

## **Appendix IV**

### **FSIS Laboratory Analytical Methods**

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The Food Safety and Inspection Service (FSIS) requires analytical methods for detecting, quantifying, and identifying residues that may be present in meat, poultry, and processed egg products. These methods can be used by the Agency for monitoring and surveillance activities to determine whether a product is adulterated and for human risk assessment evaluations. The Agency uses available methodology to take appropriate regulatory action against adulterated products, consistent with the reliability of the analytical data. This section describes the types of methods used by FSIS to conduct analyses.

**Table AIV**  
**Analytical Methods**  
**2005 National Residue Program**

Compound Class	Compound	Analytical Method			Minimum Proficiency Level <sup>a</sup>			
		Screen	Determinative (quantitative)	Confirmatory (identification)	Screen	Determinative (quantitative)	Confirmatory (identification)	
Antibiotics	Carbadox		GC-ECD	TBD		15 ppb	TBD	
	Chloramphenicol		GC	GC-MS		0.25 ppb (M)	0.30 ppb (M)	
	Florfenicol		HPLC	GC/SIM-MS		1.9 ppm (L)	1.9 ppm (L)	
Antibiotics : <i>beta</i> -Lactams	Amoxicillin	7-Plate Bioassay	Bioassay			TBD		
	Ampicillin					0.01 ppm		
	Cefazolin					0.02 ppm		
	Cloxacillin					TBD		
	Desacetyl cephapirin					0.1 ppm		
	Desfuroylceftiofur cysteine disulfide (DCCD)					0.05 ppm		
	Dicloxacillin					0.05 ppm		
	Nafcillin							
	Penicillin-G					0.05 ppm		
Antibiotics : Tetracyclines	Chlortetracycline	7-Plate Bioassay	Bioassay	HPLC (chemistry)	0.5 ppm	0.08 ppm		
	Oxytetracycline							
	Tetracycline							
Antibiotics: Macrolides	Clindamycin	7-Plate Bioassay	Bioassay	MS		0.1 ppm		
	Erythromycin							
	Lincomycin							
	Pirlimycin							
	Tilmicosin		HPLC- Ion Pairing			0.05 ppm		
	Tylosin		Bioassay			0.1 ppm		
							300 ppb (M) 600 ppb (L,K)	
							0.1 ppm	
							0.2 ppm	
							0.1 ppm	

**Table AIV – continued**  
**Analytical Methods**  
**2005 National Residue Program**

Compound Class	Compound	Analytical Method			Minimum Proficiency Level <sup>a</sup>		
		Screen	Determinative (quantitative)	Confirmatory (identification)	Screen	Determinative (quantitative)	Confirmatory (identification)
Antibiotics: Aminoglycosides	Amikacin	7-Plate Bioassay		MS			1.0 ppm (L,K), 0.4 ppm (M)
	Apramycin						0.4 ppm (K) 0.1 ppm (L,M)
	Dihydrostreptomycin		Bioassay				0.4 ppm (L,K,M)
	Gentamicin		Bioassay			0.15 ppm	0.1 ppm (K,M), 0.4 (L)
	Hygromycin						1.0 ppm (L,K) 0.4 ppm (M)
	Kanamycin						4.0 ppm(L), 2.0 ppm (K), 0.4 ppm (M)
	Neomycin		Bioassay			0.25 ppm	0.1ppm (K,M), 0.4 (L)
	Spectinomycin					10.0 ppm	1.0 ppm (L) 0.4 ppm (K) 0.25 ppm (M)
	Streptomycin		Bioassay			0.1 ppm	0.4 ppm (L,K,M)
	Tobramycin						1.0 ppm (L) 0.1 ppm (K,M)
Arsenicals	Arsenicals		AA	AA		0.2 ppm	0.2 ppm
Avermectins	Ivermectin		HPLC	HPLC/APCI-MS		7.5 ppb	25 ppb
	Doramectin						
	Moxidectin						
<i>beta</i> -Agonists	Cimaterol	ELISA			6 ppb		
	Clenbuterol	ELISA		LC/MS-MS	3 ppb		TBD
	Ractopamine		HPLC	LC/MS		1 ppb (M), 25 ppb (L)	1 ppb
	Salbutamol	ELISA			3 ppb		
Hormones, synthetic	Diethylstilbestrol (DES)		GC-MS	GC-MS		0.5 ppb	1.0 ppb (L,M)
	Zeranol	ELISA	GC-MS	GC-MS		0.5 ppb	5.0 ppb (L)
	<i>alpha</i> -Trenbolone			GC/MS-MS			5.0 ppb (L)
	<i>beta</i> -Trenbolone			GC/MS-MS			5.0 ppb (M)

**Table AIV – *continued***  
**Analytical Methods**  
**2005 National Residue Program**

Compound Class	Compound	Analytical Method			Minimum Proficiency Level <sup>a</sup>		
		Screen	Determinative (quantitative)	Confirmatory (identification)	Screen	Determinative (quantitative)	Confirmatory (identification)
Nonsteroidal Anti-inflammatory Drugs (NSAIDs)	Dipyrrones <sup>b</sup>	HPLC	HPLC		0.2 ppm	0.2 ppm	
	Flunixin	ELISA	HPLC	HPLC/ESI-MS-MS	50 ppb	62.5 ppb	125 ppb
	Phenylbutazone	ELISA		HPLC/ESI-MS-MS	50 ppb		50 ppb
Anabolic Steroids	Melengesterol Acetate (MGA)		GC/ECD	HPLC/APCI-MS		10 ppb	12.5 ppb
Sulfonamides	Sulapyridine						
	Sulfadiazine						
	Sulfathiazole						
	Sulfamerazine						
	Sulfamethazine						
	Sulfachloropyridazine						
	Sulfamethoxypyridazine						
	Sulfaquinoxaline						
	Sulfadimethoxine						
	Sulfaethoxypyridazine						
	Sulfaphenazole						
	Sulfatroxazole						
	Sulfisoxazole						
Thyreostats	Sulfadoxine						
	2-Mercaptobenzimidazole						
	6-Methyl-2-thiouracil						
	2-Mercapto-1-methylimidazole						
	6-Phenyl-2-thiouracil			HPLC/MS-MS			25 ppb

**Table AIV – continued**  
**Analytical Methods**  
**2005 National Residue Program**

Compound Class	Compound	Analytical Method			Minimum Proficiency Level <sup>a</sup>		
		Screen	Determinative (quantitative)	Confirmatory (identification)	Screen	Determinative (quantitative)	Confirmatory (identification)
Thyreostats (continued)	6-Propyl-2-thiouracil			HPLC/MS-MS			25 ppb
	2-Thiouracil						
CHCs/COPs/PCBs	Aldrin	GPC with GC-EC	GC-MS		0.10 ppm		
	<i>alpha</i> -BHC				0.10 ppm	0.01 ppm	
	Captan				0.04 ppm		
	Carbophenothion				0.06 ppm		
	Chlorfenvinphos					0.05 ppm	
	Chlorpyrifos					0.10 ppm	
	<i>cis</i> -chlordane					0.30 ppm	
	Coumaphos-O					0.20 ppm	
	Coumaphos-S					0.20 ppm	
	Dieldrin					0.10 ppm	0.01 ppm
	Endosulfan I				0.02 ppm		
	Endosulfan II					0.04 ppm	
	Endrin					0.10 ppm	0.03 ppm
	HCB					0.10 ppm	0.01 ppm
	Heptachlor epoxide					0.10 ppm	0.10 ppm
	Heptacholr					0.10 ppm	0.01 ppm
	Kepone				0.06 ppm		
	Lindane					0.10 ppm	0.01 ppm
	Linuron				0.50 ppm		
	Methoxychlor					0.50 ppm	0.15 ppm
	Mirex					0.10 ppm	

**Table AIV – continued**  
**Analytical Methods**  
**2005 National Residue Program**

Compound Class	Compound	Analytical Method			Minimum Proficiency Level <sup>a</sup>		
		Screen	Determinative (quantitative)	Confirmatory (identification)	Screen	Determinative (quantitative)	Confirmatory (identification)
CHCs/COPs/PCBs (continued)	Nonachlor	GPC with GC-EC	GC-MS	0.15 ppm	0.15 ppm	0.15 ppm	0.15 ppm
	o,p'-TDE						
	Oxychlordane						
	p,p'-DDE						
	p,p'-DDT						
	p,p'-TDE						
	PCB 1260						
	PCB 1254						
	PCB 1242						
	PCB 1248						
	Phosalone						
	Ronnel						
	Stirofos						
	Toxaphene						
	trans-chlordanne						

a Minimum Proficiency Level: The lowest amount of individual residue or sample component that FSIS requires its laboratories to reliably detect, quantify, or confirm. This is usually the lowest amount for which the method used by FSIS laboratories has been validated.

b 4-methylaminoantipyrine, 4-formylaminoantipyrine, and 4-aminoantipyrine

**Table AIV – *continued***  
**Analytical Methods**  
**2005 National Residue Program**

Key:

AA = Atomic Absorption Spectroscopy

APCI = Atmospheric Pressure Chemical Ionization

CHCs = Chlorinated hydrocarbons

COPs = Chlorinated organophosphates

ECD = Electron Capture Detection

ELISA = Enzyme Linked Immunosorbent Assay

GC = Gas Chromatography

GPC = Gel Permeation Chromatography

HPLC = high performance liquid chromatography

K = Kidney

L = Liver

M = Muscle

Method detection limit = The lowest quantity of residue (or sample component) that can be reliably observed or found in the sample matrix by the analytical methodology used.

MS = Mass Spectroscopy

NA = not applicable

PCBs = Polychlorinated biphenyls

ppb = parts per billion

ppm = parts per million

SIM = selected ion mode

TBD = To be determined

TLC = Thin Layer Chromatography

## **Appendix V**

### **Statistical Table**

## Statistical Table

Table V, *Statistical Table*, indicates the number of samples required to ensure detection of a violation that affects a given percentage of the sampled population.

Table AV  
Statistical Table  
2005 FSIS National Residue Program

Percentage Violative in Sampled Population	Probability of Detection (Percent)			
	90	95	99	99.9
	Samples Required			
10	22	29	44	66
5	45	59	90	135
1	230	299	459	688
0.5	460	598	919	1,379
0.1	2,302	2,995	4,603	6,905
0.05	4,605	5,990	9,209	13,813