Confirmatory analysis of antibacterials residues in food of animal origin in Poland

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INTRODUCTION

The widespread and off-label use of antimicrobials in veterinary medicine may results in a risk of their residues in food of animal origin. The residues may pose a risk factor for public health. To consumer health protection a monitoring programmes for the control of the presence of these substances in food chain is defined and implemented in EU countries. In the national control programme of antibiotics and chemotherapeutics in Poland, the confirmatory analyses are performed by a multi-residue LC-MS/MS method. The method has been validated according to European Decision 2002/657/EC. The analytical determination was carried out in different biological material like muscle, kidney, milk and eggs. The confirmatory control of antibacterials include the ten groups of antibiotics and chemiotherapeutics.

MATERIALS AND METHODS

SAMPLE COLLECTION

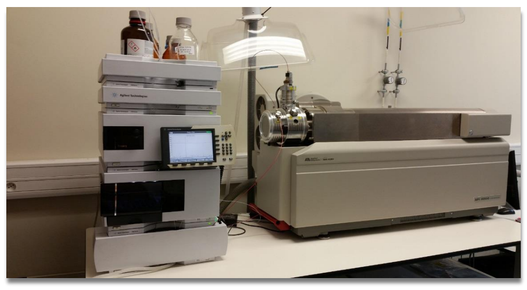
The samples were collected by district veterinary inspectors within the framework of official control, according to National Residues Control Plan. Samples were sent to the National Reference Laboratory (NRL) for drug residues or to one of the six regional laboratories approved to performed analyses. During a period of two years (2013-2014) a total of 14471 of bovine, porcine, poultry muscle and kidney samples, 3759 milk samples and 572 eggs samples were analysed for antibacterials. In muscle, kidney and milk two different sample preparation steps were used. The extraction of sulphonamides, fluoroquinolones, penicillins, cephalosporins, macrolides, diaminopyrimidines, pleuromutilins was carried out by acetonitrile, while for tetracyclines, aminoglicosides and lincosamides trichloracetic acid was used. In eggs one extraction for all groups of analytes was applied.

ANALYTICAL METHOD

Extraction and clean-up (muscle, kidney and milk)

2g biological material
6 ml 5% TCA
0.22 mm PVDF filters

2g biological material
8 ml ACN
evaporation N_2,
mobile phase dilution
0.22 mm PVDF filters



LC-MS/MS analysis

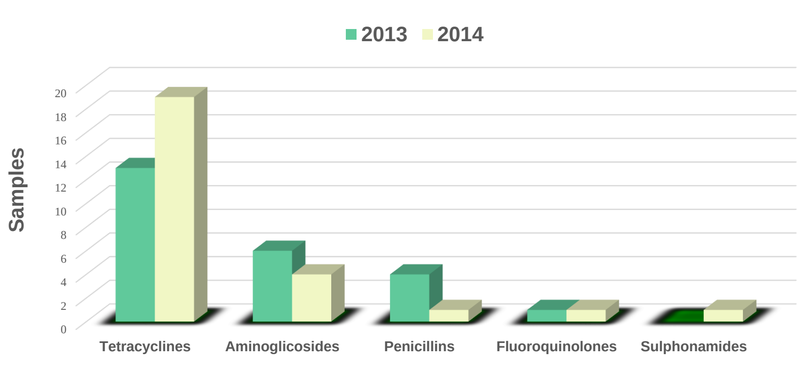
API 4000, ESI, MRM mode
Mobile phase: heptafluorobutyric acid, ACN
Column: Luna C18

Extraction and clean-up (eggs)

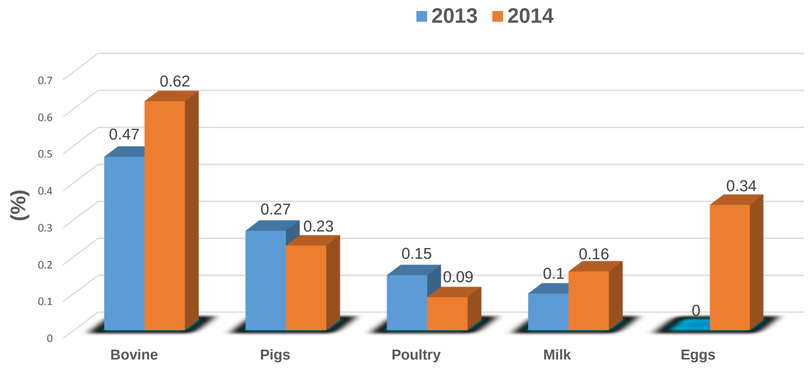
1 g biological material
1 ml oxalic acid pH 4.0
0.5 ml 0.1 M Na_2EDTA
8 ml ACN
Oasis HLB columns
evaporation N_2
mobile phase dilution
0.22 mm PVDF filters

RESULTS

The percentage of non-compliant samples for antibacterials in 2013 was 0.24% (9230 tested samples, targeted samples). The similar result in 2014 was observed, where only 0.25% of samples reported with violative residues (9572 tested samples, targeted samples). The number of tested samples in different species in 2013-2014 was as follows: 2938 in bovine, 7483 in porcine and 4050 in poultry. The highest percentages of non-compliant results in bovine were reported. Within the period of 2013 - 2014, the most common detected group of antibacterials were tetracyclines.



The groups of antibacterials confirmed during 2013-2014



The percentage of non-compliant samples reported in 2013 and 2014

CONCLUSIONS

The monitoring and the control of antibacterials in food ensure the safety of food supply. The use of official validated method is an important analytical tool to guaranteed a good level of consumer health protection. The two years of monitoring results (2013-2014) indicate the small percentage of non-compliant results in Poland.

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