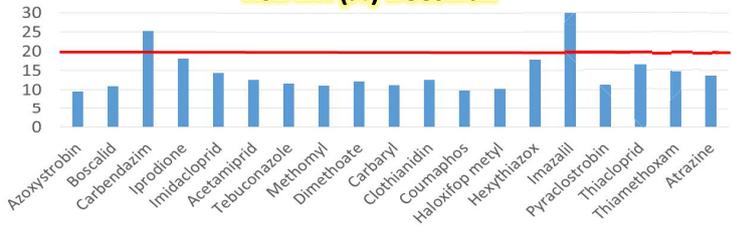
ACKNOWLEDGEMENTS

RESULTS

Shewhart charts



RSDwR (%) Beeswax



RSDwR (%) Pollen

