

APPENDIX IV. ANALYTICAL METHODS, 2002 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.

MS -- Mass Spectrometry

NA -- Not Applicable

ppb -- Parts per billion

ppm -- Parts per million

SIM -- Selected-Ion Monitoring Mode

TBD -- To Be Determined

Table A IV
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>	Determ.	HPLC	25 ppb 50 ppb 12.5 ppb 50 ppb 50 ppb 50 ppb 50 ppb 25 ppb
	Enrofloxacin			
	Ciprofloxacin			
	Desethylene ciprofloxacin			
	Sarafloxacin			
	Danofloxacin			
	Difloxacin			
	Marbofloxacin			
	Orbifloxacin			
	Tilmicosin	Determ.	HPLC- Ion Pairing	Muscle 300 ppb Liver and Kidney 600 ppb
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 0.08 ppm 0.1 ppm 0.25 ppm 0.05 ppm 0.15 ppm 0.01 ppm 0.25 ppm 10 ppm 0.2 ppm	
Penicillin				
Chlortetracycline				
Tetracycline or Oxytetracycline				
Streptomycin				
Neomycin				
Erythromycin				
Gentamicin				
Ampicillin				
Novobiocin				
Spectinomycin				
Tylosin				
Arsenicals	Arsenicals	Determ.	Atomic Absorption Spectrophotometry	
Avermectins	Ivermectin Doramectin Moxidectin	Determ.	HPLC	2.0 ppb
		Confirm.	APCI-LC-MS	25 ppb
Beta Agonists	Ractopamine	Determ.	HPLC	
		Confirm.	LC/MS	25ppb
	Clenbuterol	Screen	ELISA	TBD
		Confirm.	LC-MS-MS	

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Compound Class	Compound	Method Type	Methodology	Method Detection Limit
Chlorinated Hydrocarbons/ Chlorinated Organophosphates/ Polychlorinated Biphenyls	<u>Organohalides:</u>			
	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			0.01 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	Determ.	GPC with GC-ECD	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	
CB 1242			0.30 ppm	
PCB 1248			0.30 ppm	
PCB1254			0.30 ppm	
PCB 1260			0.30 ppm	
	Confirm.	GC-MS	NA	
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

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Compound Class	Compound	Method Type	Methodology	Method Detection Limit
Sulfonamides	Sulfapyridine	Determ.	TLC	0.05 ppm
	Sulfadiazine			
	Sulfathiazole			
	Sulfamerazine			
	Sulfamethazine			
	Sulfachloropyridazine	Confirm.	GC-MS	NA
	Sulfamethoxypryridazine			
	Sulfaquinoxaline			
	Sulfadimethoxine			
	Sulfaethoxypyridazine			
	Sulfaphenazole			
	Sulfatroxazole			
	Sulfisoxazole			
	Sulfadoxine			