

# Acaricides and related compounds in bovine hair and poultry feather: Development and validation of a LC-HRMS screening method

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## **Introduction and Objective**

Acaricides could be used as veterinary drugs or pesticides to eliminate mites and ticks. Several acaricides are allowed to be used in animal husbandry and are registered as veterinary drugs. However, most acaricides are not registered as veterinary drugs. In 2017 unregistered (for poultry) Fipronil was used in many poultry farms in the Netherlands and the compound was detected in eggs. This raised the question: which (unregistered) acaricides and related compounds, with similar properties, are used in animal husbandry? To answer this question, a qualitative UHPLC-high resolution MS screening method was developed and validated for the screening of a high number of acaricides and related compounds in feathers and hair and a survey study was carried out to monitor the use of these compounds.

#### **Results validation**

The validation was performed for 198 acaricides and related compounds according to Commission Decision 2002/657/EC.

• Of the 198 compounds only 21 are not detectable at a level of 1000 µg/kg.

#### **Developed and validated method**



#### Survey

For the survey 63 bovine hair samples and 48 feather samples were screened with this method. Results are presented in figure 2. These results were confirmed with a targeted LC-MSMS method according to CD 2002/657/EC.





**Figure 1.** Overall CCβ of 177 acaricides and related compounds in bovine hair and poultry feather.

• The method is selective for 99% of the compounds. It is only not selective for pyrethrin I without measuring pyrethrin II.

• The method is robust, except



Chicken	Turkey	Bovine
Unregistered compounds		Unregistered compounds
<ul><li>Fipronil (+metabolite)</li><li>Bendiocarb</li></ul>		<ul><li>Allethrin</li><li>Diazinon</li></ul>
Registered compounds		Registered compounds
<ul> <li>Ponazuril/Toltrazuril</li> <li>Flubendazole</li> <li>Pyriproxyfen</li> <li>Spinosyn A &amp; D</li> </ul>	<ul> <li>Ponazuril/Toltrazuril</li> <li>Thiamethoxam</li> <li>Levamisole</li> </ul>	<ul> <li>Thiamethoxam</li> <li>Cyromazine</li> <li>Permethrin</li> <li>Pyrethrin/Cinerin</li> <li>Imidacloprid</li> <li>Ivermectine</li> <li>Triclabendazole (+metabolite)</li> </ul>

Figure 2. Confirmed compounds in bovine hair and poultry feathers based on initial screening results

The presence of fipronil is not surprising because the feathers came from a location infected with fipronil.

#### **Conclusions**

- The method of analysis developed in this study is suitable for the screening of 177 acaricides and related compounds in bovine hair and poultry feather. The qualitative screening method was fully validated according to CD 2002/657/EC.
- Several registered and unregistered acaricides and related compounds were found in the survey in bovine hair and poultry

for dichlorphos and formothion. Evaporating the sample extract for half an hour after dryness has a negative effect on these compounds.

• 97% of the compounds are stable in the final extract for 1 month in the freezer.

- feather. The survey demonstrates the usefulness of the developed screening method. The method can be used for monitoring the use of acaricides and related compounds in animal husbandry.
- The method can be used to detect any application of unregistered acaricides at an early stage.

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