Expansion of cereal multi residue method with pesticides planned for review under regulation No 396/2005 Article 12

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Publication date: 2016

Document Version
Publisher’s PDF, also known as Version of record

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Citation (APA):
Expansion of cereal multi residue method with pesticides planned for review under regulation No 396/2005 Article 12

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Introduction

Article 12 in Regulation No. 396/2005 states that assessment of existing MRLs should be performed by the Authority within a period of 12 months from the date of the inclusion or non-inclusion of an active substance in Annex I to Directive 91/141/EEC. All compounds included in the present validation are included in the so-called MRL progress list, which lists the active compounds coming up for review under article 12 in Regulation No. 396/2005. In connection with the review, information on the availability of methods for enforcement, availability of standards and achievable LOQs are needed. In order to obtain this information the present validation work was performed.

Validation, of the 22 pesticides or metabolites of pesticides, was performed on oat, rye and wheat samples spiked with 0.01, 0.02 and 0.1 mg/kg using QuEChERS extraction according to CEN method 15662 for dry matrices. Though extracts were withdrawn prior to dSPE in order to determine if specific analytes were adsorbed by the PSA.

Validation results

<table>
<thead>
<tr>
<th>Spike level mg/kg</th>
<th>LC-MS/MS</th>
<th>GC-MS/MS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>with PSA clean-up</td>
<td>no clean-up</td>
</tr>
<tr>
<td></td>
<td>Recovery %</td>
<td>RSDr %</td>
</tr>
<tr>
<td>0.01</td>
<td>96 14 105</td>
<td>101 8 15</td>
</tr>
<tr>
<td>0.02</td>
<td>101 8 15</td>
<td>100 6 10</td>
</tr>
<tr>
<td>0.01</td>
<td>96 14 105</td>
<td>101 8 15</td>
</tr>
<tr>
<td>0.02</td>
<td>101 8 15</td>
<td>100 6 10</td>
</tr>
</tbody>
</table>

Conclusion

All 22 analytes were LC-MS/MS amenable, and 9 also GC-MS/MS amenable. Using the QuEChERS CEN method 15662 for cereals, including the dSPE with PSA, 17 analytes were validated at 0.01 mg/kg using LC-MS/MS.

If the results for oat were excluded the RSDr and RSDR would for several analytes be reduced, e.g. for difenacoum (LC) and spirotetramat-keto-hydroxy (GC).

21 analytes were amenable to QuEChERS without dSPE clean-up and detection by LC-MS/MS. Only for pyridalyl were the inclusion of the dSPE step required. Excluding the dSPE step reduced the RSDr for several analytes considerably.

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