Project Title: Method Development and Analysis of Ground Beef Samples for Residues

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Institution(s): Kansas Diagnostic and Analytical Services, Inc. DBA PharmCATS Bioanalytical Services

Completion Date: 2009

Background

This study originated from a desire to evaluate drug residue concentrations in ground beef products originating from different production systems.

Objectives

This study was designed to evaluate concentrations of 22 drugs in ground beef products originating from 8 sources representing 4 production types: conventional fed cattle production, market cows and bulls, USDA certified organic, and natural production systems.

Methodology

Target assay analytes

The drugs in the final report differ slightly from the initial proposal, but differences in method development and extraction were not sufficient to merit alterations in the budget or outcomes. For example, results are not reported for parent drugs where a metabolite is the target residue.

	Specific Compound	Tolerance in ng/g	
Aminoglycosides	Gentamincin	None	No tolerance in cattle
	Amikacin	None	No tolerance in cattle
	Neomycin	1200	Bovine muscle
Beta-lactams	Penicillin	50	Bovine edible tissues
	Ampicillin	10	Bovine edible tissues
	Desfuroylceftiofur	2000	Bovne muscle
Fluorquinolones	Danofloxacin	200	Bovine - muscle
	Ciprofloxacin	None	Tolerance in cattle liver - no muscle tolerance
Macrolides	Erythromycin	100	Bovine edible tissues
	Tylosin	200	Bovine muscle
	Tilmicosin	100	Bovine muscle
Phenicols	Florfenicol	300	Bovine muscle
Sulfas	Sulfamethazine	100	Bovine tissues
	Sulfadimethoxine	100	Bovine tissues
Tetracyclines	Oxytetracycline	2000	Bovine muscle (sum of all tetracycline residues)
	Chlortetracycline	2000	Bovine muscle (sum of all tetracycline residues)
	Tetracycline	2000	Bovine muscle (sum of all tetracycline residues)

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Streptogramins	Virginiamycin	None	Tolerance not required for cattle
NSAIDs	Flunixin Meglumine	25	Bovine muscle
	Phenylbutazone	None	No tolerance in cattle
Beta-agonists	Zilpaterol	None	cattle liver - no muscle tolerance
	Ractopamine	30	Bovine muscle

Sample collection
Four-hundred ground beef samples were collected from 8 processing plants representing conventional fed cattle production (2), conventional cull cows (2), organic production (2), and natural production systems with specifications of no drugs administered to the cattle (2).

The samples were collected at the plants by Mitch Bowling, Colorado State University, and shipped on dry ice or delivered to PharmCATS Bioanalytical Services, Manhattan, KS. The samples were stored at -80°C until analysis. Details of the collection procedures and types of production systems are included in Mr. Bowling's dissertation and publication.

Analytical methods
Tissue extraction and High Pressure Liquid Chromatography/Mass Spectrometry (HPLC/MS-MS) methods were developed in the PharmCATS Bioanalytical Services laboratory. Details for these methods are reported in the final dissertation and publication of Mitch Bowling. The publication is in process and will be submitted as an addendum to this report.

These methods are not the FDA-approved residue testing methods and the results should not be interpreted as representing regulatory determinations.

Five different extractions were necessary to accommodate analysis of all drugs. This means the 400 samples were submitted to 5 different extraction processes, for a total of 2000 extractions during the analysis period of the study. Within each extraction, there was also the possibility of the need for different runs of the processed sample with altered analytical conditions.

For each group of samples subjected to each of the 5 extractions (110 occurrences) there was an accompanying standard curve and quality control (QC) samples, for an addition of approximately 1300 additional extractions. Extensive extractions were also conducted during the method development phase of the project.

Findings

At the time of this report, the raw data have been turned over to a third party for analysis and inclusion in a dissertation and peer-reviewed publication. An amended report will include the results.