

 **USDA**
United States Department of Agriculture

One Team, One Purpose

Food Safety and Inspection Service
Protecting Public Health and Preventing Foodborne Illness

**US Department of Agriculture
Food Safety and Inspection Service**

**National Residue Program:
Lab Methodology and Results**

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US Department of Agriculture Food Safety and Inspection Service

- I. Background
- II. FSIS Chemistry Methods (primary screens)
 - Pesticides
 - Veterinary Drugs
 - Heavy Metals
- III. Preliminary Test Results – Domestic Scheduled Samples
- IV. Preliminary Test Results – Inspector Generated Samples (In-Plant Screening and Lab Analysis)
- V. Preliminary Test Results – Import Samples
- VI. Where can I find more information?

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Food Safety and Inspection Service:

I. Background

National Residue Program (NRP): Goals/Purpose

- Provide a structured process for identifying and evaluating chemical compounds of concern in food animals.
- Test for the presence of chemical compounds: veterinary drugs, pesticides, hormones, and environmental contaminants in meat, poultry, and egg products.
- Identify need for regulatory follow-up when violative levels of chemicals residues are found.

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Food Safety and Inspection Service:

I. Background

National Residue Program (NRP): Process

- FSIS inspectors collect samples of meat, poultry, and *Siluriformes* (catfish) muscle tissue as well as egg products at federally inspected establishments.
- Samples are analyzed at FSIS laboratories for chemical residues of veterinary drugs, pesticides, and environmental contaminants.
- Laboratory confirmed results are compared with tolerances established by the FDA and the EPA, to prevent adulterated products from entering into commerce.

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Food Safety and Inspection Service:

I. Background

Laboratory System



Western Laboratory
(Albany, CA)

Midwestern Laboratory
(St. Louis, MO)

Eastern Laboratory and
LQAS
(Athens, GA)

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Food Safety and Inspection Service:

I. Background

Analytical Chemistry Capabilities of the Laboratories

Midwestern Laboratory

Inspector-generated residue analysis, primarily veterinary drugs

Western Laboratory and Eastern Laboratory

Scheduled chemistry analysis as described in the National Residue Program (NRP)

EL – Metals; food chemistry

WL – Pesticides

All three labs are ISO 17025 and ALACC accredited

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Food Safety and Inspection Service:

II. Chemistry Methods

Multi-Residue Screens to Support the NRP

Pesticides

- Based on QuEChERS extraction developed at USDA ARS
- Muscle, not fat, of various animal species
- Uses UPLC-MS-MS

Multi-Class Residue Method (MRM)

- Based on QuEChERS extraction
- Muscle, kidney of various animal species
- Uses UPLC-MS-MS

Central framework to add analytes, matrices



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Food Safety and Inspection Service:

II. Chemistry Methods

Advantages --- Updated Screens

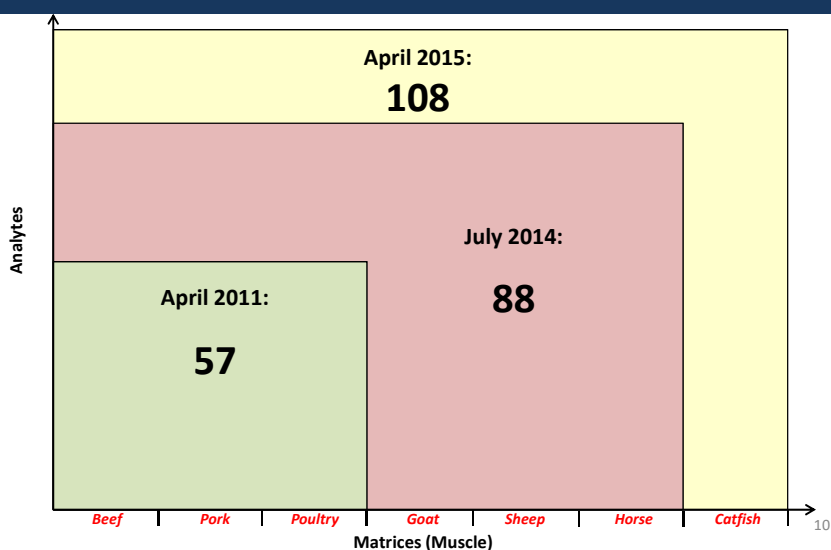
- Selective (UPLC-MS-MS; ICP-MS)
- Efficient (labs can maintain high throughput)
- Sensitive (achieve lower levels of detection)
- Fit for Purpose:
 - Validated using FSIS lab system procedures that conform with the ISO 17025 Standard
 - LQAS reviews and approves methods based on FSIS-generated validation data

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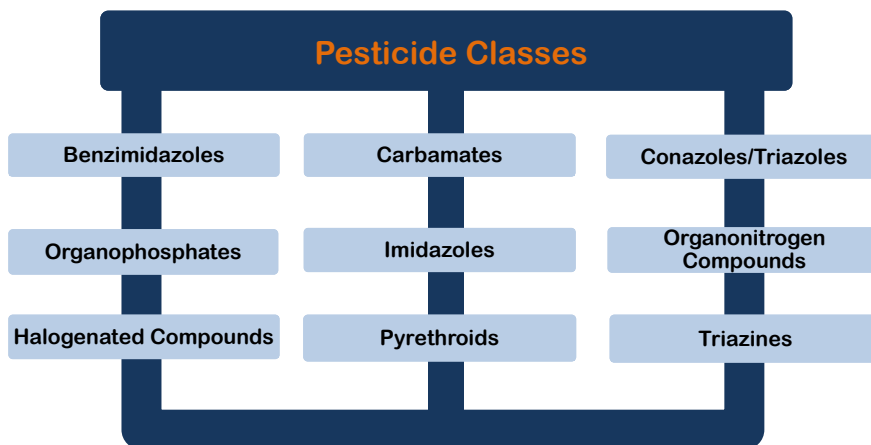
Food Safety and Inspection Service:

II. Chemistry Methods

Evolution of Pesticides Screen Capability

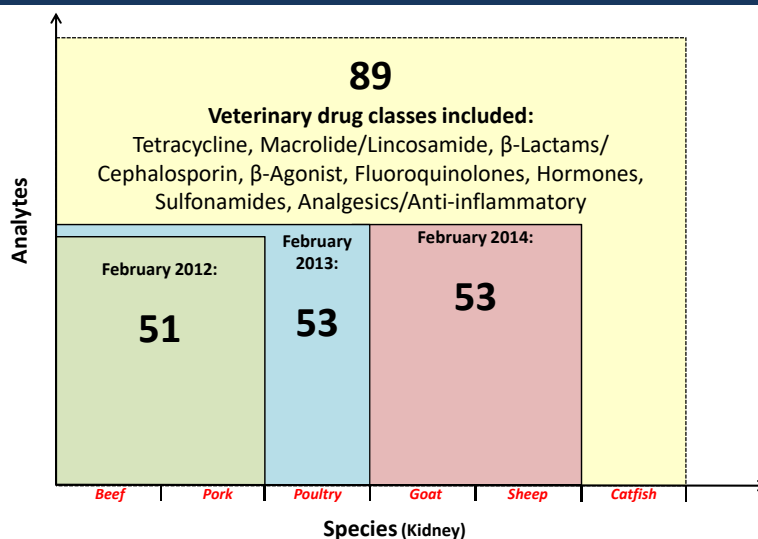


Food Safety and Inspection Service:
 II. Chemistry Methods
 Pesticides Method



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Food Safety and Inspection Service:
 II. Chemistry Methods
 Evolution of Multi-Class Residue Screen Capability



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Food Safety and Inspection Service:

II. Chemistry Methods

Metals Screen Capability

Metals	Matrices
2 (Pb, Cd)	Muscle, Liver, Kidney of Beef, Pork, Poultry; Processed Products; Catfish Muscle
15 (Se, Mn, Mo, Tl, Co, Fe, Zn, Cu, Ni, Al, B, Ba, Cr, V, Sr)	Processed Products, Muscle of Beef, Pork, Poultry



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Food Safety and Inspection Service:

III. Preliminary Test Results – Domestic Scheduled Samples

Year	Number of Samples	FSIS Lab Total Chemical Analyses	# of Lab confirmed violative Carcasses	# of Lab confirmed Non-violative Positive Analyses	# of Distinct Violative Chemical Residues
* FY 2013	4,583	426,386	15	23	8
FY 2014	6,066	576,140	10	34	10
FY 2015 (Preliminary)	6,444	578,837	13	22	9
FY 2016 Oct 2015-Mar 2016 (preliminary)	3,498	421,606	7	14	9

Source: FSIS Data Warehouse (DW) & Public Health Information System (PHIS)

* Results from Jan 2013 to Sept 2013;

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Food Safety and Inspection Service:
**III. Preliminary Test Results – Domestic Scheduled Samples
 Animal Class vs. Chemical Method (FY 2015)**

Animal Class	Chemical Method								
	Amino-glycosides	Arsenic	Avermectins	Beta-Agonists	Carbadox	Hormones	MRM	Metals	Pesticides
Beef Cows	749	371	372	377	-	328	750	121	266
Bob Veal	485	263	263	222	-	229	486	117	146
Dairy Cows	732	376	378	354	-	327	733	116	248
Goats	141	142	142	-	-	-	240	-	98
Heifers	475	258	262	214	-	230	476	94	143
Market Swine	802	416	417	385	384	-	805	134	298
Mature Turkeys	4	4	-	-	-	-	4	27	-
Sheep	144	143	143	-	-	-	286	-	138
Sows	753	394	396	2	-	-	755	130	255
Steers	476	262	264	214	-	222	480	102	145
Young Chickens	697	363	-	-	-	-	701	116	241
Young Turkeys	705	349	-	-	-	-	704	121	254
Beef Cows	6,163	3,341	2,637	1,769	384	1,336	6,420	1,078	2,232

Food Safety and Inspection Service:
**IV. Preliminary Test Results – Inspector Generated Samples
 In-Plant Screening**

From FSIS Directive 10,800.1:

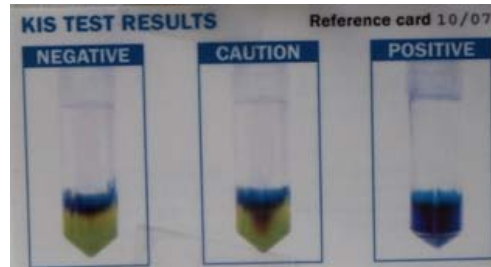
II. INSPECTOR-GENERATED SAMPLING

A. In-Plant Screening Test (KIS™ Test)

Under the direction of the Public Health Veterinarian (PHV), In-Plant Personnel (IPP) are to conduct a KIS™ test on any carcass that, based on herd history or ante-mortem or post-mortem inspection findings, may contain a violative drug residue.

Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples
In-Plant Screening

- KIS™ = Kidney Inhibition Swab
- Score kidney, collect juice using swab
- Incubate in bacteria/agar solution
- If bacteria produce acid, no antimicrobial drugs are present, and the test is yellow (negative)
- If bacteria are inhibited due to antimicrobial drugs, the color remains purple (positive)



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Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples

In-Plant and Lab Results

	In-Plant Result	Violative	Not Detected	Non-Violative	Discarded
FY2015	Negative	4	135	3	0
	Positive	788	2515	588	22
	TOTAL	792	2650	591	22
First Half of FY2016	In-Plant Result	Violative	Not Detected	Non-Violative	Discarded
	Negative	2	13	1	0
	Positive	391	1293	234	7
	TOTAL	393	1306	235	7

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Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples

**Number of violative samples and violative results,
 FY 2015 and First Half of FY 2016**

	FY 2015	First Half of FY 2016
Number of violative samples	792	393
Number of violative results	907	447
Number of samples containing multiple violative compounds	92	48

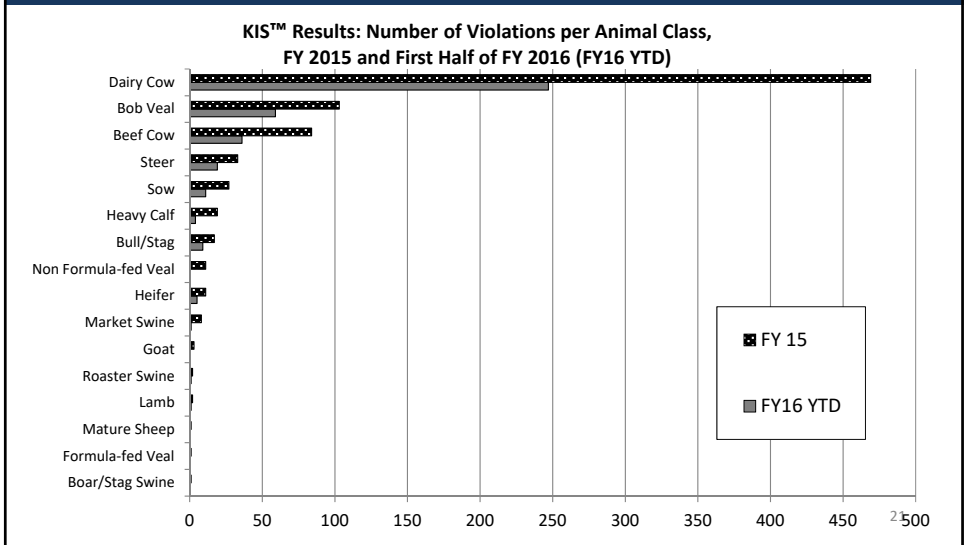
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Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples

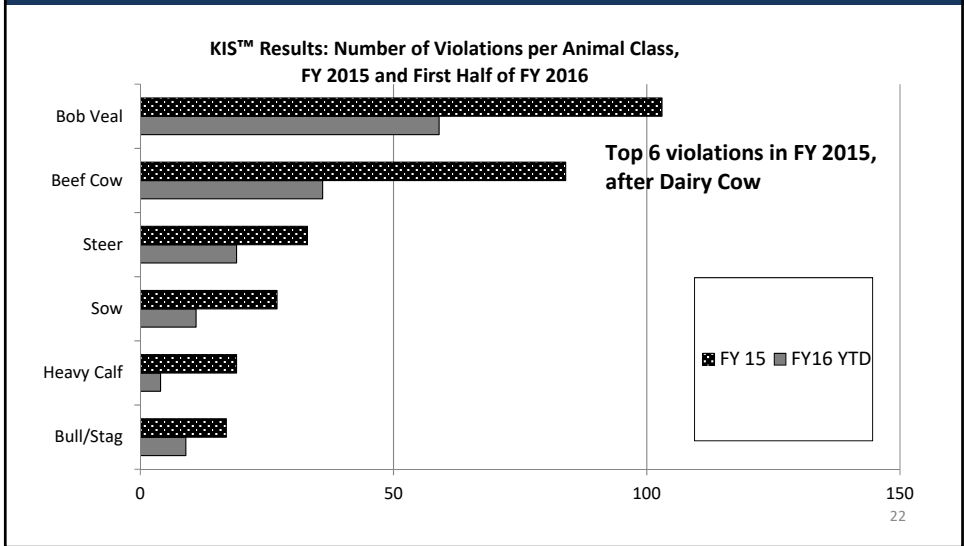
- FY 2015:
 - **Dairy cows** and **bob veal** accounted for **72%** of KIS™ test violations.
 - **16** livestock production classes were represented (Figures 5 and 6).
- First Half of FY 2016:
 - **Dairy cows** and **bob veal** account for **78%** of KIS™ test violations.
 - **15** livestock production classes are represented (no Boar/Stag Swine yet).

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Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples



Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples



Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples

**Number of Samples with a Given Number of Distinct Compounds
 FY 2015 and First Half of FY 2016**

# of Distinct Compounds in Sample	Number of Samples	
	FY 2015	First Half of FY 2016
1	700	345
2	76	42
3	12	6
4	2	0
5	1	0
6	1	0

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Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples

FY 2015: Two Samples Contained 4 Violative Compounds

Bob Veal	Steer
Ciprofloxacin (kidney)	Cefazolin (kidney)
Desethylene ciprofloxacin (liver)	Sulfamethazine (liver)
Enrofloxacin (kidney)	Sulfamethoxypridazine (kidney)
Flunixin (kidney)	Tilmicosin (liver)
Tulathromycin (kidney; non-violative)	Oxytetracycline (kidney; non-violative)

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Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples

FY 2015: Two Samples Contained 5+ Violative Compounds

Bob Veal	Heavy Calf
Ciprofloxacin (kidney)	Desfuroylceftiofur (kidney)
Desethylene ciprofloxacin (liver)	Florfenicol (muscle)
Enrofloxacin (kidney)	Flunixin (muscle)
Neomycin (kidney)	Gentamycin Sulfate (kidney)
Penicillin (kidney)	Penicillin (kidney)
Sulfadiazine (kidney)	Penicillin (muscle; non-violative)
Tetracycline (kidney; non-violative)	

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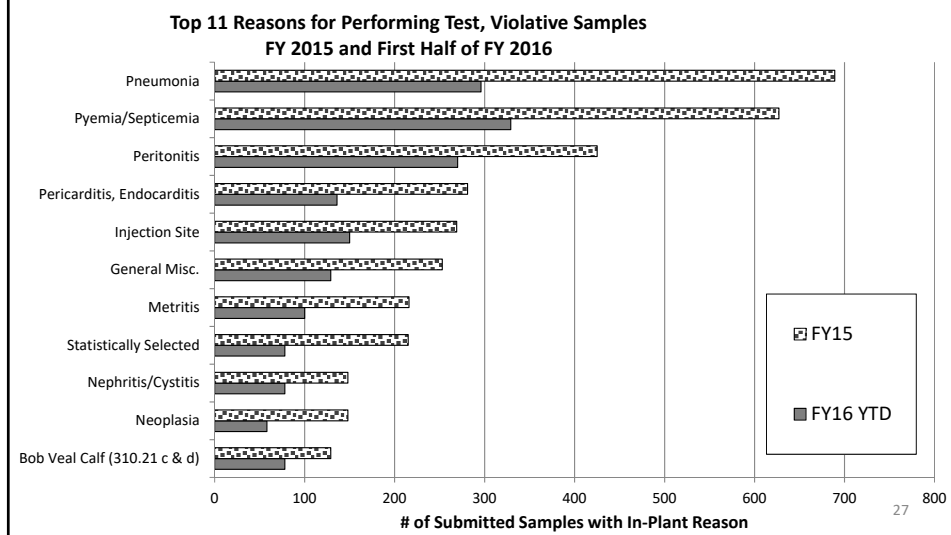
Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples

FSIS personnel can choose from 30+ reasons for performing a KIS™ test.

- Most reasons cite a medical condition
- FY 2015:
 - The **top 11** reasons accounted for **82%** of violations
 - The **top 2** reasons (**pneumonia** and **pyemia/septicemia**) accounted for **32%** of violations.
- First Half of FY 2016:
 - The **top 11** reasons are the same top 11 of FY 2015, but in a different order. They account for **86%** of violations.
 - The **top 2** reasons (**pyemia/septicemia** and **pneumonia**) also account for **32%** of violations.

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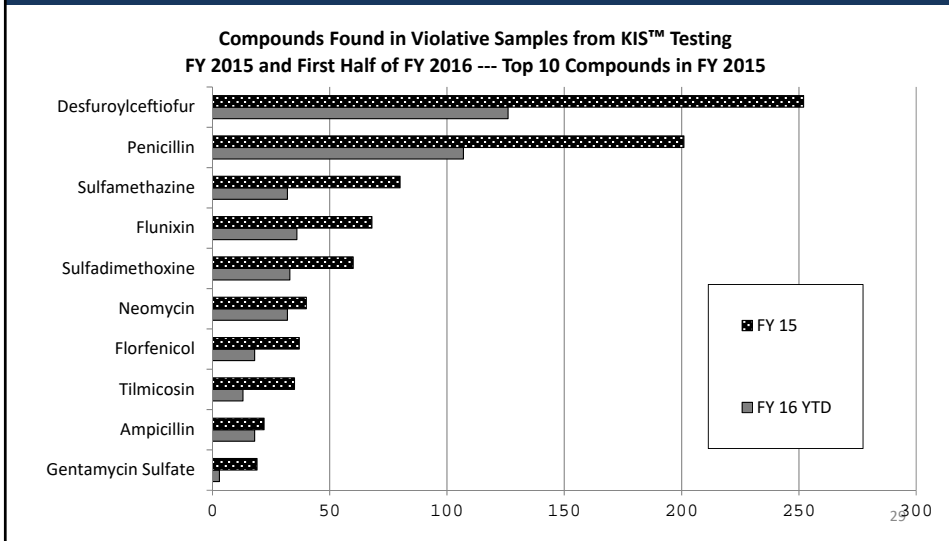
Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples



Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples

- In FY 2015, the **top 2 compounds** accounted for **50% (453)** of all violative results (907 total):
 - Desfuroylceftiofur (252)
 - Penicillin (201)
- FY 2015: **32 compounds** with at least one violative finding.
- FY 2016 YTD: **25 compounds** have at least one violative finding.

Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples



Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples

Year	Top Three Violative Chemical Residues	Top Three Positive Non-Violative Chemical Residues
* FY 2013	Ceftiofur Penicillin Neomycin	Oxytetracycline Neomycin Ceftiofur
FY 2014	Ceftiofur Penicillin Neomycin	Oxytetracycline Tulathromycin Penicillin
FY 2015 (Preliminary)	Ceftiofur Penicillin Sulfamethazine	Tulathromycin Oxytetracycline Ceftiofur

Source: FSIS Data Warehouse (DW) & Public Health Information System (PHIS)
 * Results from Jan 2013 to Sept 2013

Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples

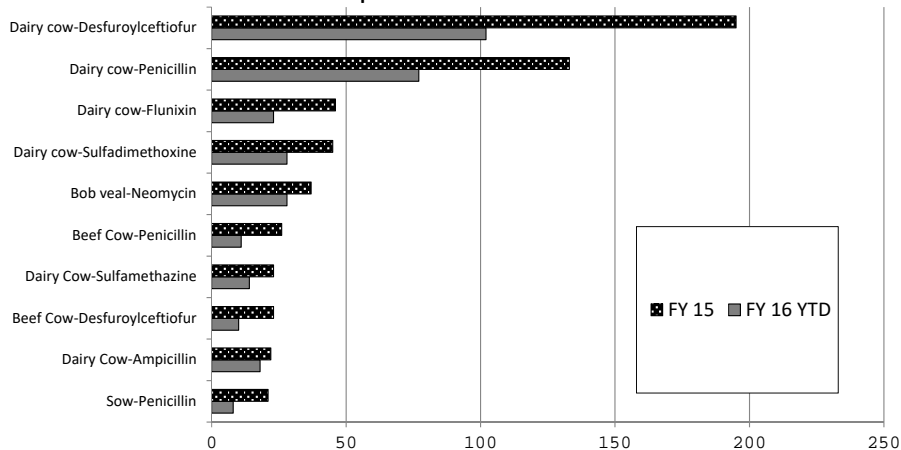
In FY 2015, the **top five** animal/compound combinations accounted for **50%** of violations:

- Dairy cow and desfuroylceftiofur
- Dairy cow and penicillin
- Dairy cow and flunixin
- Dairy cow and sulfadimethoxine
- Bob veal and neomycin

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Food Safety and Inspection Service:
IV. Preliminary Test Results – Inspector Generated Samples

Animal/Compound Combinations, FY 2015 and First Half of FY 2016
Top 10 in FY 2015



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Food Safety and Inspection Service: V. Preliminary Test Results – Import Samples

Year	Number of Samples (number of analytes)	Total Violations
*FY 2013	817 (993)	4 All Ivermectin
FY 2014	1,967 (5,104)	8 7 Ivermectin 1 Zilpaterol
FY 2015 (preliminary)	2,918 (15,221)	7 6 Pesticide 1 Arsenic
FY 2016 Oct 2015-Mar 2016 (preliminary)	1,239 (5,665)	9 All Ethion

Source: FSIS Data Warehouse (DW) & Public Health Information System (PHIS)
* Results from Jan 2013 to Sept 2013

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Food Safety and Inspection Service: VI. Where can I find more information?

For [Chemistry Methods](#): FSIS Chemistry Laboratory Guidebook (CLG) website



Chemistry Laboratory Guidebook

This **Chemistry Laboratory Guidebook** contains test methods used by FSIS Laboratories to support the Agency's inspection program, ensuring that meat, poultry, and egg products are safe, wholesome and accurately labeled.

The Guidebook contains methods for the analysis of food composition, food additives, nutrients, veterinary drug and pesticide residues. Methods are designed to provide analysts with written documentation to facilitate training, performance, quality assessment, and interpretation of data.

The contents of this Guidebook are continuously revised and updated. Future updates will include other methods previously published in the printed version of the Guidebook, which is no longer available for distribution.



Receive email notification when the Chemistry Laboratory Guidebook is updated.

To get updates
by email



Method Number	Method Title	Effective Date
FOOD CHEMISTRY		
F01	Moisture Determination (PDF Only)	Aug 10, 2009
F02	Protein Determination by Combustion (PDF Only)	Jul 27, 2009
F03	Determination of Fat (PDF Only)	Aug 10, 2009
F04	Determination of Salt (PDF Only)	Jul 27, 2009

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Food Safety and Inspection Service: VI. Where can I find more information?

Residue Sampling Plans (a.k.a. the **FSIS Blue Book**)

Residue Sample Results (a.k.a. the **FSIS Red Book**)

<http://www.fsis.usda.gov/wps/portal/fsis/topics/data-collection-and-reports/chemistry/residue-chemistry>

Residue Chemistry

 Receive email notification when the Residue Repeat Violators List has been updated.

- [Residue Testing; National Residue Program](#)
 - [Residue Repeat Violators List](#)
- [Screening Tests](#)
- [Dioxins](#)
- [Melamine](#)

RESIDUE TESTING; NATIONAL RESIDUE PROGRAM

2016 Residue Sampling Plans ("Blue Book")

This is the most recent "Blue Book" explaining of the process used to plan the U.S. National Residue Program (NRP) for Meat, Poultry, and Egg Products. [Program plans for past years](#) are also available.

2013 and 2014 U.S. National Residue Sample Results ("Red Book")

The most recent Red Books explain FSIS' chemical residue sampling plans and present NRP testing results. [Earlier editions of the Red Book](#) are also available.

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Food Safety and Inspection Service: Questions?

Acknowledgments:

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