

Hans Wunsch, Markus Walter, Kai Lenz, Eckard Jantzen, Juergen Kuballa

GALAB Laboratories, Max-Planck-Str. 1, D-21502 Geesthacht, Germany
Tel +49 (0) 4152-889-400, Fax +49 (0) 4152-889-401, www.galab.de, info@galab.de

Introduction

GALAB Laboratories is an international operating service provider of quality control analysis for food and feeding stuff products. Our highly qualified staff, modern accredited laboratories and state-of-the-art in analytical instrumentation guarantee best quality of our services. Our commitment to customer demands and satisfaction make us a reliable and sustained partner for the food and animal feed industry as well as the authorities. GALAB Laboratories provides a wide range of vet drug residue analysis in different matrices at lowest levels for high product safety.

Antibiotics are mainly used in food and feeding stuff for the treatment of bacterial diseases. As there is a wide spectrum of antibiotics the need to detect these compounds at very sensitive levels comes more and more into focus for the food and feeding stuff industry. In Annex 3 of the Council Directive 2377/90 compounds are listed with maximum residue limits (MRLs) in different matrices for e.g. sulfonamides, tetracyclines,

quinolones. In Annex 4 MRLs could not be elaborated which means a zero tolerance in all foodstuffs of animal origin for e.g. nitrofurans metabolites, chloramphenicol, imidazoles. The presence of these compounds at trace level and the complexity of different matrices requires very sensitive methods of determination. According to the German Foodstuff Law of 2003 lasalocid must not be used in feeding stuff to laying hens older than 16 weeks. The aim of the developed method was to establish a method for the reliable detection of coccidiostats, e.g. lasalocid in food and animal feed.

The poster shows an example of a contamination of feeding stuff and eggs at trace level by coccidiostats incl. method validation according to DIN ISO 32645 and Council Directive 96/23/EC. LC-MS-MS technique offers the possibility to develop sensitive and specific residue methods to ensure the quality of food and feeding stuff products.

Experiments

Feeding stuff: Extraction/clean-Up of coccidiostats

- 2 g sample
- NaOH
- ethylacetate
- cyclohexane
- SEC
- LC-MS-MS (API 4000)

Food: Extraction/clean-Up of coccidiostats

- 2 g sample
- H₂O/NaOH
- ethylacetate
- cyclohexane
- SEC
- LC-MS-MS (API 4000)

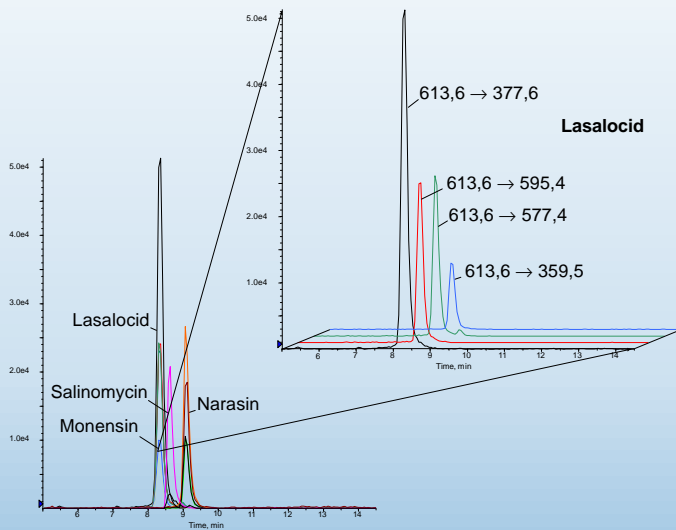


Fig. 1: Real feeding stuff sample containing lasalocid of a concentration of 3,5 µg/kg, four transitions of lasalocid are shown

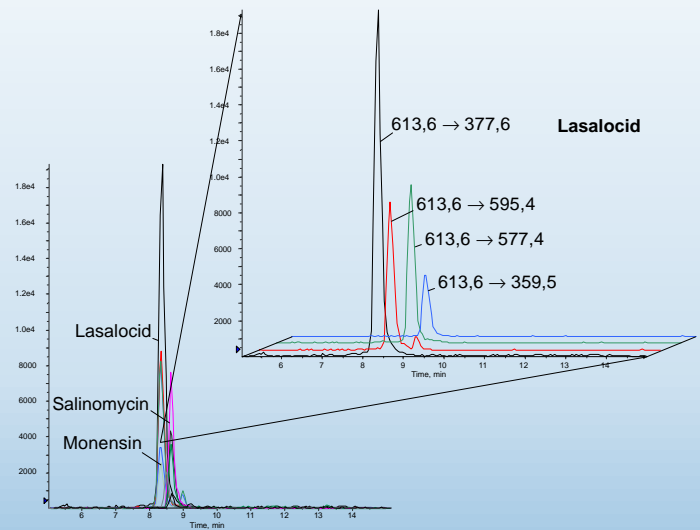


Fig. 2: Real egg sample containing lasalocid of a concentration of 1,5 µg/kg, four transitions of lasalocid are shown

Validation Data

Method Validation According to DIN ISO 32645 and Council Directive 96/23 EC:

Method validation data for the coccidiostats determination were calculated following DIN ISO 32645 and Council Directive 96/23 EC.

The cc α and cc β values were evaluated by equidistant matrix sample calibration experiments. The application range for all shown coccidiostats in food and animal feed is between 1,0 and 20 µg/kg.

The recovery rates for the coccidiostats in this validation are in the range of 61 to 70 %.

Validation Data of Coccidiostats in Food and Feeding Stuff						
compound	cc α	cc β	LOD	IDL	precision	recovery
	[µg/kg]	[µg/kg]	[µg/kg]	[µg/kg]	VC [%]	[%]
feeding stuff						
Lasalocid	0,11	0,22	0,11	0,22	1,1	68
Narasin	0,22	0,44	0,22	0,44	1,9	61
Salinomycin	0,18	0,35	0,18	0,35	2,1	70
Monensin	0,19	0,38	0,19	0,38	2,0	69
egg						
Lasalocid	0,18	0,37	0,18	0,37	1,7	61
Narasin	0,18	0,36	0,18	0,36	1,6	67
Salinomycin	0,16	0,32	0,16	0,32	1,5	62
Monensin	0,08	0,16	0,08	0,16	0,7	70

LOD = limit of detection
IDL = identification limit
LOD, IDL according to DIN ISO 32645
VC [%] = variation coefficient
cc α , cc β according to Council Directive 96/23/EC

Conclusion

A highly sensitive and specific LC-MS-MS method for the quantitative determination of four coccidiostats (lasalocid, monensin, salinomycin and narasin) in food and feeding stuff has been developed and validated according to the Council Directive 96/23/EC and DIN ISO 32645. The Identification limit (IDL) for the coccidiostats are in the range of

0,16 to 0,44 µg/kg. Lowest concentrations of coccidiostats are detectable by using LC-MS-MS. GALAB Laboratories offers a highly sensitive method for the determination of coccidiostats in food and feeding stuff for a high product safety.