

APPENDIX V. ANALYTICAL METHODS, 2000 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 AV, *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.

MS -- Mass Spectrometry

NA -- Not Applicable

ppb -- Parts per billion

ppm -- Parts per million

SIM -- Selected-Ion Monitoring Mode

TBD -- To Be Determined

Table AV
Analytical Methods
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Compound Class	Compound	Method Type	Methodology	Method Detection Limit	
Antibiotics	Carbadox	Determ.	GC-ECD	7.5 ppb	
		Confirm.	GC-MS-SIM	NA	
	Chloramphenicol	Determ.	GC	0.25 ppb	
		Confirm.	GC-MS	0.5 ppb	
	Florfenicol	Confirm.	GC-MS	1.9 ppm	
	<u>Fluoroquinolones:</u>				
	Enrofloxacin	Determ.	HPLC	25 ppb	
	Ciprofloxacin			50 ppb	
	Desethylene ciprofloxacin			12.5 ppb	
	Sarafloxacin			50 ppb	
	Danofloxacin			50 ppb	
	Difloxacin			50 ppb	
	Marbofloxacin			50 ppb	
	Orbifloxacin			25 ppb	
	Tilmicosin			Determ.	HPLC- Ion Pairing
Confirm.		APCI-LC-MS	0.05 ppm		
<u>Antibiotics in FSIS</u> <u>Bioassay Method:</u>					
Penicillin	Determ.	7-plate microbiological inhibition assay	0.01 ppm		
Chlortetracycline			0.01 ppm		
Tetracycline or Oxytetracycline			0.08 ppm		
Streptomycin			0.1 ppm		
Neomycin			0.25 ppm		
Erythromycin			0.05 ppm		
Gentamicin sulfate			0.15 ppm		
Ampicillin			0.01 ppm		
Novobiocin			0.25 ppm		
Spectinomycin			10 ppm		
Tylosin			0.2 ppm		
Arsenicals	Arsenicals	Determ.	Atomic Absorption Spectrophotometry	0.2 ppm	
Avermectins	Ivermectin Doramectin Moxidectin	Determ.	HPLC	2.0 ppb	
		Confirm.	APCI-LC-MS	25 ppb	

Table AV- continued
Analytical Methods
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Compound Class	Compound	Method Type	Methodology	Method Detection Limit
Beta Agonists	Ractopamine	Determ.	HPLC	
		Confirm.	LC/MS	25ppb
	Clenbuterol	Screen	ELISA	TBD
		Confirm.	LC-MS-MS	
Chlorinated Hydrocarbons/ Chlorinated Organophosphates/ Polychlorinated Biphenyls	<u>Organohalides:</u>	Determ.	GPC with GC- ECD	
	HCB			0.01 ppm
	Alpha BHC			0.01 ppm
	Lindane			0.01 ppm
	Heptachlor			0.01 ppm
	Aldrin			0.02 ppm
	Ronnel			0.02 ppm
	Linuron			0.25 ppm
	Oxychlordane			0.02 ppm
	Chlorpyrifos			0.05 ppm
	Nonachlor			0.03 ppm
	Heptachlor epoxide			0.01 ppm
	Endosulfan I			0.02 ppm
	Trans-chlordane			0.10 ppm
	Cis-chlordane			0.10 ppm
	Chlorfenvinphos			0.03 ppm
	Dieldrin			0.01 ppm
	P, p'-DDE			0.02 ppm
	Captan			0.02 ppm
	Stirofos			0.05 ppm
	Kepone			0.03 ppm
	Endrin			0.03 ppm
	P, p'-TDE			0.03 ppm
	O, p'-DDT			0.04 ppm
	Endosulfan II			0.02 ppm
	P, p'-DDT			0.03 ppm
	Carbophenothion			0.03 ppm
	Mirex			0.04 ppm
	Methoxychlor			0.15 ppm
	Phosalone			0.01 ppm
	Coumaphos-O			0.15 ppm
	Coumaphos-S			0.15 ppm
	Toxaphene			0.50 ppm
PCB 1242	0.30 ppm			
PCB 1248	0.30 ppm			
PCB 1254	0.30 ppm			
PCB 1260	0.30 ppm			
	Confirm.	GC-MS	NA	

Table AV- continued
Analytical Methods
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Compound Class	Compound	Method Type	Methodology	Method Detection Limit
Hormones, synthetic	DES/Zeranol	Determ. & Confirm.	GC-MS	0.5 ppb
Nonsteroidal Anti-inflammatory Drugs (NSAID's)	Phenylbutazone	Determ.	GPC with GC-ECD	TBD
		Confirm.	GC-MS	TBD
Steroids	Melengesterol Acetate (MGA)	Determ.	GC	5 ppb
		Confirm.		NA
Sulfonamides	Sulfapyridine Sulfadiazine Sulfathiazole Sulfamerazine Sulfamethazine Sulfachloropyridazine Sulfamethoxypryridazine Sulfaquinoxaline Sulfadimethoxine Sulfaethoxypryridazine Sulfaphenazole Sulfatroxazole Sulfisoxazole	Determ.	TLC	0.05 ppm
		Confirm.	GC-MS	NA

APPENDIX VI. STATISTICAL TABLE

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

**Table AVI
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