PREDECISIONAL

United States Department of Agriculture
Food Safety and Inspection Service

2009 – 2010: Analysis of Heavy Metals and Veterinary Drugs in 741 Catfish Samples from Retail Markets in the United States

ABSTRACT

The United States Department of Agriculture, Food Safety and Inspection Service (FSIS) tested 741 catfish samples collected to represent catfish consumption in the U.S. over the course of one year, July 1, 2009 through June 30, 2010. Samples of retail catfish were taken from retail markets in the U.S. and were tested for the presence of arsenic, cadmium, lead, mercury, chloramphenicol, gentian violet, malachite green, and nitrofurans (furazolidone and furaltadone).[1] Twenty samples (2.71%) had detectable heavy metal residues. Six samples (0.81%) had detectable residues of the unapproved veterinary drugs (i.e., those prohibited by the Food and Drug Administration) gentian violet and malachite green. None of the samples exceeded published violative levels for the heavy metals identified in this report. Veterinary drugs residues exceeded the regulatory zero tolerance for these compounds. The sample program was not designed to allow conclusions by country of origin of the catfish, the data is separated by whether or not the catfish was of domestic versus foreign origin.

[1] Nitrofurans are synthetic broad spectrum antibiotics with antibacterial and antiprotozoan properties. AOZ (3-amino-2-oxazolidinone and AMOZ (3-amino-5-morpholino-methyl-2-oxazolidinone) are metabolites of two nitrofuran parent drugs, furazolidone and furaltadone.

INTRODUCTION

The Food, Conservation, and Energy Act of 2008 (Public Law 110-246, section 11016 – known as the 2008 Farm Bill) amended the Federal Meat Inspection Act (FMIA) to include "catfish" as an amenable species subject to inspection by FSIS.

This report is the second in a series of catfish studies funded and conducted by FSIS to fill information gaps regarding chemical residues and veterinary drug residues associated with the consumption of catfish in the United States.

Presented are the results of analysis for arsenic, cadmium, lead, mercury, gentian violet, malachite green, nitrofurans (furazolidone (AOZ) and furaltadone (AMOZ)), and chloramphenicol.

High Priority Hazards Identified by FSIS

Veterinary Drugs

- Malachite green
- Gentian violet
- Nitrofurans

Environmental Contaminants - Inorganic Compounds

- Lead
- Arsenic
- Mercury
- Cadmium

METHODS

Sampling Plan

The USDA Agricultural Marketing Service (AMS), Pesticide Data Program (PDP) used the same sampling plan it previously had used for the collection of the 2008 to 2009 samples, except that 3 pounds of each sample were collected in this study, compared to 2 pounds in the previous study. Split samples were prepared by AMS' PDP sample collectors. One pound was sent to National Science Laboratory, Gastonia, North Carolina, and 2 pounds were sent directly to the FSIS Eastern Laboratory, Athens, Georgia. The AMS samples were collected via a stratified random sampling plan designed to represent catfish consumption in the United States. The results of the AMS study will be published by PDP in their annual report. The reader should contact PDP for additional information about this study.

Methods used by AMS are described in the PDP, Annual Summary, Calendar Year 2008, on page 11(see http://www.ams.usda.gov/pdp).

The applicable PDP Standard Operating Procedures (PDP SAMP PROC-01 through PDP SAMP PROC-04) may be accessed on the internet through the following link for more information on AMS sampling procedures:

- http://www.ams.usda.gov/AMSv1.0/ams.fetchTemplateData.do?template=TemplateO&topNav=&leftNav=ScienceandLaboratories&page=PDPSOPsforSamplingProcedures&description=PDP+Sampling+SOPs&acct=pestcddataprg

FSIS Analysis of Samples for 2009-2010

Samples were analyzed by AMS' National Science Laboratory, Gastonia, North Carolina, for pesticide residues and tested for arsenic, cadmium, lead, mercury, chloramphenicol, gentian violet, malachite green, and nitrofurans at the FSIS Eastern Laboratory, Athens, Georgia, and the FSIS Western Laboratory, Alameda, California. FSIS samples were analyzed from July 1, 2009 through June 30, 2010 using validated methods published in the Chemistry Laboratory Guidebook available through the FSIS website:

- http://www.fsis.usda.gov/Science/Chemistry_Lab_Guidebook/index.asp

FSIS Minimum Proficiency Levels

- ***arsenic, 200 ppb***
- ***lead, 25 ppb***
- ***cadmium, 10 ppb***
- ***mercury, 200 ppb***

The laboratory procedures for reporting the results of lead and cadmium in ppb and mercury and arsenic in ppm are based on established toxicity and tolerance levels reported for human and animal exposure studies. The sensitivity levels by which laboratory equipment are calibrated for these metals are also based on the toxicity levels that impact public health.

The laboratory procedures for reporting of malachite green and gentian violet at the ppb level followed a two tiered analysis process. An enzyme-linked immunosorbent assay (ELISA) method was used to rapidly screen catfish samples for malachite green and gentian violet concentrations down to 1 ppb. Samples that exhibited a positive ELISA response were then sent on for a confirmatory analysis using a liquid chromatography tandem mass detection (LC-MS-MS) system. Due to the design of ELISA analysis method, any presumptive positive finding could be the result of any individual dye, its metabolite, or the sum of all dye components in reduced presence. In contrast, the confirmatory method using LC-MS-MS is a more expensive and laborious analysis to run, but has the ability to distinguish between malachite green, gentian violet and their metabolites at 5-10 times the sensitivity of the ELISA method. Since the presence of malachite green, gentian violet or metabolites at any level still constitutes the use of the product, all reported positives for gentian violet or malachite green will include both the parent compound and any metabolites detected at or below 1 ppb.

Analyses of samples for nitrofurans, furazolidone (AOZ) and furaltadone (AMOZ) were conducted using an LC-MS-MS system setup to do simultaneous screening and confirmation in a single run.

Heavy Metals

The Food and Drug Administration action level in fish (edible portion only) for methyl mercury is 1000 ppb. The term *action level* refers to levels recommended by the FDA when pesticide residues occur in food or feed commodities for reasons other than the direct application of the pesticide. Mercury occurs naturally in the environment and can also be released into the air through industrial pollution. Mercury falls from the air and can accumulate in streams and oceans and is turned into methylmercury in the water.

The U.S. does not have published regulatory levels for arsenic, cadmium, and lead.

The Codex Alimentarius Commission has adopted a Codex maximum level of 300 ppb for lead and 500 ppb for mercury.

Veterinary Drugs

The following drugs, families of drugs, and substances are prohibited for extralabel animal drug uses in food producing animals:

- Chloramphenicol
- Nitrofurazone

Malachite green and gentian violet are not approved for any food producing animals.

RESULTS

Seven hundred and forty one sub-samples were received at FSIS laboratories from samples collected by AMS during the period July 2009 to June 2010. Five hundred and fifty-six were domestic samples, 179 were imported, and 6 were unlabeled regarding country of origin (see Table 1).

There were 15 domestic samples (2.17%) that had detectable residues of lead and cadmium and 11 imported samples (2.79%) that had detectable residues of lead. The concentrations of these metals in the fish samples did not exceed regulatory levels or the Codex MRL. Mercury was not detected in either domestic or imported catfish samples (see Table 2).

Seven hundred and twenty-seven samples of the 741 subsamples were analyzed for unapproved veterinary drugs. Five samples from domestic products and 1 imported product had detectable residues of unapproved drug residues. Of the 545 domestic samples tested, 5 (0.92%) contained gentian violet. One imported sample of the 176 imported samples tested (0.57%) contained malachite green (see Tables 3&4).

Table 1 - Origin of Catfish Samples, Number of Samples Analyzed, Percent Sampled, and Percent of Detectable Residues

Origin of Sample		Number of Samples	Percent Sampled	Samples with Detectable Residues of Heavy Metals	Percent of Detectable Residues
Domestic	United States	556	75.03%	15	2.71%
Import		179	24.16%	5	2.79%
Unknown	Unknown	6	0.81%	0	0
Total		**741**	**100.00%**	**20**	2.71%

Table 1 presents the distribution of samples by origin and the number with detectable residues of heavy metals. There were 20 domestic and 5 imported samples with detectable residues of heavy metals, respectively. A majority of the catfish samples, 556, of 741 (75.03%) was from the United States. Imported samples, 179, constituted 24.16%.

Table 2 – Number of Catfish Samples by Origin, Percent of Detectable Residues, and Range of Heavy Metals Analyzed

Origin of Sample	Heavy Metal Type	Number of Samples	Samples with Detectable Residues	Percent of Detectable Residues	Range
Domestic	Arsenic, (ppb)	556	0	0.00%	N/A
	Cadmium (ppb)	556	10	1.80%	10.00 - 27.55
	Lead, ppb	556	5	0.90%	26.02 - 68.93
	Mercury (ppb)	199	0	0.00%	N/A
Import	Arsenic (ppb)	179	0	0.00%	N/A
	Cadmium (ppb)	179	0	0.00%	N/A
	Lead (ppb)	179	5	2.79%	29.07 - 173.36
	Mercury (ppb)	66	0	0.00%	N/A
Unknown	Arsenic (ppb)	6	0	0.00%	N/A
	Cadmium (ppb)	6	0	0.00%	N/A
	Lead (ppb)	6	0	0.00%	N/A
	Mercury (ppb)	4	0	0.00%	N/A
All	Arsenic (ppb)	741	0	0.00%	N/A
	Cadmium (ppb)	741	10	1.35%	10.00 - 27.55
	Lead (ppb)	741	10	1.35%	26.02 - 173.36
	Mercury (ppb)	269	0	0.00%	N/A

Table 2 presents the origin of samples collected, the number of samples, the number of samples with detectable residues, and the range of heavy metals analyzed. Twenty detectable residues of heavy metals were found in domestic and imported samples. Ten domestic samples had detectable residues for cadmium and five samples for lead. The range for lead was 26.02 ppb to 68.93 ppb while the range for cadmium was 10.00 ppb to 27.55 ppb. Five imported samples had detectable residues for lead. The range for lead was 29.07 – 173.36 ppb.

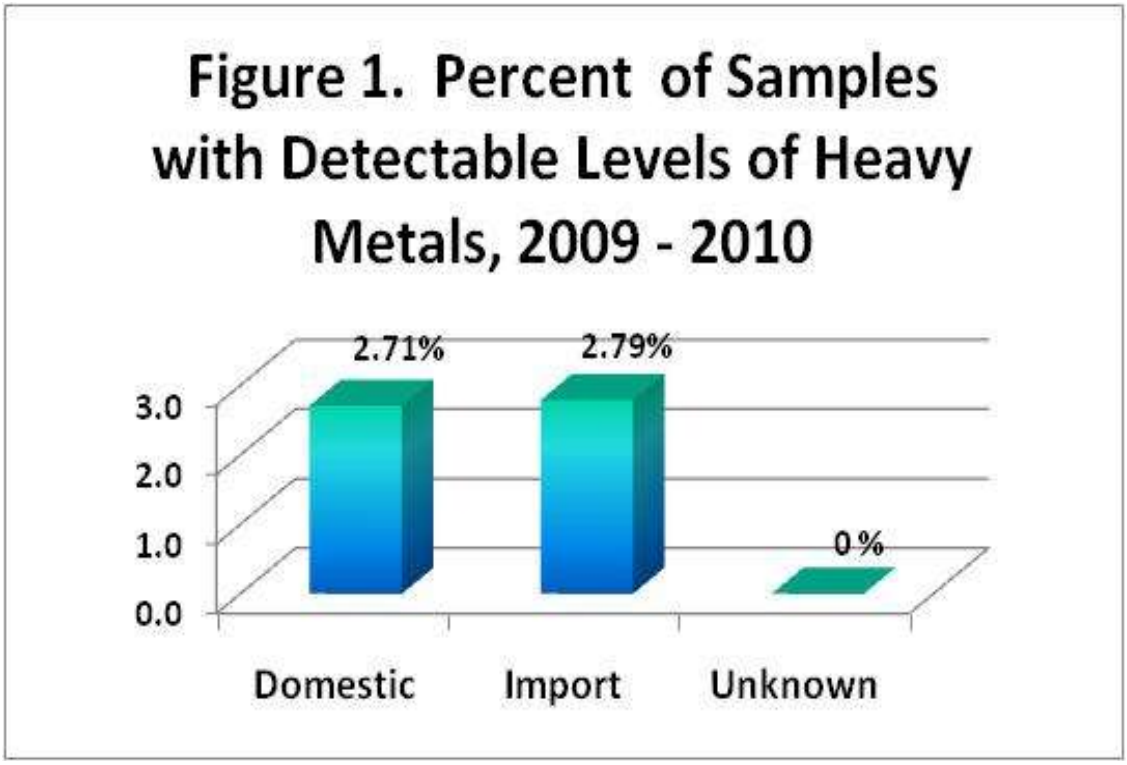

Figure 1 presents the percentage of samples with detectable residues of heavy metals. Fifteen domestic (2.71%) and 5 imported (2.79) samples had detectable residues for lead.

Table 3 – Number of Samples, Percent Sampled, Samples with Detectable Residues, and Number of Detectable Residues of Veterinary Drugs by Origin of Samples

Origin of Sample		Number of Samples Analyzed	Percent Sampled	Samples with Residues for Veterinary Drugs
Domestic	United States	545	74.96%	5
Import		176	24.21%	1
Unknown	Unknown	6	0.83%	0
Total		727	100.00%	6

Table 3 presents the completed confirmation test results for veterinary drug residues. There were 6 detectable residues. gentian violet, from 5 domestic samples, and malachite green from 1 imported sample.

Table 4 – Number of Samples, Percent Sampled, and Detectable Residues of Veterinary Drugs by Origin of Samples

Origin of Sample	Number and Type of Detectable Residues of Veterinary Drugs by Origin					
	Furaltadone (AMOZ)	Furazolidone (AOZ)	Chloram-phenicol	Gentian Violet	Malachite Green	Total Detectable Residues
Domestic						
No. of Samples	545	545	464	545	545	
Samples with Detectable residues	0	0	0	5	0	5
Percent Positive	0.00%	0.00%	0.00%	0.92%	0.00%	
Import						
No. of Samples	177	177	153	176	176	
Samples with Detectable residues	0	0	0	0	1	1
Percent Positive	0.00%	0.00%	0.00%	0.00%	0.57%	
Unknown						
No. of Samples	6	6	5	6	6	
Samples with Detectable residues	0	0	0	0	0	0
Percent Positive	0.00%	0.00%	0.00%	0.00%	0.00%	
ALL						
No. of Samples	728	728	622	727	727	
Samples with Detectable residues	0	0	0	5	1	6
Percent Positive	0.00%	0.00%	0.00%	0.69%	0.14%	

Table 4 presents the completed 2009-2010 number and pecentage of domestic, imported, and unlabled confirmation test results for veterinary drug residues, gentian violet, malachite green. Five domestic samples (0.92%) had detectable residues of gentian violet and 1 imported sample (0.57%) was found with malachite green residue.

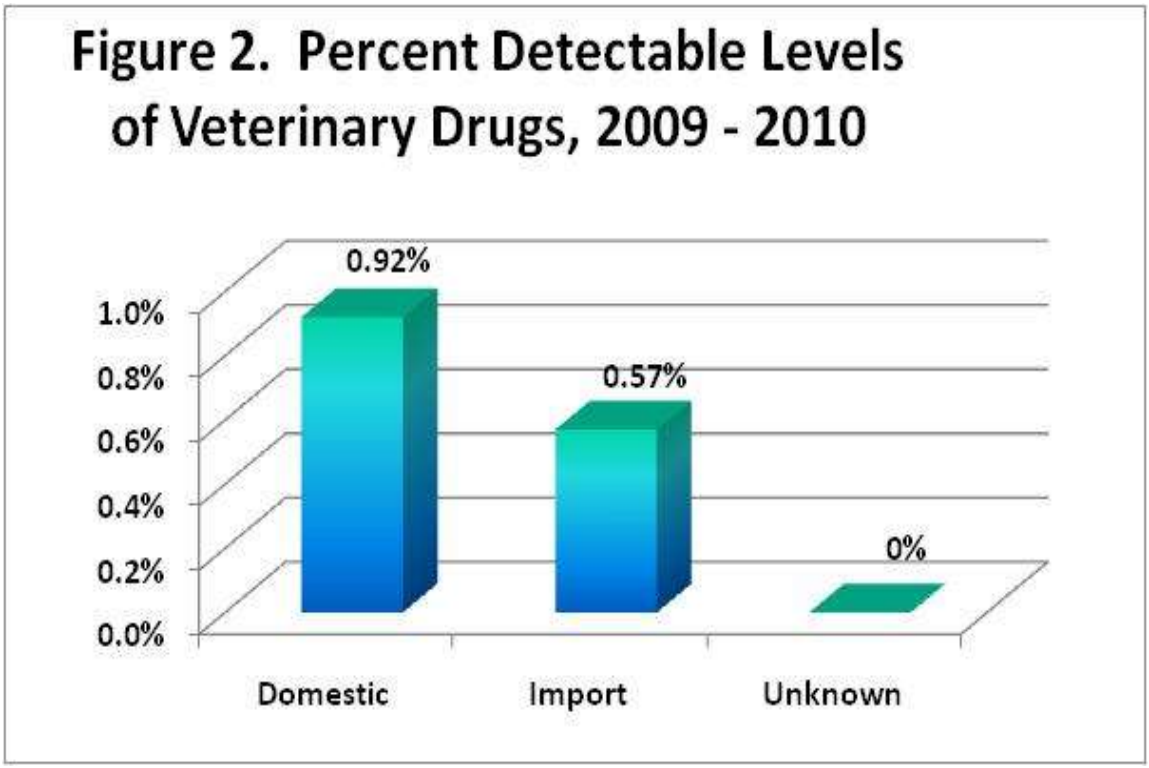

Figure 2 presents the percentage of detectable residues of veterinary drugs in domestic, imported, and unknown samples. There were 5 detectable residues for gentian violet out of 545 domestic samples (0.92%) and 1 detectable level for malachite green out of 176 imported samples (0.57%). None of the unknown samples had detectable residues of veterinary drugs.

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