

# **APPENDIX IV. ANALYTICAL METHODS, 2003 FSIS NATIONAL RESIDUE PROGRAM**

## **INTRODUCTION**

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.

## **KEY TO ABBREVIATIONS**

**APCI** -- Atmospheric Pressure Chemical Ionization

**Confirm** -- Confirmatory Method

**Determ.** -- Determinative Method

**ECD** -- Electron Capture Detector

**ELISA** -- Enzyme-Linked Immunosorbent Assay

**GC** -- Gas Chromatography

**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** -- The lowest level at which the 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 AIV  
Analytical Methods  
2003 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	25 ppb	25 ppb		
	Enrofloxacin			50 ppb	50 ppb		
	Ciprofloxacin			12.5 ppb	12.5 ppb		
	Desethylene ciprofloxacin						
	Sarafloxacin					50 ppb	50 ppb
	Danofloxacin					5 ppb	5 ppb
	Difloxacin					50 ppb	50 ppb
	Marbofloxacin					50 ppb	50 ppb
	Orbifloxacin					25 ppb	25 ppb
	Tilmicosin	Determ.	HPLC- Ion Pairing	Muscle 300 ppb Liver and Kidney 600ppb			
		Confirm.	APCI-LC-MS	0.05 ppm			
	<u>Antibiotics in FSIS Bioassay Method:</u>	Determ.	7-plate microbiological inhibition assay	0.01 ppm	0.01 ppm		
Penicillin	0.01 ppm			0.01 ppm			
Chlortetracycline	0.08 ppm			0.08 ppm			
Tetracycline or Oxytetracycline							
Streptomycin					0.1 ppm	0.1 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 ppm	10 ppm	
Tylosin					0.2 ppm	0.2 ppm	
Arsenicals	Arsenicals	Determ.	Atomic Absorption Spectrophotometry		0.2 ppm		
Avermectins	Ivermectin Doramectin Moxidectin	Determ.	HPLC	2.0 ppb	7.5 ppb		
		Confirm.	APCI/LC/MS	25 ppb			

**Table AIV - 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	25ppb	
	Clenbuterol	Screen	ELISA	TBD	
		Confirm.	LC-MS-MS		
Chlorinated Hydrocarbons/ Chlorinated Organophosphates/ Polychlorinated biphenyls	<u>Organohalides:</u>	Determ.	GPC with GC- EC		
	HCB			0.10ppm	0.10ppm
	Alpha BHC			0.10ppm	0.10ppm
	Lindane			0.10ppm	0.10ppm
	Heptachlor			0.10ppm	0.10ppm
	Aldrin			0.10ppm	0.10ppm
	Ronnel			0.06ppm	0.06ppm
	Linuron			0.50ppm	0.50ppm
	Oxychlorthane			0.06ppm	0.06ppm
	Chlorpyrifos			0.10ppm	0.10ppm
	Nonchlor			0.15ppm	0.15ppm
	Heptachlor epoxide			0.10ppm	0.10ppm
	Endosulfan I			0.01ppm	0.01ppm
	Ttrans-chlordane			0.30ppm	0.30ppm
	Cis-chlordane			0.30ppm	0.30ppm
	Chlorfenvinphos			0.06ppm	0.06ppm
	Dieldrin			0.10ppm	0.10ppm
	P,p'-DDE			0.10ppm	0.10ppm
	Captan			0.04ppm	0.04ppm
	Stirofos			0.06ppm	0.06ppm
	Kepone			0.06ppm	0.06ppm
	Endrin			0.10ppm	0.10ppm
	P,p'-TDE			0.15ppm	0.15ppm
	O,p'-DDT			0.15ppm	0.15ppm
	Endosulfan II			0.06ppm	0.06ppm
	P,p'-DDT			0.15ppm	0.15ppm
	Carbophenothion			0.06ppm	0.06ppm
	Mirex			0.10ppm	0.10ppm
	Methoxychlor			0.50ppm	0.50ppm
	Phosalone			0.02ppm	0.02ppm
	Coumaphos-O			0.20ppm	0.20ppm
	Coumaphos-S			0.20ppm	0.20ppm
	Toxaphene	1.00ppm	1.00ppm		
PCB 1242	0.50ppm	0.50ppm			
PCB 1248	0.50ppm	0.50ppm			
PCB 1254	0.50ppm	0.50ppm			
PCB 1260	0.50ppm	0.50ppm			
	Confirm.	GC-MS	NA	NA	

**Table AIV - continued**  
**Analytical Methods**  
**2003 National Residue Program**

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	TBD	TBD
		Confirm.	GC-MS	TBD	
Steroids	Melengesterol Acetate (MGA)	Determ.	GC	5 ppb	10 ppb
		Confirm.		NA	
Sulfonamides	Sulfapyridine Sulfadiazine Sulfathiazole Sulfamerazine Sulfamethazine Sulfachloropyridazine Sulfamethoxypryridazine Sulfaquinoxaline Sulfadimethoxine Sulfaethoxypryridazine 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 V. 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 A V**  
**Statistical Table**  
**2003 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