

# QuPPE, a Soon to be Official Method for Polar Pesticides

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## Introduction

The development and validation of methods for the analysis of pesticides that are not amenable to multiresidue methods is one of the core activities of the EU Reference Laboratory for Single Residue Methods (EURL-SRM).



Highly polar pesticides have been excluded for a long time from the routine scope of laboratory investigations due to a lack of simple methods that are able to cover more than one pesticide at a same time. With the QuPPE (**Q**uick **P**olar **P**esticides) method the EURL-SRM has filled this gap.

## Analytical method

The goal was to develop a simple fast and readily transferable method for these compounds by employing a common extraction step followed by as few as possible LC-MS/MS runs using standard instrumental configurations. The use of isotope labeled analogues of the target analytes as internal standards (ILISs) eliminated various sources of errors and helped to keep the method streamlined and simple. Quantification is, in most cases, performed with the help of ILISs.

Weigh 10 g sample homogenate in 50 mL centrifuge tube

Add 10 mL MeOH containing 1% formic acid

Shake thoroughly for 1 min

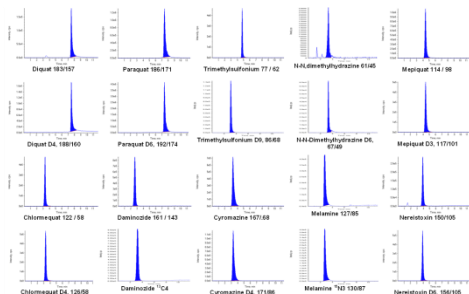
Centrifuge at 4000 rpm for 5 min

Filter 1.5-3 mL supernatant into a plastic tube to remove any particles (syringe filter 0.45 µm)

Transfer 1 mL extract to plastic vials

LC-MS/MS analysis

Typical chromatograms of standards (0.1 mg kg<sup>-1</sup>) and the corresponding labeled ILIS in ACN.



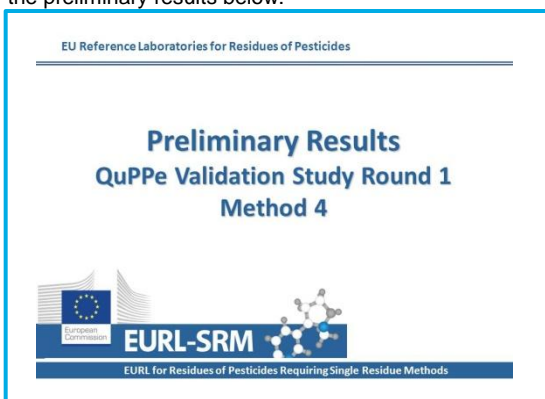
Method details: [www.eurl-pesticides.eu/](http://www.eurl-pesticides.eu/)

## Validation Study

With the intention of turning the QuPPE method into a standard procedure (CEN), the EURL-SRM, in collaboration with German NRL-SRM, organized an interlaboratory validation study. The whole validation round focused on LC-MS/MS method 4 and included the following substances: Cyromazine, Daminozide Chloromequat, Mepiquat, Trimethylsulfonium, Nereis-toxin, Paraquat, Diquat and Melamine. Validation experiments were conducted on five matrices avocado, grape, potato and rye at three different levels (0.01, 0.05 and 0.2 mg kg<sup>-1</sup>) and milk at two different levels (0.01 and 0.05 mg kg<sup>-1</sup>).

## Results

Sixteen laboratories from eight countries participated in the first part of the validation with some results still being expected. You can find the preliminary results below:



## Summary

According to the preliminary results from the validation with exception of Paraquat and Diquat all compounds were successfully validated. Normally Diquat and Paraquat require different extraction conditions and an extra heating step. For these two compounds, an extra validation round is planned. Rye was the most problematic commodity at low levels. Following proper evaluation the data will be uploaded onto the EURL-DataPool. Round 2 will focus on the method 1.3.<sup>1</sup>

## Acknowledgement

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## Reference

1. [www.eurl-pesticides.eu](http://www.eurl-pesticides.eu)