

# Validation of pesticides in honey without sample preparation – dilute and shoot



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## Introduction

- Honey: high sugar, low fat content; pesticides to be found are more polar
- LC-Q-TOF: allows for detection of a nearly unlimited number of analytes in a single run
- Objective of this study:
  - simplification of sample preparation strategies → test of a generic sample preparation method
  - development of a LC-Q-TOF based screening method for > 300 pesticides in honey
  - validation according to SANTE/11312/2021 [1]



## Methodology

Table 1: LC-Q-TOF instrument parameters

LC-system	UHPLC
Type	Nexera X2, Shimadzu
Analytical column	Shim-pack Velox Biphenyl 2.1 mm x 100 mm, 2.7 μm
Injection volume	5 μL
Eluent A	water + 2 mM ammonium formate + 0.004% formic acid
Eluent B	methanol + 2 mM ammonium formate + 0.004% formic acid
Total run time	15 min
MS-system	Q-TOF
Type	LC-MS-9030, Shimadzu
Ionization mode	ESI +, ESI -
Resolution	30,000
Data acquisition	DIA
CE spread	5 – 55 V

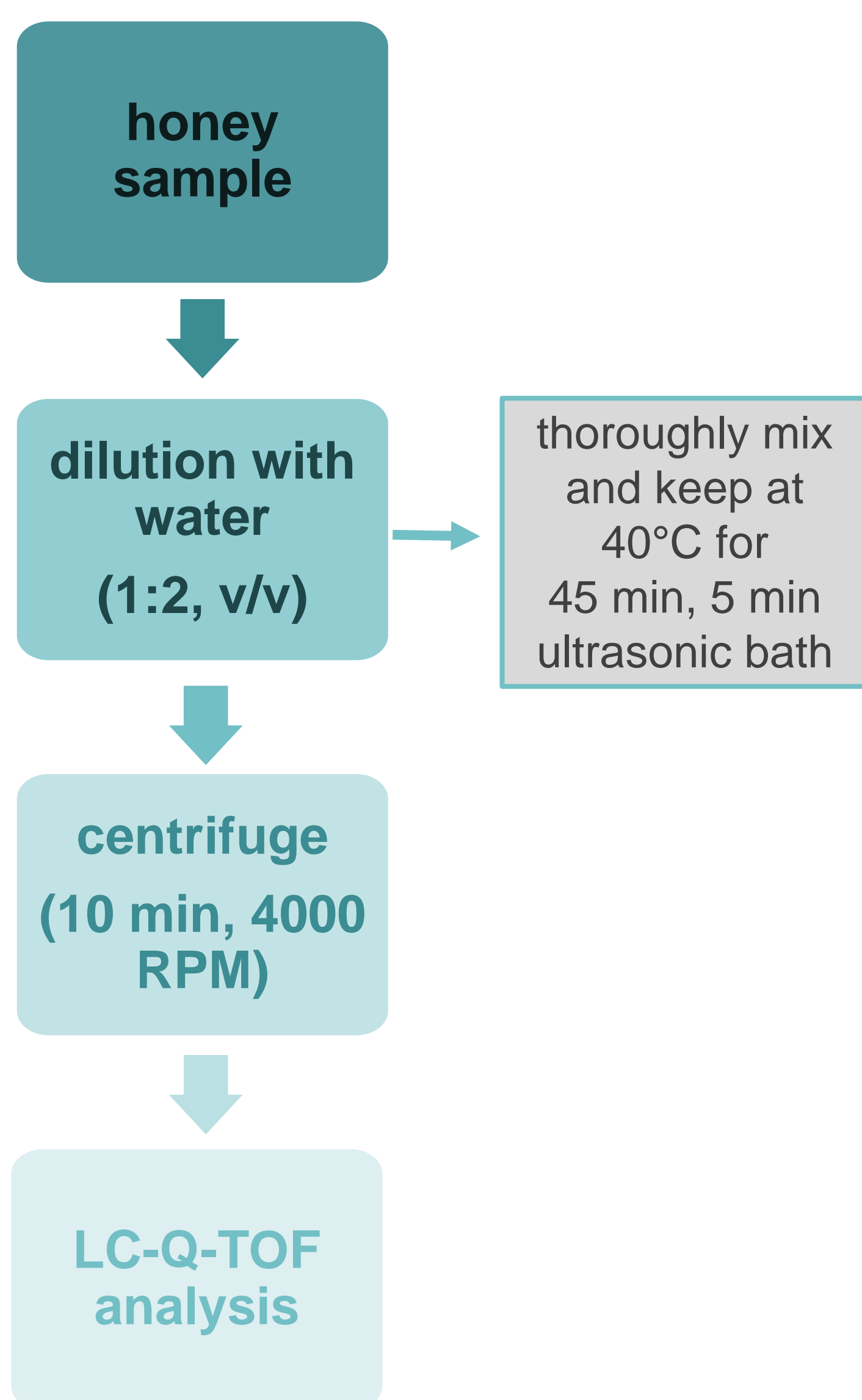


Fig. 1: Sample preparation strategy „dilute and shoot“



Fig. 2: LC-Q-TOF, Shimadzu

## Results

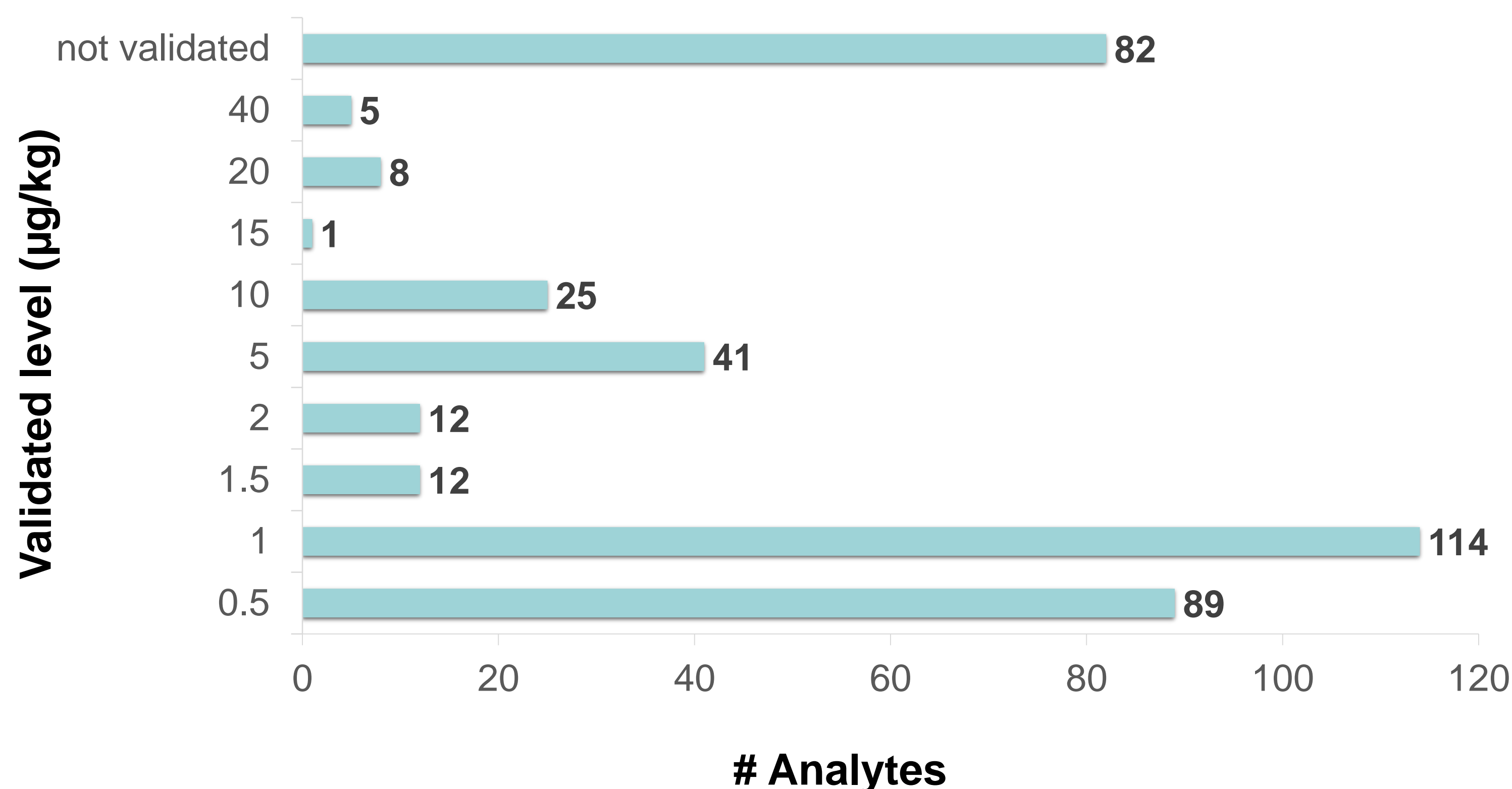


Fig. 3: Number of compounds meeting the method validation criteria

### > 82 analytes could not be validated:

- for 24 analytes at least one method validation criteria was not fulfilled (e.g. precision >20%)
- 19 analytes were successfully validated in previous studies using QuEChERS method
- some compounds (e.g. topramezone, fipronil + metabolites) are known to be single residue method (SRM) compounds
- some compounds (e.g. dithianon, ethoxyquin) are instable during sample preparation

## Conclusion

### > Pros

- rapid and sensitive multi-residue method for simultaneous determination of >300 pesticides
- analytes with a wide range of physico-chemical properties are covered
- suitable for screening of unknown pesticides and/or metabolites → non-target analysis

### > Cons

- low removal of matrix (sugar)
- higher ion contamination compared to a QuEChERS extract

> for routine analysis QuEChERS is better suited!

## References

[1] Analytical quality control and method validation procedures for pesticide residues analysis in food and feed SANTE 11312/2021

[2] EURL-AO report: Analysis of pesticide residues in honey using LC-Q-TOF; available from CIRCA BC