

APPENDIX VI. ANALYTICAL METHODS, 2001 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
2001 National Residue Program**

Compound Class	Compound	Method Type	Methodology	Method Detection Limit	Minimum Reportable Level
Antibiotics	Carbadox	Determ.	GC-ECD	7.5 ppb	15 ppb
		Confirm.	GC-MS-SIM	NA	30 ppb
	Chloramphenicol	Determ.	GC	0.50 ppb	0.50 ppb
		Confirm.	GC-MS	0.5 ppb	0.5 ppb
	Florfenicol	Confirm.	GC-MS	1.9 ppm	1.9 ppm
	<u>Fluoroquinolones:</u>	Determ.	HPLC	Enrofloxacin 25 ppb	25 ppb
	Ciprofloxacin 50 ppb			50 ppb	
	Desethylene ciprofloxacin 12.5 ppb			12.5 ppb	
	Sarafloxacin 50 ppb			50 ppb	
	Danofloxacin 5.0 ppb			5.0 ppb	
	Difloxacin 50 ppb			50 ppb	
	Marbofloxacin 50 ppb			50 ppb	
	Orbifloxacin 25 ppb			25 ppb	
	Tilmicosin			Determ.	HPLC- Ion Pairing
		Confirm.	APCI-LC-MS	0.05 ppm	
<u>Antibiotics in FSIS Bioassay Method:</u>	Determ.	7-plate microbiological inhibition assay	Penicillin 0.01 ppm	0.01 ppm	
Chlortetracycline 0.01 ppm			0.01 ppm		
Tetracycline or Oxytetracycline 0.08 ppm			0.08 ppm		
Streptomycin 0.10 ppm			0.10 ppm		
Neomycin 0.25 ppm			0.25 ppm		
Erythromycin 0.05 ppm			0.05 ppm		
Gentamicin 0.15 ppm			0.15 ppm		
Ampicillin 0.01 ppm			0.01 ppm		
Nnovobiocin 0.25 ppm			0.25 ppm		
Spectinomycin 10.0 ppm			10.0 ppm		
Tylosin 0.20 ppm			0.20 ppm		
Arsenicals			Arsenicals	Determ.	Atomic Absorption Spectrophotometry
Avermectins	Ivermectin Doramectin Moxidectin	Determ.	HPLC	2.0 ppb	7.5 ppb
		Confirm.	APCI/LC/MS	25 ppb	

Table AVI - continued
Analytical Methods
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Compound Class	Compound	Method Type	Methodology	Method Detection Limits	Minimum Reportable Level
Beta -Agonists	Ractopamine	Determ.	HPLC		Muscle 25 ppb Liver 75 ppb
		Confirm.	LC/MS	25 ppb	
	Clenbuterol	Screen	ELISA	3 ppb	3 ppb
		Confirm.	LC-MS-MS		
Chlorinated Hydrocarbons/ Chlorinated Organophosphates/ Polychlorinated biphenyls	<u>Organohalides:</u>	Determ.	GPC with GC-EC		
	HCB			0.10 ppm	0.10 ppm
	Alpha BHC			0.10 ppm	0.10 ppm
	Lindane			0.10 ppm	0.10 ppm
	Heptachlor			0.10 ppm	0.10 ppm
	Aldrin			0.10 ppm	0.10 ppm
	Ronnel			0.06 ppm	0.06 ppm
	Linuron			0.50 ppm	0.50 ppm
	Oxychlorthane			0.06 ppm	0.06 ppm
	Chlorpyrifos			0.10 ppm	0.10 ppm
	Nonchlor			0.15 ppm	0.15 ppm
	Heptachlor epoxide			0.10 ppm	0.10 ppm
	Endosulfan I			0.02 ppm	0.02 ppm
	Ttrans-chlordane			0.30 ppm	0.30 ppm
	Cis-chlordane			0.30 ppm	0.30 ppm
	Chlorfenvinphos			0.06 ppm	0.06 ppm
	Dieldrin			0.10 ppm	0.10 ppm
	P,p'-DDE			0.10 ppm	0.10 ppm
	Captan			0.04 ppm	0.04 ppm
	Stirofos			0.06 ppm	0.06 ppm
	Kepone			0.06 ppm	0.06 ppm
	Endrin			0.10 ppm	0.10 ppm
	P,p'-TDE			0.15 ppm	0.15 ppm
	O,p'-DDT			0.15 ppm	0.15 ppm
	Endosulfan II			0.06 ppm	0.06 ppm
	P,p'-DDT			0.15 ppm	0.15 ppm
	Carbophenothion			0.06 ppm	0.06 ppm
	Mirex			0.10 ppm	0.10 ppm
	Methoxychlor			0.50 ppm	0.50 ppm
	Phosalone			0.02 ppm	0.02 ppm
	Coumaphos-O			0.20 ppm	0.20 ppm
	Coumaphos-S			0.20 ppm	0.20 ppm
	Toxaphene	1.00 ppm	1.00 ppm		
PCB 1242	0.50 ppm	0.50 ppm			
PCB 1248	0.50 ppm	0.50 ppm			
PCB 1254	0.50 ppm	0.50 ppm			
PCB 1260	0.50 ppm	0.50 ppm			
		Confirm.	GC-MS	NA	NA

Table AVI - continued
Analytical Methods
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Compound Class	Compound	Method Type	Methodology	Method Detection Limits	Minimum Reportable Level
Hormones, synthetic	DES Zeranol	Determ. & Confirm.	GC-MS	0.5 ppb 1.0 ppb	0.5 ppb 1.0 ppb
Nonsteroidal Anti-inflammatory Drugs (NSAIDs)	Phenylbutazone	Determ.	GPC with GC-ECD	0.5 ppm	0.5 ppm
		Confirm.	GC-MS	0.05 ppm	0.05 ppm
Steroids	Melengesterol Acetate (MGA)	Determ.	GC	5 ppb	10 ppb
		Confirm.		NA	
Sulfonamides	Sulfapyridine Sulfadiazine Sulfathiazole Sulfamerazine Sulfamethazine Sulfachloropyridazine Sulfamethoxypryridazine Sulfaquinoxaline Sulfadimethoxine Sulfaethoxypyridazine Sulfaphenazole Sulfatroxazole Sulfisoxazole Sulfadoxine	Determ.	TLC	0.05 ppm	0.05 ppm
		Confirm.	GC-MS	NA	NA

Confirm .= Confirmatory Method

Determ. = Determinative Method

NA = Not Applicable

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