

# **Appendix I**

## **Tissues Required for Laboratory Analysis**

## Tissues Required for Laboratory Analysis

Table A-I Lists the tissue, the amount required for analysis, and the laboratory to which the tissue is sent.

Table A-I			
Residue	Tissue Analyzed	Quantity (lb)	Lab
Antibiotics	Kidney, liver, muscle	1	ML <sup>1</sup>
Arsenicals	Liver, muscle	1	EL <sup>2</sup>
Avermectins	Liver, muscle	1	EL
<i>beta</i> -Agonists <sup>3</sup>	Eyeball, liver	1	WL <sup>4</sup>
Berenil	Whole blood for plasma	1	FDA-Denver
Carbadox	Liver, muscle	1	WL
Chloramphenicol	Muscle	1	EL
Chlorinated hydrocarbons/chlorinated organophosphates/Phenylbutazone	Fat	1	WL
Dipyrones <sup>5</sup>	Muscle	1	WL
Florfenicol	Liver, muscle	1	EL
Flunixin	Liver, muscle	1	ML
Lead and Cadmium	Kidney, muscle	1	EL
Melengesterol acetate (MGA)	Fat	1	WL
Nitrofurans	Liver, muscle	1	WL
Nitroimidazoles	Muscle	1	EL

<sup>1</sup> FSIS Midwestern Laboratory

<sup>2</sup> FSIS Eastern Laboratory

<sup>3</sup> Clenbuterol, salbutamol, and cimaterol.

<sup>4</sup> FSIS Western Laboratory

<sup>5</sup> 4-Methylaminoantipyrine, 4-formylaminoantipyrine, and 4-aminoantipyrine.

<b>Table A-I</b>			
<b>Residue</b>	<b>Tissue Analyzed</b>	<b>Quantity (lb)</b>	<b>Lab</b>
Phenylbutazone by Immunoassay	Kidney, muscle <sup>6</sup>	1	WL
Ractopamine	Liver, muscle	1	WL
Sulfonamides	Liver, muscle	1	EL
Thyreostats	Muscle (or thyroid)	1	EL
Trenbolone	Liver, muscle	1	ML
Zeranol	Liver, muscle	1	ML

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<sup>6</sup> Method has not been validated in muscle.

# **Appendix II**

## **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 AII  
Analytical Methods  
2006 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-ECD	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	HPLC/MS- MS		TBD	TBD
	Ampicillin					0.01 ppm	10 ppb
	Cefazolin					0.02 ppm	50 ppb
	Cloxacillin					TBD	TBD
	Desacetyl cephalixin					0.1 ppm	100 ppb
	Desfuoylceftiofur cysteine disulfide (DCCD)					0.05 ppm	50 ppb
	Dicloxacillin					0.05 ppm	50 ppb
	Nafcillin						20 ppb
	Penicillin-G					0.05 ppm	50 ppb
	Oxacillin						TBD
Antibiotics : Tetracyclines	Chlortetracycline	7-Plate Bioassay	Bioassay	HPLC	0.01 ppm	0.05 ppm	0.5 ppm
	Oxytetracycline				0.5 ppm	0.40 ppm	
	Tetracycline						
Antibiotics: Macrolides	Clindamycin	7-Plate Bioassay		HPLC/MS- MS			0.1 ppm
	Erythromycin		Bioassay			0.05 ppm	0.1 ppm
	Lincomycin						0.1 ppm
	Pirlimycin						0.1 ppm
	Tilmicosin		HPLC- Ion Pairing			300 ppb (M) 600 ppb (L,K)	0.1 ppm
	Tylosin		Bioassay			0.2 ppm	0.1 ppm

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		Screen	Determinative (quantitative)	Confirmatory (identification)	Screen	Determinative (quantitative)	Confirmatory (identification)
Antibiotics: Aminoglycosides	Amikacin	7-Plate Bioassay		HPLC/MS- MS			1.0 ppm (L,K), 0.4 ppm (M)
	Apramycin					0.4 ppm (K) 0.1 ppm (L,M)	
	Dihydrostreptomycin		Bioassay			0.5 ppm	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		AAS	AAS		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	Diethylstilbesterol (DES)		GC-MS	GC-MS		0.5 ppb	1.0 ppb (L,M)
	Zeranol	ELISA	GC-MS	GC-MS	0.5 ppb	1.0 ppb	5.0 ppb (L)
	<i>alpha</i> -Trenbolone			GC/MS-MS	5.0 ppb		5.0 ppb (L)
	<i>beta</i> -Trenbolone			GC/MS-MS			5.0 ppb (M)

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<i>Compound Class</i>	<i>Compound</i>	<i>Analytical Method</i>			<i>Minimum Proficiency Level<sup>a</sup></i>		
		<i>Screen</i>	<i>Determinative (quantitative)</i>	<i>Confirmatory (identification)</i>	<i>Screen</i>	<i>Determinative (quantitative)</i>	<i>Confirmatory (identification)</i>
Nonsteroidal Anti-inflammatory Drugs (NSAIDs)	Dipyrones <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)	ELISA	GC/ECD	HPLC/APCI-MS	5 ppb	10 ppb	12.5 ppb
Sulfonamides	Sulfapyridine		TLC	GC/ESI-MS		0.05 ppm	0.1 ppm
	Sulfadiazine						
	Sulfathiazole						
	Sulfamerazine						
	Sulfamethazine						
	Sulfachloropyridazine						
	Sulfamethoxypryridazine						
	Sulfaquinoxaline						
	Sulfadimethoxine						
	Sulfaethoxypyridazine						
	Sulfaphenazole						
	Sulfatroxazole						
Sulfisoxazole							
Sulfadoxine							
Thyreostats	2-Mercaptobenzimidazole			HPLC/MS-MS			25 ppb
	6-Methyl-2-thiouracil						
	2-Mercapto-1-methylimidazole						
	6-Phenyl-2-thiouracil						



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<i>Compound Class</i>	<i>Compound</i>	<i>Analytical Method</i>			<i>Minimum Proficiency Level<sup>a</sup></i>			
		<i>Screen</i>	<i>Determinative (quantitative)</i>	<i>Confirmatory (identification)</i>	<i>Screen</i>	<i>Determinative (quantitative)</i>	<i>Confirmatory (identification)</i>	
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
	Heptachlor						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				

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		Screen	Determinative (quantitative)	Confirmatory (identification)	Screen	Determinative (quantitative)	Confirmatory (identification)
CHCs/COPs/PCBs (continued)	Nonachlor		GPC with GC-EC	GC-MS		0.15 ppm	
	o,p'-TDE					0.15 ppm	
	Oxychlorane					0.04 ppm	0.1 ppm
	p,p'-DDE					0.10 ppm	0.02 ppm
	p,p'-DDT					0.15 ppm	0.04 ppm
	p,p'-TDE					0.15 ppm	0.04 ppm
	PCB 1260					0.50 ppm	
	PCB 1254					0.50 ppm	
	PCB 1242					0.50 ppm	
	PCB 1248					0.50 ppm	
	Phosalone					0.02 ppm	
	Ronnel					0.03 ppm	
	Stirofos					0.06 ppm	
	Toxaphene					1.00 ppm	
trans-chlordane			0.30 ppm				

a. Minimum Proficiency Level: The minimum concentration of a residue at which an analytical result will be used to assess a laboratory's quantification capability. This concentration is an estimate of the smallest concentration for which the average coefficient of variation (CV) for reproducibility (i.e., combined within and between laboratory variability) does not exceed 20 percent (9 CFR 318.21).

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

**Table AII**  
**Analytical Methods**  
**2006 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 III**

## **Statistical Table**

## Statistical Table

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

Table AIII  
Statistical Table  
2006 FSIS National Residue Program

<i>Percentage Violative in Sampled Population</i>	<i>Probability of Detection (Percent)</i>			
	90	95	99	99.9
	<i>Samples Required</i>			
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