

# **APPENDIX VI. ANALYTICAL METHODS, 2002 FSIS NATIONAL RESIDUE PROGRAM**

Implementing the NRP requires analytical methods for detecting, quantifying, and identifying residues that may be present in meat, poultry, and egg products. Results from the residue testing are used by the Agency to determine whether a product is adulterated. The following Table AVI, *Analytical Methods*, describes the types of methods used by FSIS to conduct analyses.

## **KEY TO ABBREVIATIONS**

**APCI** -- Atmospheric Pressure Chemical Ionization

**Confirm.** -- Confirmatory Method

**Determ.** -- Determinative Method

**ECD** -- Electron Capture Detector

**ELISA** -- Enzyme-Linked Immuno Sorbent Assay

**GC** -- Gas Chromatograph

**GPC** -- Gel Permeation Chromatography

**HPLC** -- High Performance Liquid Chromatography

**Method Detection Limit** -- The lowest amount of individual residue or sample component that can be reliably observed or found in the sample matrix by the current appropriate analytical methodology.

**Minimum Reportable Level** -- Lowest level at which an analytical result is reported.

**MS** -- Mass Spectrometry

**NA** -- Not Applicable

**ppb** -- Parts per billion

**ppm** -- Parts per million

**SIM** -- Selected-Ion Monitoring Mode

**TBD** -- To Be Determined

**Table AVI  
Analytical Methods<sup>a</sup>  
2002 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		0.30 ppb	0.30 ppb		
	Florfenicol		HPLC	GC-MS		1.9 ppm (L)	1.9 ppm (L)		
Antibiotics : beta-Lactams	Penicillin	7-Plate Bioassay	Bioassay			0.01 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						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)	600 ppb
	Tylosin						Bioassay	0.2 ppm	0.1 ppm
Antibiotics: Aminoglycosides	Amikacin	7-Plate Bioassay	Bioassay	MS			1.0 ppm (L,K), 0.4 ppm (M)		
	Apramycin						0.4 ppm (K) 0.1 ppm (L,M)		
	Dihydrostreptomycin						0.4 ppm (L,K,M)		
	Gentamicin						0.15 ppm	0.1 ppm (K,M)	
	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)
	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)

**Table AVI – continued**  
**Analytical Methods<sup>a</sup>**  
**2002 National Residue Program**

Compound Class	Compound	Analytical Method			Minimum Proficiency Level <sup>a</sup>		
		Screen	Determinative (quantitative)	Confirmatory (identification)	Screen	Determinative (quantitative)	Confirmatory (identification)
Arsenicals	Arsenicals		AA	AA		0.2 ppm	0.2 ppm
Avermectins	Ivermectin, Doramectin, Moxidectin		HPLC	APCI/LC/MS		7.5 ppb	25 ppb
beta -Agonists	Cimaterol	ELISA			6 ppb		
	Clenbuterol	ELISA		LC-MS-MS	3 ppb		TBD
	Ractopamine		HPLC	LC/MS		1 ppb	1 ppb
	Salbutamol	ELISA			3 ppb		
Hormones, synthetic	DES		GC-MS	GC-MS		0.5 ppb	1.0 ppb
	Zeranol		GC-MS	GC-MS		0.5 ppb	1.0 ppb
Nonsteroidal Anti-inflammatory Drugs (NSAIDs)	Phenylbutazone	ELISA		LC-MS-MS	50 ppb		50 ppb
	Flunixin	ELISA	HPLC	LC/MS	50 ppb	31.3 ppb	125 ppb
Steroids	Melengesterol Acetate		GC	LC/MS		5 ppb	12.5 ppb
Sulfonamides	Sulfapyridine		TLC	GC-MS		0.05 ppm	0.05 ppm
	Sulfadiazine						
	Sulfathiazole						
	Sulfamerazine						
	Sulfamethazine						
	Sulfachloropyridazine						
	Sulfamethoxypryridazine						
	Sulfaquinoxaline						
	Sulfadimethoxine						
	Sulfaethoxypryridazine						
	Sulfaphenazole						

**Table AVI – continued**  
**Analytical Methods<sup>a</sup>**  
**2002 National Residue Program**

Compound Class	Compound	Analytical Method			Minimum Proficiency Level <sup>a</sup>			
		Screen	Determinative (quantitative)	Confirmatory (identification)	Screen	Determinative (quantitative)	Confirmatory (identification)	
Sulfonamides (continued)	Sulfatroxazole		TLC	GC-MS		0.05 ppm	0.05 ppm	
	Sulfisoxazole							
	Sulfadoxine							
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						0.06 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					0.01 ppm		
	Endosulfan II						0.06 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						

**Table AVI – continued**  
**Analytical Methods<sup>a</sup>**  
**2002 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)	Nonchlor		GPC with GC-EC	GC-MS		0.15 ppm	
	o,p'-DDT				0.15 ppm		
	Oxychlorthane					0.06 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	
	Phosalone				0.02 ppm		
	Ronnel					0.06 ppm	
	Stirofos					0.06 ppm	
	Toxaphene					1.00 ppm	
	trans-chlordane					0.30 ppm	

a Laboratory analytical methods reported here are current through May of 2004.

b 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.

**Key:**

L = Liver

K = Kidney

M = Muscle

AA = Atomic Absorption Spectroscopy

CHCs = Chlorinated hydrocarbons

COPs = Chlorinated organophosphates

PCBs = Polychlorinated biphenyls

GC = Gas Chromatography

MS = Mass Spectroscopy

GPC = Gel Permeation Chromatography

**Table AVI – *continued***  
**Analytical Methods<sup>a</sup>**  
**2002 National Residue Program**

TLC = Thin Layer Chromatography

ECD = Electron Capture Detection

ELISA = Enzyme Linked Immunosorbent Assay

ppm = parts per million

ppb = parts per billion

APCI = Atmospheric Pressure Chemical Ionization

HPLC = High Performance Liquid Chromatography

TBD = To be determined

## APPENDIX VII. STATISTICAL TABLE

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

**Table AVII  
Statistical Table**

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