Introduction
The newly developed QuEChERS-multiresidue method has been successfully employed at the CVUA Stuttgart laboratory for two years. The method is clearly less expensive and more efficient than traditional methods, which are typically more complicated, laborious, time consuming, and require high amounts of solvents. The amenability of the final extracts (in acetonitrile) to both, LC and GC analysis further ensures a wide analytical scope.

Mini-Multiresidue-Method (QuEChERS)
The QuEChERS-method is characterized by a streamlined procedure. Many complicated analytical steps commonly employed in traditional methods have been omitted or replaced by easier ones, as shown below.

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
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<tbody>
<tr>
<td>1</td>
<td>Weigh 10 g of Sample (50 mL Teflon-Tube)</td>
</tr>
<tr>
<td>2</td>
<td>Add 10 mL Acetonitrile (acidified with HAc for samples with pH &gt; 5)</td>
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<tr>
<td>3</td>
<td>Shake vigorously 1 min</td>
</tr>
<tr>
<td>4</td>
<td>Add 4 g MgSO₄ and 1 g NaCl</td>
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<tr>
<td>5</td>
<td>Shake vigorously 1 min</td>
</tr>
<tr>
<td>6</td>
<td>Add ISTD-Solution</td>
</tr>
<tr>
<td>7</td>
<td>Shake 30 s and centrifuge</td>
</tr>
<tr>
<td>8</td>
<td>Take Aliquot and Add MgSO₄ (and Sorbent)</td>
</tr>
<tr>
<td>9</td>
<td>Shake 30 s and centrifuge</td>
</tr>
<tr>
<td>10</td>
<td>Add 0.1% HAc and “Analyte Protectants”</td>
</tr>
<tr>
<td>11</td>
<td>GC-MSD and LC-MS</td>
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</tbody>
</table>
1. Weigh 10 g Sample

2. Add 10 mL MeCN (acidified with HAc for samples with high pH)

3. Shake Intensively for 1 min

4. (Pre-)Weigh 4 g MgSO$_4$ + 1 g NaCl (the use of a scoop simplifies this step)

5. Add to the Tube

6. Shake Intensively for 1 min
7. Add ISTD

8. Shake for 30 s

9. Centrifuge (ca. 5 min)

10. Separated Raw Extract

11. (Pre-) Weigh MgSO₄ and PSA
    (the use of a scoop simplifies this step)

12. Add Extract to Tube and Shake ca. 30 s
13. Centrifuge (ca. 2 min)

14. Purified Extract

15. Add “Analyte Protectants” (optional)

16. Ready for GC or LC analysis

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<thead>
<tr>
<th>Complicated or Error-Prone Steps in Traditional Methods</th>
<th>Simplified Alternatives</th>
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<tr>
<td>Sample processing/homogenization</td>
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<td>Blending (e.g. with Ultra-Turrax)</td>
<td>Shaking</td>
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<tr>
<td>Filtration of matrix bulk</td>
<td>Centrifugation</td>
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<tr>
<td>Multiple partitioning steps</td>
<td>Single partitioning (“On-line”-approach)</td>
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<tr>
<td>Separation/transfers of entire extract</td>
<td>Take aliquots (use ISTD)</td>
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<tr>
<td>Use of excessive glassware</td>
<td>Extraction/partitioning in single vessel</td>
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<tr>
<td>Evaporation/reconstitution</td>
<td>Large volume inj.; sensitive instruments</td>
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<td>Classical SPE with columns &amp; manifold</td>
<td>Dispersive SPE</td>
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Advantages

- **Rapid** (8 samples in less than 40 min)
- **Simple** (no laborious steps, minimal sources of errors)
- **Cheap** (ca. 1 € per sample for the sample preparation, for 1 mL extract)
- **Low solvent consumption** (10 mL acetonitrile)
- **Practically no glassware needed**
- **Covers a wide pesticide range** (polars, pH-dependent compounds)
- **Extract in acetonitrile** (GC- and LC-amenable)

Validation Studies

**Recoveries:** Following the above procedure recovery studies have been performed for more than 330 pesticides. The recoveries achieved from various matrices lay between 70 and 110 % (usually between 90 and 100 %), and RSD’s mostly between 2 and 8 %. Some typical results are shown below.

Note: Some of the recovery tests were performed without the use of PSA for cleanup. In the case of acidic and alkaline-sensitive compounds this was done on purpose to avoid losses. However, there are also some cases (e.g. azinphos-methyl) in the table below, were the not use of PSA was merely due to the experimental setup.
QuEChERS-Recoveries (exemplary)

Matrix: Apple (if not else mentioned)
Fortification Level: 0.1 mg/kg (n=5)
Calibration Method: Matrix matched
Determination: GC-MSD (SIM); LC-MS (SIM); LC-MS/MS
RSDs: Mostly between 2-8 % (not shown here)

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**QuEChERS-Recoveries (exemplary)**

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## QuEChERS-Recoveries (exemplary)

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### EU-PT 4

**Participants:**
111 Laboratories

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**Note:** Acidified acetonitrile was used for extraction to minimize degradation of base sensitive compounds.
Analytical range

The recovery studies have shown that the method is capable of effectively extracting analytes of a broader polarity range than traditional methods. Excellent recoveries were achieved for analytes with log Kow ranging from −2.6 to 7.1. Basic and acidic pesticides, usually requiring a drastic pH shift in traditional methods, were recovered to a high percentage even at pH levels where they are expected to be present predominantly in the ionic form.

Implementation in the Laboratory - Experiences

The introduction of the QuEChERS method for routine analyses in our laboratory (CVUA Stuttgart) resulted in a dramatic reduction of solvent consumption. The savings resulting from the reduction of solvent consumption were as high as 15000 € per year compared to previous years where a method with a consumption of 215 mL halogen-free solvent per analysis was used. Compared to the common Specht and Specht-Online methods the solvent consumption is reduced by a factor > 50.
In terms of manual work, the introduction of the method resulted in a 6-8-fold reduction of the time invested in sample preparation compared to the optimised Specht-Online method. The method's fastness and the inclusion of several “difficult” analytes that were only analyzed by additional single residue methods in the past, allowed us to shift personnel from the sample preparation to the increasingly demanding instrumental analysis area.
The introduction of the QuEChERS method has furthermore enabled us to gradually expand the spectrum of targeted pesticides both, directly (broader analyte spectrum covered) and indirectly (more personnel available to operate novel GC & LC instruments) and contributed to the reduction of the large “grey area” of pesticides for which no residue data existed in the past. This fact is reflected by the increasing number of different pesticides found in fruit and vegetables.