United States Department of Agriculture
Food Safety and Inspection Service

2008 – 2009: Analysis of Heavy Metals and Veterinary Drugs in 737 Catfish Samples from Retail Markets in the United States

ABSTRACT

The United States Department of Agriculture's Food Safety and Inspection Service (FSIS) tested 737 catfish samples collected to represent catfish consumption in the U.S. over the course of one year, April 2008 through March 2009. Samples of seafood labeled as catfish were taken from retail markets throughout the U.S. and were tested for the presence of arsenic, cadmium, lead, mercury, chloramphenicol, gentian violet, malachite green, and nitrofurans[1]. Seventeen of the 737 samples (2.31%) had detectable heavy metal residues. There were seven samples (0.96%) with detectable residues of the unapproved veterinary drugs (i.e., those prohibited by the Food and Drug Administration), gentian violet, malachite green, and nitrofurans (furazolidone and furaltadone). None of the samples exceeded published regulatory residues 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 first in a series of catfish studies funded and conducted by FSIS to fill information gaps regarding chemical residues and foodborne pathogens 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) collected catfish samples from April 2008 through March 2009 at the request of the Environmental Protection Agency (EPA). The AMS samples were collected via a stratified random sampling plan[2] designed to represent catfish consumption in the United States. A detailed summary of the sampling method employed by AMS are described in the Pesticide Data Program (PDP), Annual Summary, Calendar Year 2008, 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

[2] The rationale behind the PDP sampling methodology is explained in the Journal of Official Statistics, Vol. 13, No. 4, 1997, pp 367-383

FSIS Analysis of Samples 2008-2009

AMS's PDP samples collected between April 2008 through March 2009 were tested by AMS for 238 pesticides. The remaining samples were prepared by AMS' National Science Laboratory, Gastonia, North Carolina, and sent to the FSIS Eastern Laboratory, Athens, Georgia, and the FSIS Western Laboratory, Alameda, California so they could be tested for arsenic, cadmium, lead, mercury, chloramphenicol, gentian violet, malachite green, and nitrofurans (furazolidone (AOZ) and furaltadone (AMOZ)). Samples were analyzed from July 2008 to June 2009 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 in 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.

RESULTS

Seven hundred and thirty-seven subsamples were received at FSIS laboratories from samples collected by AMS during the period, April 2008 through March 2009. Five hundred and seventy three were domestic samples, 151 were from foreign countries and 13 were unlabeled regarding country of origin (see Table I).

There were 6 domestic samples (1.05%) that had detectable residues of lead and cadmium and 11 imported samples (7.29%) that had detectable residues of lead and arsenic. 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 Tables 2 & 3).

Seven hundred and thirty samples of the 737 subsamples were analyzed for unapproved veterinary drugs. Seven samples from domestic and imported products had 10 detectable residues of unapproved drugs (1.36%). Of the 421, domestic samples tested, 4 contained gentian violet (0.95%) and 1 contained malachite green (0.24%). Whereas, of the 99 imported samples tested, 2 (2.02%), 1(1.01%), 1(1.01%) and 1(1.01%) contained gentian violet, malachite green, furazolidone (AOZ) and furaltadone (AMOZ), respectively (see Tables 4 & 5).

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.

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

Origin of Sample		Number of Samples Analyzed	Percent Sampled	Samples With Detectable Residues for Heavy Metals	Heavy Metal Detectable Residues	Percent of Samples With Detectable Residues
Domestic	United States	573	77.75%	6	6	1.05%
Import		151	20.49%	11	12	7.29%
Unknown	Unknown	13	1.76%	0	0	0%
Total		**737**	**100.00%**	**17**	**18**	**2.31%**

Table 1 presents the distribution of samples by origin and the number with detectable residues of heavy metals. There were 6 domestic and 11 imported samples with detectable residues of heavy metals, respectively. A majority of the catfish samples, 573, out of a total of 737 (77.70%) were from the United States. Imported samples, 151, constituted 20.49%.

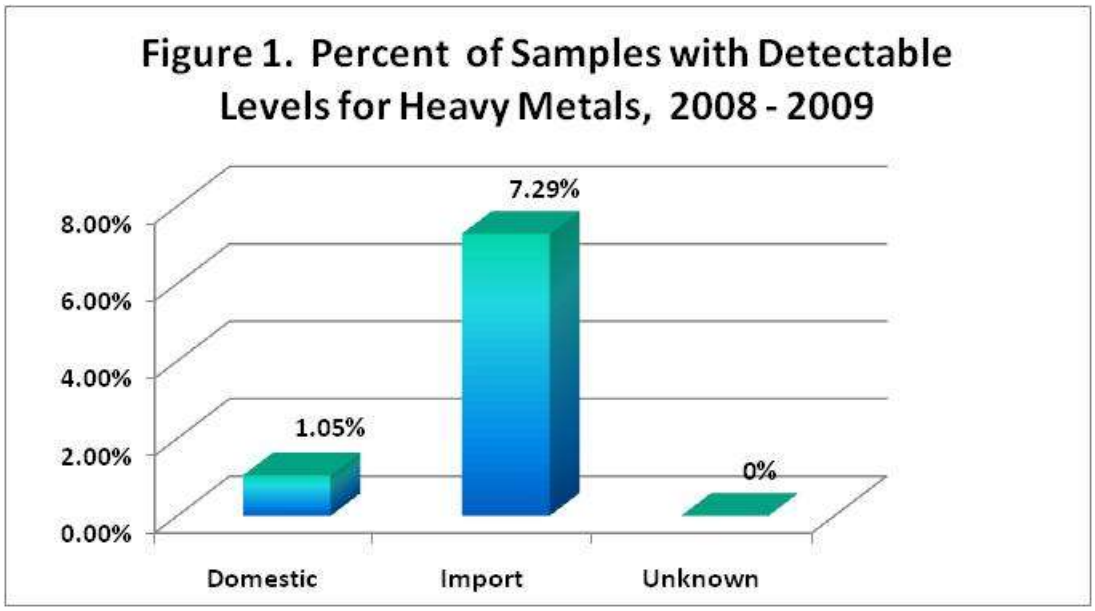

Figure 1 presents the percentage of samples with detectable residues for heavy metals. There were 6 out of 573 domestic samples (1.05%) and 11 out of 151 imported samples (7.29%) with detectable residues of heavy metals.

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 Analyzed	Samples with Detectable Residues	Percent of Detectable Residues	Range
Domestic	Arsenic (ppb)	573	0	0.00%	0
	Cadmium (ppb)	572	2	0.35%	10.09-13.11
	Lead (ppb)	572	4	0.70%	29.49-76.92
	Mercury (ppb)	573	0	0.00%	0
Import	Arsenic (ppb)	149	2	1.34%	1030-1640
	Cadmium (ppb)	151	0	0.00%	0
	Lead (ppb)	151	10	6.62%	27.96-103.24
	Mercury (ppb)	151	0	0.00%	0
Unknown	Arsenic (ppb)	13	0	0.00%	0
	Cadmium (ppb)	13	0	0.00%	0
	Lead (ppb)	13	0	0.00%	0
	Mercury (ppb)	13	0	0.00%	0
All	Arsenic (ppb)	735	2	0.27%	1030-1640
	Cadmium (ppb)	736	2	0.27%	10.09-13.11
	Lead (ppb)	736	14	1.90%	27.96-103.24
	Mercury (ppb)	737	0	0.00%	0

Table 2 presents the origin of samples collected, the type and the number of samples analyzed, the number of samples with detectable residues, and the range of heavy metals. Eighteen detectable residues of heavy metals were found in domestic and imported samples. Four domestic samples had detectable residues for lead and 2 samples for cadmium. The range for lead was 29.49 ppb to 76.92 ppb while the range for cadmium was 10.90 ppb to 13.11 ppb.

Twelve imported samples had detectable residues for lead and arsenic. Ten samples had detectable residues for lead and 2 for arsenic. The range for lead was 27.96 ppb to 103.24 ppb while the range for arsenic was 1030 ppb to 1640 ppb.

Table 3 – Origin of Samples, and Number of Detectable Residues of Heavy Metals

Origin of Sample	Number of Detectable Residues of Heavy Metals			
	Arsenic (ppb)	Cadmium (ppb)	Lead (ppb)	Mercury (ppb)
Domestic (United States)	0	2	4	0
Import	2	0	10	0
Unknown	0	0	0	0
Total	2	2	14	0

Table 3 presents the samples with detectable residues for heavy metals by origin for domestic and imported catfish samples. There were a total of 18 detectable residues of arsenic, lead and cadmium.

Table 4 – 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 Detectable Residues for Veterinary Drugs	Number of Detectable Residues for Veterinary Drugs
Domestic	United States	573	78.50%	4	5
Import		145	19.86%	3	5
Unknown	Unknown	12	1.64%	0	0
Total		730	100.00%	7	10

Table 4 presents the completed confirmation test results for veterinary drug residues. There were 10 detectable residues of nitrofurans (furazolidone (AOZ) and furaltadone (AMOZ)), malachite green and gentian violet, 5 from domestic samples, and 5 from imported samples.

Table 5 – Number, Type, and Percentage of Detectable Residues of Veterinary Drugs from Domestic, Imported, and Unknown Samples

Origin of Sample	Number and Type of Detectable Residues of Veterinary Drugs by Origin					
	Furaltadone (AMOZ) ppb	Furazolidone (AOZ) ppb	Chloram-phenicol ppb	Gentian Violet ppb	Malachite Green ppb	Total Detectable Residues
Domestic						
No. of Samples	420	420	573	421	421	
Samples with Detectable residues	0	0	0	4	1	5
Percent Positive	0.00%	0.00%	0.00%	0.95%	0.24%	
Import						
No. of Samples	99	99	145	99	99	
Samples with Detectable residues	1	1	0	2	1	5
Percent Positive	1.01%	1.01%	0.00%	2.02%	1.01%	
Unknown						
No. of Samples	8	8	12	8	8	
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	527	527	730	528	528	
Samples with Detectable residues	1	1	0	6	2	10
Percent Positive	0.19%	0.19%	0.00%	1.14%	0.38%	

Table 5 presents the number and percentage of domestic, imported, and unlabeled samples with detectable residues for the veterinary drugs, gentian violet, malachite green, furazolidone (AOZ), and furaltadone (AMOZ). There were a total of 10 detectable residues of veterinary drugs. Five domestic samples (1.19%) had detectable residues, 4 for gentian violet, and 1 for malachite green. Five imported samples (5.05%) had detectable residues for veterinary drug, 1 each for AOZ and AMOZ, 2 for gentian violet, and 1 for malachite green.

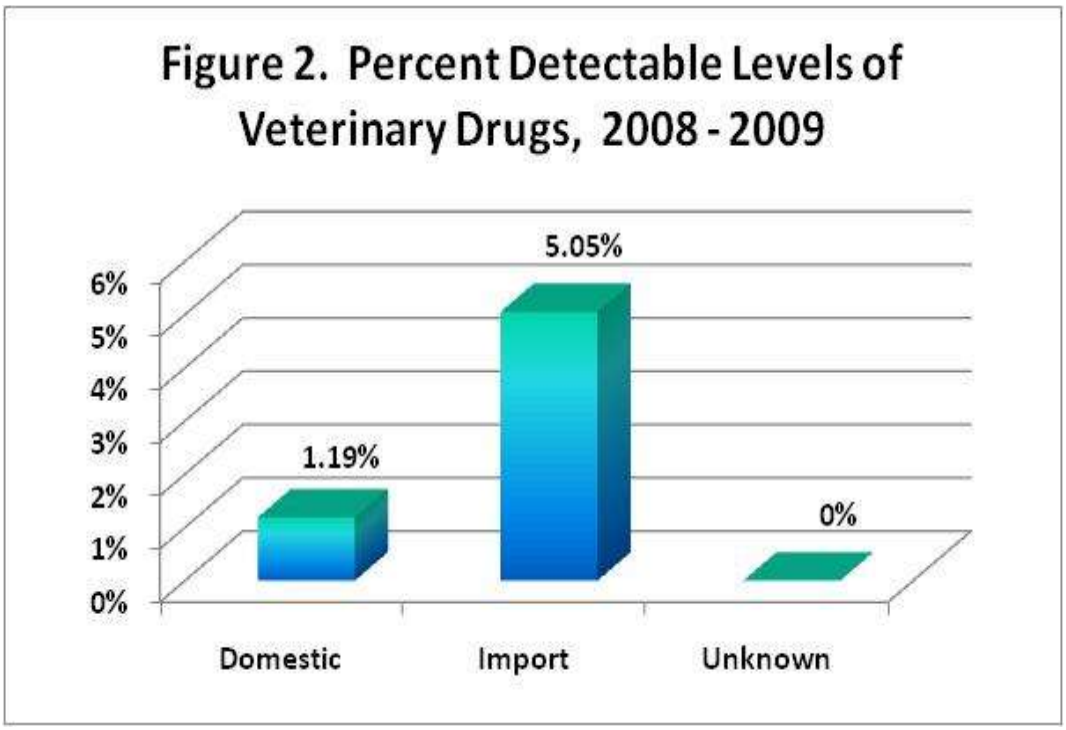

Figure 2 presents the percent detectable residues of veterinary drugs for domestic, imported, and unknown samples. There were 5 detectable residues out of 421 samples (1.19%) for gentian violet and malachite green, and 5 detectable residues out of 99 imported samples (5.05%) for the nitrofurans (AMOZ and AOZ), gentian violet and malachite green. None of the unknown samples had detectable residues of veterinary drugs.

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