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Herrmann, Susan Strange; Poulsen, Mette Erecius

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Hay for livestock feeding – Method validation

Susan Strange Herrmann and Mette Erecius Poulsen
DTU, National Food Institute, Kemitorvet, 2800 Kgs. Lyngby, Denmark. e-mail: sher@food.dtu.dk

Background:
Hay and grass may account for up to 100% of the feed for dairy and beef cattle. Thus hay may be of high importance for the pesticide residue exposure of livestock. Hay was therefore chosen as test material for EUPCT-CF12 carried in January/February 2018. Method performance using QuEChERS according to EN 15662 and dSPE employing three different kits (Table 1) were studied for a selection of analytes (Figure 1). Using 1 gram of sample QuEChERS (EN 15662) performed equally well or better than when modifying the method by using one of the three other dSPE kits. 402 pesticides and metabolites of pesticides were therefore validated on hay using QuEChERS extraction according to EN 15662 and analysis by LC-MSMS and GC-MSMS. The pesticides and metabolites validated are listed in Table 3. The validation was performed in accordance with the requirements outlined in SANTE/11813/20173.

Analytical procedure:

Figure 1: Recoveries of selection of pesticides spiked into blank hay samples (0.05 mg/kg) using QuEChERS extraction with dSPE according to proc. 1-4.

Table 1: Test of dSPE procedure for clean-up of QuEChERS hay extracts

<table>
<thead>
<tr>
<th>dSPE kit</th>
<th>Super+ QuE</th>
<th>QuE</th>
<th>Sup+ QuE</th>
<th>QuE</th>
<th>Sup+ QuE</th>
<th>QuE</th>
<th>Sup+ QuE</th>
<th>QuE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Contents</td>
<td>100 mg Supelclean PSA 900 mg MgSO4</td>
<td>100 mg Supelclean PSA 90 mg citrate</td>
<td>100 mg Supelclean PSA 90 mg citrate</td>
<td>100 mg Supelclean PSA 90 mg citrate</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Recovery</td>
<td>0.05</td>
<td>0.05</td>
<td>0.05</td>
<td>0.05</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Results</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Homogenisation of test material

Chopping and milling (size < 0.1 mm)

Figure 2: GC-MSMS 219 39 38 59(47)*-128 93 2-28 10 296

Results:

Table 2: Overall results for validation performed on hay using QuEChERS (EN 15662) with sample size reduced to 1 gram. (*) recoveries for chlorothalonil was 47%

<table>
<thead>
<tr>
<th>LOQ (mg/kg)</th>
<th>Recoveries</th>
<th>RSDr</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.025</td>
<td>0.05</td>
<td>0.5</td>
</tr>
<tr>
<td>GC-MSMS</td>
<td>219</td>
<td>219</td>
</tr>
<tr>
<td>LC-MSMS</td>
<td>214</td>
<td>214</td>
</tr>
<tr>
<td>Total no. of validated compounds excluding duplicates</td>
<td>402</td>
<td></td>
</tr>
</tbody>
</table>

Conclusion: QuEChERS (EN 15662) gave satisfactory extraction of analytes from hay (Figure 1, Table 2). In total 402 pesticides and metabolites of pesticides were validated. 296 were GC-MSMS amenable compounds and 297 were LC-MSMS amenable. An LOQ of 0.025 mg/kg were obtained for majority of the pesticides and metabolites (see Table 2).

From inspection of GC-MS full scan chromatogram the hay extract obtained with QuEChERS (EN 15662) was found to be relatively low compared to the amount of co-extract observed for oat (Figure 2).