

A New Automated On-line QuEChERS - HPLC Direct Injection Clean-up for Difficult and Standard Matrices

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Introduction and Background

Quick, Easy, Cheap, Effective, Rugged, and Safe: QuEChERS: The QuEChERS methodology has become the leading method for pesticide determination in food and feed labs throughout the world. Nevertheless, in many cases difficult to analyse matrices, such as teas or spices, were analysed without using QuEChERS. Consequently, many labs are searching for a working and automated alternative. The new QuEChERS automation concept is based on the second clean-up step applying a non-dispersive approach in specifically adapted cartridges containing a proprietary sorbent. Furthermore, as a novelty the eluted extract is subsequently on-line injected onto the LC-MS/MS. Because of the high matrix-retention capacities of the cartridge in combination with the non-dispersive and concentration approach, a minimised ion suppression with high recoveries and low standard deviations is observed. Since the new methodology is successfully applied to tea, standard matrices such as apples, bell peppers, lettuce, and lemons were tested as well and gave comparable results. On this poster, the automation concept of the FREESTYLE SPE - HPLC Direct Injection system designed by LCTech GmbH is presented as well as the results for tea matrix.

Material and Methods

Homogenise 5 g of tea in a blender and weigh out 2 g into a 15 mL Falcon tube. Add 10 mL of water, 10 µL of internal standard and vortex vigorously for 1 min. Wait for 10 min. Add 10 mL of acetonitrile (pesticide grade), buffer mix, and vortex vigorously for 1 min. Afterwards centrifuge for 5 min at 3,000 rpm. Pipette 3 mL of the supernatant, fill it into a 4 mL vial with septum and sealing cap, and put the sample into the robotic system FREESTYLE QuEChERS.

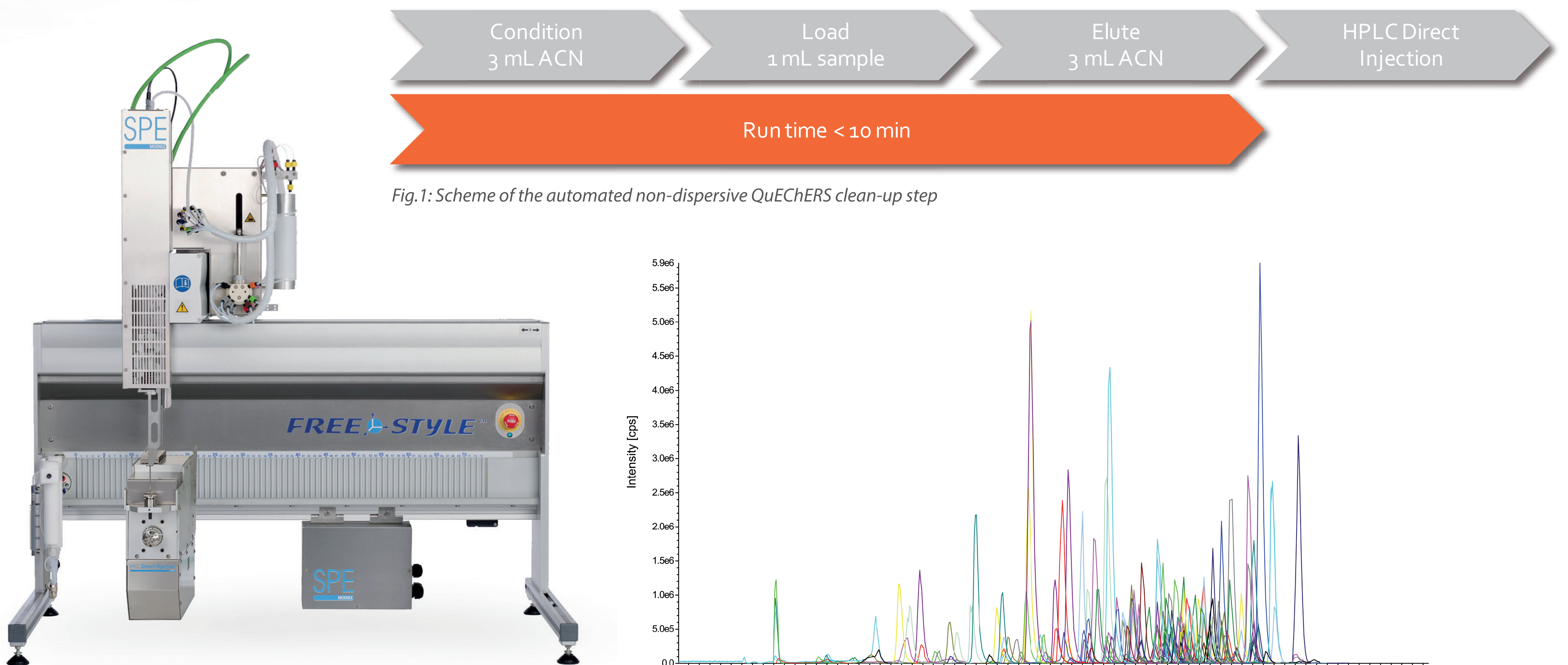


Fig. 1: Scheme of the automated non-dispersive QuEChERS clean-up step

Fig. 2: Robotic system FREESTYLE SPE with HPLC Direct Injection module

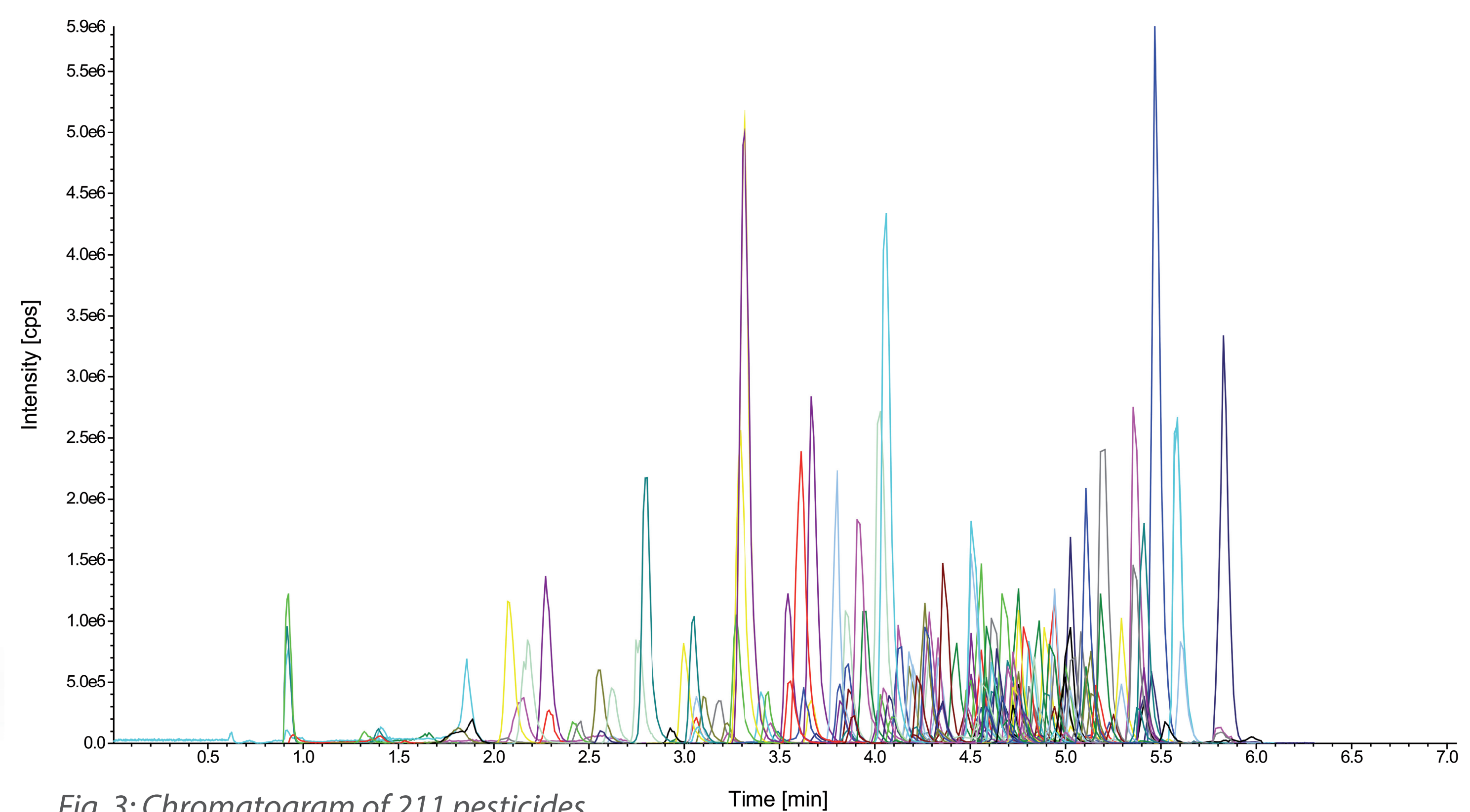


Fig. 3: Chromatogram of 211 pesticides

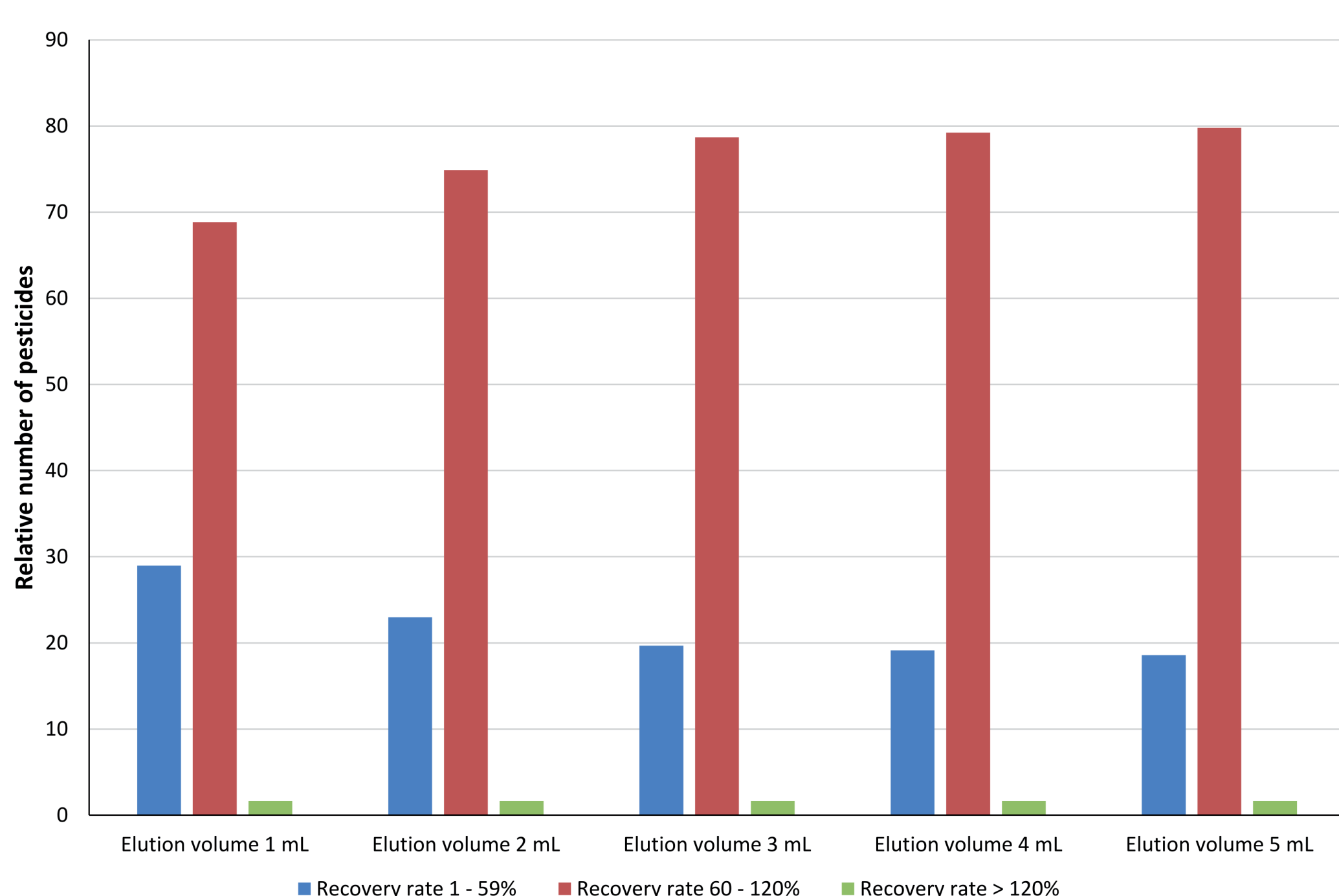


Fig. 4: Relative number of pesticides at different elution volumes

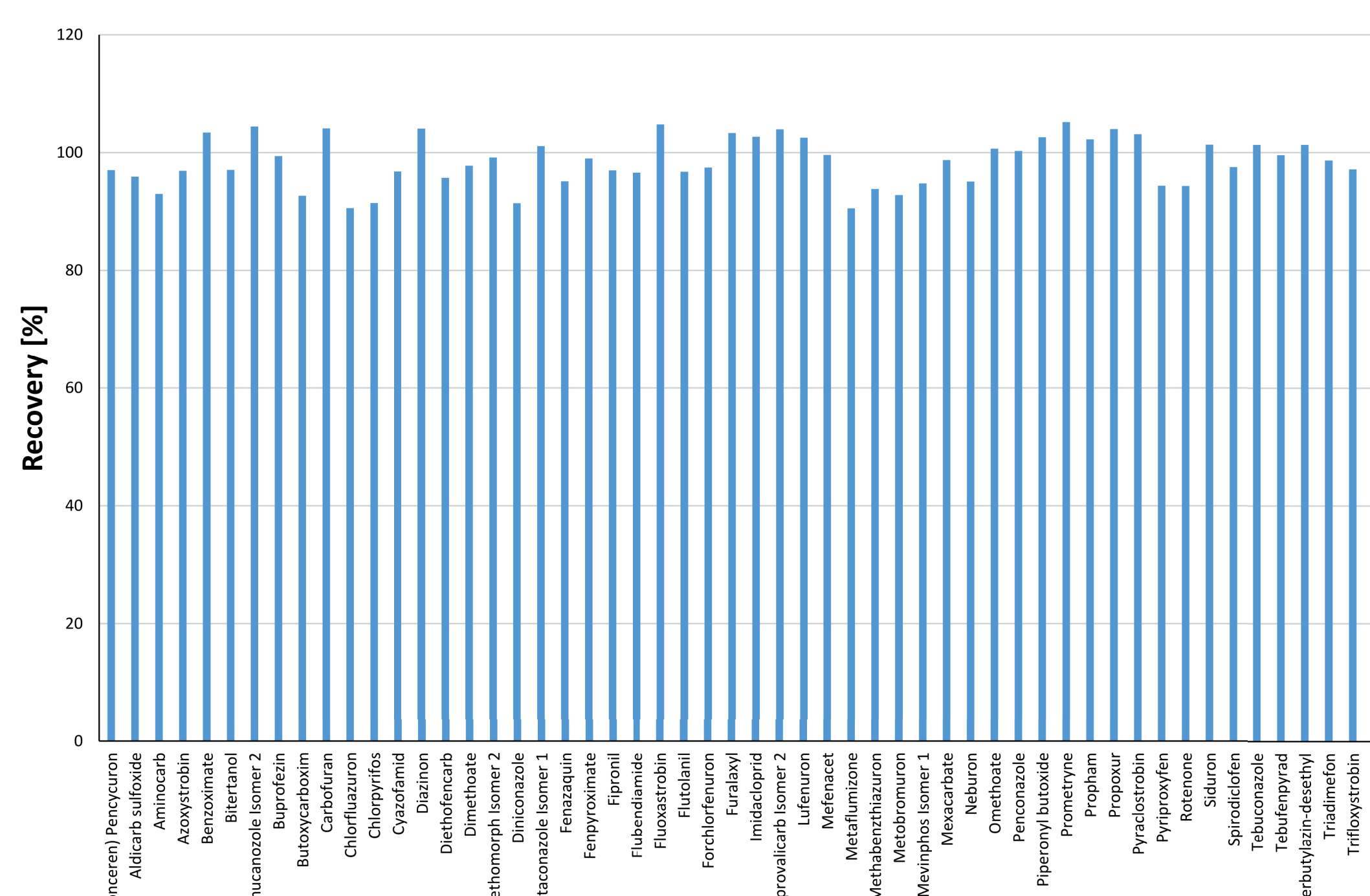


Fig. 5: Recovery rates of selected pesticides



Results and Discussion

Tea as a difficult matrix as well as four other vegetable and fruit matrices were tested on the new FREESTYLE QuEChERS automation in combination with a specifically adapted cartridge. The new cartridge works very well with acetate and citrate buffered extracts, respectively, where 77 % of the analytes could be detected within the accepted recovery range of 60 to 120 % with a mean value of 98 %. 19 % were below the accepted range, still with a mean recovery of 28 %; thus they are quantifiable. The overall mean recovery < 120 % was 92 %. 2 % had a recovery higher than 120 %, and 5 pesticides seem not to be applicable to this methodology. Due to the high level of automation and the non-dispersive approach, the extracts were cleaner compared to a standard QuEChERS approach and showed a very good reproducibility. The well-established and approved FREESTYLE system is a simple but robust platform for this application. Besides, the system does not have too much moving or electrical parts that require service and downtime. Furthermore, as the system can work fully unattended over night or the weekend, and a run takes less than 10 min, it is a great support for any high-throughput routine pesticide lab.

➔ For more information, please have a look at the corresponding application note at www.lctech.de/en/downloads/applications