

Animal Drug Residues...Where do they reside?

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Abstract

Concerns with Animal Drug Residues have increased over the past several years. The FDA recently released the Veterinary Feed Directive (VFD) "to promote the judicious use of antimicrobials in food-producing animals". Veterinary drugs are utilized by producers for disease treatment, herd health management, growth promotion, and meat quality management. Several drug residues can remain in the animal and as a result enter into the food chain. A limited study was performed to determine where the drug residues were located and at what concentrations. Samples of urine, blood, muscle tissue, liver and fat from each animal were obtained and tested by LCMSMS. Samples were prepared and analyzed using a modified FSIS method. Data obtained contained 29 different antibiotics and growth promoters.

Introduction

Because of the recent emphasis on "antibiotic-free" meat, this investigation was performed. All of the beef cattle samples that were collected were considered to be antibiotic free. This study was performed for a few reasons:

- To see if any antibiotics were present
- If any were present, where they were most concentrated
- If they would appear in all of the different samples collected
- Would there be any correlation

Procedure

Samples of urine, blood, muscle tissue, fat and liver from each animal were collected.

All samples were analyzed by a modified FSIS LCMSMS method.

- Samples were ground if necessary
- Weighed and extracted with 80/20 Acetonitrile/water
- They were then purified with C18
- Defatted with Hexane
- The Acetonitrile layer was then evaporated
- Then reconstituted with 14/86 Acetonitrile/0.1% Formic Acid

Compound	Precursor (m/z)	Fragment 1/Fragment 2 (m/z)
Zilpaterol	262.233	244.1/185.1
Monensin	688.460	635.4/461.7
Lasalocid	572.600	237.2/337.3
MGA	397.432	337.3/279.2
Ractopamine	302.357	284.1/164.1
Trenbolone	271.338	199.0/115.1
Progesterone	315.395	97.1/109.1
Testosterone	289.353	97.1/109.1
Sulfadimethoxine	311.106	156.1/92.0
Erythromycin	734.383	158.3/83.1
Enrofloxacin	360.167	316.3/245.2
Ceftiofur	523.948	241.0/125.1
Oxytetracycline	461.161	426.4/443.4
Tilmicosin	869.533	88.2/174.3
Danofloxacin	358.149	340.2/82.2
Sulfamethazine	279.095	186.0/92.2
Tylosin	916.537	174.2/83.0
Chlortetracycline	479.125	444.5/462.4
Penicillin G	334.928	160.0/176.2
Florfenicol	375.148	340.1/241.0
Spectinomycin	333.131	98.2/140.2
Penicillin V	351.000	160.0/192.0
Amikacin	586.250	163.2/425.3
Zeranol	323.200	305.2/189.2
Gentamicin	478.353	322.3/157.2
Neomycin	615.347	161.2/293.0
Streptomycin	582.400	263.0/246.4
Bambermycin	613.134	377.2/595.2
Tulathromycin	806.436	577.4/158.0

Results

The following shows the percent of each antibiotic found in the various types of samples:

	Urine	Blood	Muscle Tissue	Liver	Fat
Bambermycin	--	--	--	10.5%	12.5%
Chlortetracycline	27.8%	--	--	5.3%	--
Danofloxacin	--	42.1%	18.8%	31.6%	18.8%
Gentamicin	--	--	--	21.1%	--
Neomycin	5.6%	--	--	5.3%	--
Oxytetracycline	44.4%	5.3%	--	5.3%	--
Penicillin V	5.6%	100.0%	--	--	--
Progesterone	--	10.5%	6.3%	--	6.3%
Spectinomycin	11.1%	--	--	100.0%	--
Sulfadimethoxine	5.6%	89.5%	12.5%	--	81.3%
Sulfamethazine	--	--	--	--	6.3%
Testosterone	--	--	18.8%	--	12.5%
Tilmicosin	--	--	--	94.7%	43.8%
Tulathromycin	--	100.0%	100.0%	100.0%	31.3%
Tylosin	--	21.1%	--	10.5%	100.0%

* Results in red are above reported maximum residue limit.

Urine Samples

	#1	#2	#3	#4	#6	#7	#8	#9	#11	#12	#12 with blood	#13	#14	#15	#16	#17	#18	#19
Chlortetracycline	340.7	2985.8	282.6	ND	60.3	ND	ND	ND	ND	ND	427.9	ND	ND	ND	ND	ND	ND	ND
Neomycin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	47.4	ND	ND	ND	ND	ND	ND
Oxytetracycline	9.8	ND	17.1	45.8	ND	ND	ND	ND	ND	ND	35.0	ND	32.9	20.2	23.6	ND	ND	37.9
Penicillin V	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	7.1	ND	ND	ND	ND
Spectinomycin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	119.1	281.2	ND	ND	ND	ND
Sulfadimethoxine	ND	ND	ND	ND	ND	ND	ND	2.8	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

Blood Samples

	#1	#2	#3	#4	#5	#6	#7	#8	#9	#10	#11	#12	#13	#14	#15	#16	#17	#18	#19
Danofloxacin	12.1	ND	7.7	ND	ND	ND	ND	7.2	5.7	ND	ND	7.2	ND	ND	ND	8.1	ND	5.7	6.6
Oxytetracycline	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	7.4	ND	ND	ND	ND	ND	ND	ND	ND
Penicillin V	6.8	11.8	19.9	49.9	32.4	50.1	15.4	28.8	41.5	35.5	9.4	68.0	18.2	26.0	12.9	13.9	19.8	42.6	36.8
Progesterone	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	8.2	ND	ND	ND	ND	8.7	ND	ND
Sulfadimethoxine	1.6	1.2	1.0	1.3	1.4	ND	2.1	1.4	1.8	1.0	1.0	1.1	1.2	1.7	2.3	ND	1.2	1.5	1.2
Tulathromycin	5.6	6.1	6.2	4.9	4.9	3.9	4.7	5.8	5.5	4.1	5.1	4.8	4.5	5.5	5.4	5.0	4.6	4.4	5.2
Tylosin	1.8	ND	1.1	ND	1.4	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.1	ND	ND

Muscle Tissue

	#1	#1	#2	#4	#8	#9	#10	#11	#12	#13	#14	#15	#16	#17	#18	#19
Danofloxacin	ND	ND	ND	ND	ND	ND	ND	ND	5.3	5.6	ND	ND	ND	ND	6.2	ND
Progesterone	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	60.9	ND	ND
Sulfadimethoxine	ND	1.5	ND	ND	ND	1.2	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Testosterone	12.8	13.5	ND	13.9	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tulathromycin	5.4	4.5	4.4	3.9	5.1	4.0	4.5	4.8	4.2	6.1	4.1	5.2	5.2	4.7	5.6	6.0

Liver Samples

	#1	#2	#3	#4	#5	#6	#8	#9	#10	#11	#12	#13	#14	#15	#16	#17	#18	#19
Bambermycin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	67.5	92.4	ND	ND	ND	ND
Chlortetracycline	ND	30.3	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Danofloxacin	ND	ND	ND	ND	26.1	ND	41.4	ND	37.2	31.9	32.5	ND	33.6	ND	ND	ND	ND	ND
Gentamicin	ND	ND	ND	194.1	ND	ND	ND	ND	ND	ND	ND	214.3	193.2	ND	ND	194.3	ND	ND
Neomycin	ND	ND	ND	ND	ND	ND	ND	ND	166.1	ND	ND	ND	ND	ND	ND	ND	ND	ND
Oxytetracycline	ND	ND	ND	ND	11.9	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Spectinomycin	281.6	642.7	330.2	419.9	562.1	143.2	481.0	313.8	525.3	250.9	467.9	1078.7	425.7	382.9	662.6	383.2	306.2	1081.1
Sulfadimethoxine	ND	1.4	ND	1.8	ND	1.7	ND	1.2	ND	1.7	ND	ND	1.0	ND	ND	1.2	1.5	ND
Tilmicosin	0.6	1.0	0.6	0.6	0.9	0.8	1.4	0.8	0.6	1.1	0.6	0.7	0.7	0.8	0.7	ND	0.4	0.9
Tulathromycin	26.8	30.3	26.8	25.0	23.2	30.3	22.2	32.5	38.3	22.1	23.8	29.1	27.6	26.9	21.4	22.7	22.6	20.9
Tylosin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.1	ND	1.7	ND	ND	ND	ND	ND

Fat Samples

	#1	#1	#2	#4	#8	#9	#10	#11	#12	#13	#14	#15	#16	#17	#18	#19
Bambermycin	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	102.3	283.3	ND	ND	ND	ND
Danofloxacin	34.3	ND	32.5	ND	ND	ND	32.3	ND	ND	ND	ND	ND	ND	ND	ND	ND
Progesterone	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	64.8	ND	ND
Sulfadimethoxine	1.2	1.9	ND	1.8	ND	2.4	1.9	2.5	2.2	2.9	1.6	1.2	1.0	1.6	1.2	ND
Sulfamethazine	ND	ND	1.6	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Testosterone	25.2	36.5	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tilmicosin	1.1	1.2	1.2	1.3	ND	ND	ND	1.0	ND	ND	1.5	1.1	ND	ND	ND	ND
Tulathromycin	18.5	15.5	12.5	19.1	ND	ND	ND	ND	ND	ND	21.8	ND	ND	ND	ND	ND
Tylosin	1.2	1.2	1.2	1.4	1.0	1.4	2.4	1.0	1.4	2.3	1.5	0.7	0.7	0.7	0.7	0.9

Conclusions

Antibiotics were present in low levels in urine, muscle tissue, liver and fat in cows that were presented as "antibiotic free". Analytical results for most compounds were below maximum residue levels, however samples were found in urine, liver and fat samples above the MRL's. This study was limited as samples were collected from only one basic geographic area. This study does indicate antibiotic ingestion from some source in these animals. Tissue determinations indicated the smallest number of positive results for the antibiotics tested. Possible additional study protocols would include both a larger geographical region and increased number of overall animals tested. Additionally for future studies inclusion of the kidneys, liver, and feces for each animal may also provide additional insight into the distribution of the ingested residues.