APPENDIX V

FSIS LABORATORY RESIDUE ANALYTICAL CAPABILITY

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INTRODUCTION

The Food Safety and Inspection Service (FSIS) requires practical analytical methods for detecting, quantifying, and identify residues that may be present in meat, poultry, and their processed products. These methods can be used by the Agency for monitoring and surveillance activities to determine whether product is adulterated and for human risk assessment (exposure) purposes. The Agency uses available methodology to take appropriate regulatory action against adulterated products, consistent with the reliability of the analytical data. However, because of the large number of potential residues that may occur in the food chain, practical methods are not available for many compounds of interest. This section describes the types of methods used by FSIS to conduct analyses and their suitability for regulatory use. A list of key terms precedes the method descriptions. Note that the chemistry method descriptions with few exceptions, are referenced to the latest edition of the FSIS Analytical Chemistry Laboratory Guidebook.

CRITERIA FOR PRACTICAL METHODS

The following criteria have been identified as primary concerns for methods suitable for regulatory use.

1. The method requires no more than 2-4 hours of analytical time per sample.

2. A quality assurance plan has been developed for the method.

KEY TO ABBREVIATIONS

AAS -- Atomic Absorption Spectrometry

AOAC -- Association of Official Analytical Chemists International

CELIA, CA -- Competitive Enzyme Labeled Immunoassay for Chloramphenicol: a laboratory test that detects and identifies chloramphenicol residues in cattle and pork muscle

ECD -- Electron capture detector

EI -- Electron impact

ELISA -- Enzyme-Linked Immuno Sorbent Assay

E-Z SCREEN -- A proprietary immunoassay system for rapid detecting and identifying various antibiotics and other residues in tissue extracts

GC -- Gas chromatography

GLC -- Gas liquid chromatography (same as GC)

GPC -- Gel Permeation Chromatography

HFAA -- Heptaflourobutyric acid anhydride

HPLC -- High pressure liquid chromatography

ICP-- Inductively Coupled Plasma Spectrophotometry

JAOAC -- Journal of the Association of Official Analytical Chemists

LDL -- Lowest detectable limit. The smallest amount of individual residue or sample component that can be reliably observed or found in the sample matrix by the current appropriate methodology. Qualitative number. Not applicable to compounds with established tolerances.

METHOD STATUS -- See discussion above

MIC -- Minimum inhibitory concentration: the minimum amount of antimicrobial compound present in a buffer extract of tissue that will inhibit bacterial growth in a cell culture media

MPL -- Minimum proficiency level: the minimum amount of analyte expected to be identified and quantified by a laboratory and upon which ongoing capability will be evaluated. It is the smallest concentration for which the predicted coefficient of variation for reproducibility (CV) is less than or equal to 20 percent and the upper 90 percent confidence level for the predicted CV is less than 30 percent

MS -- Mass spectrometry

NADA -- New Animal Drug Application, issued by the Center for Veterinary Medicine Food and Drug Administration (CVM-FDA)

NE -- Level not established

NICI -- Negative ion chemical ionization

PICI -- Positive ion chemical ionization

PP -- Processed product

QUANTIFICATION -- The determination of the amount of residue present in a sample

ppb -- Parts per billion

ppm -- Parts per million

QAP—Quality Assurance Plan

REFERENCE METHODS -- Analytical procedures by which other methods may be evaluated and for which standards are established. These methods are considered suitable for regulatory use in the National Residue Program

RESIDUE -- Any compound present in edible or target tissues of the animal that results from that compound's use or inadvertent introduction into the animal. "Residue" includes the compound itself, its metabolite, and other substances formed in or on food because of the compound's use or inadvertent

SOS -- Sulfa-on-Site: a rapid in-plant chemical screening test for detecting sulfonamide residues in food animal urine or serum that provides same-day results

STOP -- Swab Test on Premises: an overnight in-plant laboratory microbiological screen test for detecting antibiotic residues in edible tissues

SWAB -- STOP precursor: an overnight laboratory microbiological screen test for detecting antibiotic residues in edibles tissues

TLC -- Thin layer chromatography

UV -- Ultraviolet spectrophotometric techniques for detection and quantification

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Albendazole (aminosulfone metabolite)	Marker residue detected and quantified by HPLC-fluorescence detection	20 ppb	50 ppb	Cattle/liver	1
	Extraction with organic solvents followed by HPLC with UV detection; confirmed by GC/EI/MS	0.05 ppm	NE	Red meat, liver, muscle	1
Aldicarb and Metabolites	GPC plus HPLC with post- column fluorescence detection; extracts verified by oxidation to the sulfone	5 ppb	10 ppb	All/liver	1
Aldrin	GPC plus GLC	0.02 ppm	0.1 ppm	All/fat	1
	Extracts from GPC or Mills confirmed by GC/MS.	0.03 ppm	NE	All/fat, PP	1
Amoxicillin Trihydrate	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	0.02 ppm	0.02 ppm	Cattle, swine/ kidney, liver, muscle	2
	Tissue extracts quantified by HPLC using fluorometer	0.01 ppm	0.01 ppm	Cattle, swine/ kidney,liver, muscle	1
Ampicillin Trihydrate	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	0.01 ppm	0.01 ppm	Cattle, swine/all	3
Apramycin	Sample extraction TLC; bioautographed using *Bacillus subtilis* as a test organism	0.05 ppm	0.1 ppm	Swine/kidney, muscle	1
Atrazine	Fat extracted using C18 columns and quantified by capillary GC with nitrogen/ phosphorous detector	5 ppb	NE	All/fat	1
	Extracts confirmed by GC/MS	5 ppb	NE	All/fat	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Bacitracin methylene disalicylate Bacitracin, zinc	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	0.05 ppm	NE	All/kidney, liver, muscle	4
Bambermycins	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	25 ppb	NE	All/kidney, liver, muscle	5
Bendiocarb	GPC plus HPLC with post-column fluorescence detection.	5 ppb	10 ppb	All/liver	1
Benomyl (Benzimidazole)	pH extraction with organic solvents; followed by HPLC ppm with UV detection; extracts derivatized and confirmed by GC/EI/MS	0.05 ppm	50 ppb	Poultry/liver, muscle	1
	Micro alumina assay: column chromatography plus GLC	0.01 ppm	NE	All/fat, PP	1
BHC	GPC plus GLC	0.01 ppm	0.1 ppm	All/fat	1
	Beta and delta isomers: GPC plus GLC	Identifi-cation only	Identifi-cation only	All/fat	1
	Extracts from GPC or Mills confirmed by GC/MS	0.02 ppm	NE	All/fat, PP	1
Bufencarb	GPC plus HPLC with post- column fluorescence detection. Extracts are subjected to reverse phase chromatography, derivatized and confirmed by GC/MS	5 ppb	10 ppb	All/liver	1
Cacodylic acid	Dry ashed tissue is dissolved and reacted to produce arsine gas, which reacts to form a blue complex for colorimetric quantification	0.05 ppm	0.20 ppm	All/kidney, liver, muscle	6

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Cadmium	Dry ashed tissue is dissolved and quantified by ICP		0.002 :g/ml	All/kidney, liver, muscle	1
Calcium	Tissue is wet ashed and titrated with specific indicator	0.03%	0.03%	All/muscle	8
	Wet ashed tissue is quantified by AAS	NE	NE	All	
Cambendazole (Benzimidazol)	Extraction with organic solvents followed by HPLC with UV detection; extracts confirmed by GC/EI/MS	0.05 ppm	50 ppb	Red meat/liver, muscle, PP	1
Captan	GPC plus GLC	0.02 ppm	0.05 ppm	Red meat/fat	1
Carbadox	Tissue extract is hydrolyzed and a derivative is prepared and separated by ion exclusion chromatography, then quantified by GC-ECD	7.5 ppb	30 ppb	Swine/liver	1
	Extracts confirmed by GC/EI/MS	7.5 ppb	NE	Swine/liver, muscle	1
Carbarsone	Dry ashed tissue is dissolved and reacted to produce arsine gas, which reacts to form blue complex for colorimetric quantification	0.05 ppm	0.20 ppm	All/kidney, liver, muscle	6
Carbaryl	GPC plus HPLC with post-column fluorescence detection	5 ppb	10 ppb	All/liver	1
	Extracts are subjected to reverse phase chromatography, derivatized and confirmed by GC/MS	10 ppb	NE		

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Carbofuran and metabolite	GPC plus HPLC with post-column fluorescence detection.	5 ppb	10 ppb	All/liver	1
	Extracts are subjected to reverse phase chromatography, derivatized and confirmed by GC/MS	10 ppb	NE		
Carbophenothion	Tissue extracts are quantified by GLC with flame photometric or nitrogen-phosphorous flame ionization detector	0.10 ppm	NE	All/liver, muscle	
	GPC plus GLC	0.03 ppm	0.20 ppm	All/fat	1
	GPC extracts are confirmed by CG/EI/MS	0.01 ppm	NE	Red meat/fat	1
Chloramphenicol	Tissue extracts are screened by E-Z screen	25 ppb	NE	Calf/muscle, kidney	9
	Tissue extract screened for chloramphenicol by CELIA CA	5 ppb	NE	Calf/muscle	10
	Extraction of parent and glucuronide using C18 columns with GC capillary quantification as the trimethylsilyl derivative	0.5 ppb	1.0 ppb	Calf/muscle	1
	Extracts are confirmed using NICI/MS	0.15 ppb	NE	Calf/muscle	1
Chloramphenicol (continued)	C18 cleanup of the hydrolyzed extract with GC capillary quantification as the trimethyl derivative	2.5 ppb	5.0 ppb	Calf/urine	1
	Extracts are confirmed using NICI/MS	5 ppb	NE	Calf/urine	1
Chlordane	GPC followed by GC-ECD	0.10 ppm	0.30 ppm	All/fat	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
	Extracts from GPC or Mills are confirmed by GC/MS	NE	NE	All/fat, PP	1
Chlordecone (Kepone)	GPC plus GLC	0.03 ppm	0.20 ppm	All/fat	1
	GPC extracts are confirmed by GC/EI/MS	0.05 ppm (poultry) 0.20 ppm (cattle)	NE	Poultry, red meat/fat	1
2-Chloro-1-(2,4,-di chlorophenyl) vinyl diethyl phosphate (chlorfenvinphos)	GPC plus GLC	0.03 ppm	0.10 ppm	All/fat	1
	GPC extracts are confirmed by CG/EI/MS	0.01 ppm (poultry) 0.10 ppm (red meat)	NE NE	Poultry, red meat/fat	1
2-Chloro-1-(2,4,5-trivinyl dimethyl phosphate (stirophos)	GPC plus GLC	0.05 ppm	0.30 ppm	All/fat	1
	GPC extracts are confirmed by GC/EI/MS	0.05 ppm (poultry) 0.10 ppm (cattle)	NE	Poultry, red meat/fat	1
Chlorpyrifos	GPC followed by GC-ECD	0.05 ppm	0.20 ppm	All/fat	1
	GPC extracts are confirmed by GC/EI/MS	0.05 ppm (poultry) 0.50 ppm (swine)	NE	Poultry, swine/fat	1
Chlortetracycline	Antibiotic screen test (Swab): ability of tissue fluids containing anti-microbial activity to inhibit microbial growth	0.01 ppm	NE	All/kidney	11
Chlortetracycline (contiued)	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	0.01 ppm	NE	All/kidney, liver, muscle	12
	Extraction using C18 columns followed by HPLC with UV detection	0.05 ppm	0.10 ppm	All/kidney, liver, muscle	1 (Guide-book draft)

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Chromium	Dry ashed tissue is extracted with organic reagent and quantified using ICP	0.01 mg/l	0.037 mg/l	All/kidney, liver, muscle	1
Clenbuterol	Liquid-liquid extraction with internal standard, detected by GC/MS of the oxazolidin-3-one derivatives.		1.0 ppb	Cattle, swine, sheep/liver, muscle.	1 (9/95)
Clopidol	Organic solvent extraction with HPLC-UV detection	0.1 ppm	NE	Poultry/liver	1
	Organic solvent extraction with GC-EC detection	0.1 ppm	NE	Poultry/liver	1
Clorsulon	Tissue extracts are quantified by HPLC-UV detection	0.25 ppm	0.50 ppm	Red meat/kidney, muscle, liver, PP	1
	Tissue extracts for HPLC are derivatized and confirmed by GC/MS	0.5 ppm	NE	Red meat/kidney, muscle, liver, PP	1
Cloxacillin	Antibiotic screen test(Swab): ability of tissue fluids containing anti- microbial activity to inhibit microbial growth	0.16 ppm	NE	All/kidney	14
	Microbiological assay combined with HPLC separation and quantified by microbial inhibition	0.02 ppm	NE	Dairy cows/ kidney, liver, muscle	15
Cobalt	Dry ashed tissue is dissolved and quantified by ICP	0.009 mg/l	0.03 mg/l	All/kidney, liver, muscle	
Copper	Dry ashed tissue is dissolved and quantified by ICP	0.006 mg/l	0.02 mg/l	All/kidney, liver, muscle	1
Coumaphos and oxygen analog	GPC followed by GC-ECD	0.15 ppm	0.30 ppm	All/fat	1
	GPC extracts are confirmed by CG/EI/MS	0.20 ppm	0.30 ppm	Red meat/fat	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Cresylic acid	Tissue extracts are derivatized and determined by GC-ECD	NE	NE	Poultry/fat	1
Crufomate (Ruelene)	Tissue extracts are quantified by GLC with flame photometric or nitrogen-phosphorous flame ionization detector	0.10 ppm	NE	All/liver, muscle	1
Cyanide salts	Aqueous extraction followed by a colori-metric determination	0.5 ppm	NE	All/all	1
	For confirmation, cyanogen chloride is produced and determined by GC/ECD	0.5 ppm	NE	All/all	1
Cyano (3-phenoxy chlorophenyl) methyl- 4-a-(methylethyl) - benzeneacetate (Fenvalerate)	Organic solvent extracts are quantified as the sum of both isomers by GC/EC; extracts are confirmed by GC/EI/MS	0.03 ppb	NE	All/fat	1
DDE (metabolites of DDT collectively reported as DDT)	GPC followed by GC-ECD	0.02 ppm	0.05 ppm	All/fat	1
	Extracts from GPC are confirmed by GC/MS (LRC)	0.02 ppm	NE	All/fat, PP	1
DDT (isomers of DDT collectively reported as DDT)	GPC followed by GC-ECD	0.04 ppm	0.15 ppm	All/fat	1
Decoquinate	Zymark Pytech System; organic extraction followed by HPLC with fluorescence detection	0.20 ppm	0.50 ppm	Cattle, poultry/ liver, muscle	1
Dexamethasone	Extract is partitioned on an SPE column with quantitation by HPLC/UV		5.0 ppb	Cattle, swine, veal, sheep/liver, muscle	1 (guide-book draft)

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Deltamethrin	Organic solvent extracts are quantified by GC/ECD;		20 ppb	Bovine, poultry/fat	1
	Solvent extraction followed by a competitive ELISA determination	0.5 ppm	NE	All/fat	1
Diazinon	Tissue extracts are quantified by GLC with flame photometric or nitrogen-phosphorous flame ionization detector	0.1 ppm	0.15 ppm	All/liver, muscle	1
Dibutyltin dilaurate	Tissue extraction acid hydrolysis-morin derivatization--HPLC-UV	0.25 ppm	NE	Turkey/liver	1
Dieldrin	GPC plus GLC	0.01 ppm	0.05 ppm	All/fat	1
	Extracts from GPC are confirmed by GC/MS	0.02 ppm	NE	All/fat, PP	1
Diethylstilbestrol (DES)	Solid phase extraction technique using an internal standard followed by methylsilation for GC/MS quantification and confirmation		0.25 ppb	Cattle, sheep/liver, muscle	1
Dihydro-streptomycin	Antibiotic screen test (Swab): ability of tissue fluids containing anti-microbial activity to inhibit microbial growth	0.25 ppm	NE	All/kidney	11
	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	0.25 ppm	NE	All/kidney, liver, muscle	16

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
3, 5-Dimethyl-4-(methylthio)phenyl ethylcarbamate and metabolite	GPC plus HPLC with post- column fluorescence detection. Extracts are subjected to reverse phase chromatography, derivatized and confirmed by GC/MS	5 ppb	10 ppb	All/liver	1
Dimetridazole and hydroxy metabolite	Extracts are quantified by HPLC/UV	1.0 ppb	NE	Turkey, swine/ muscle	1
	Tissue extracts from HPLC are confirmed by GC/NICI/MS	1.0 ppb	NE	Turkey, swine/ muscle	1
Dioxacarb	GPC plus HPLC with post- column fluorescence detection	5 ppb	10 ppb	All/liver	1
	Extracts are subjected to reverse phase chromatography, derivatized and confirmed by GC/MS	10 ppb	NE		
Dioxathion	Tissue extracts are quantified by GLC with flame photometric or nitrogen-phosphorous flame ionization detector	0.10 ppm	NE	All/liver, muscle	
Dodecachloro-octahydro-1,3,4-metheno-2H-cyclobuta(cd)-pentalene[Mirex]	GPC plus GLC	0.04 ppm	0.10 ppm	All/fat	1
	Extracts from GPC or Mills are confirmed by GC/MS	0.05 ppm	NE	All/fat, PP	1
Doramectin	Tissue extracts are quantified by HPLC fluorescence detection	2 ppb	7.5 ppb	Red meat/liver, muscle	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Endosulfan I	GPC plus GLC	0.01 ppm	0.10 ppm	All/fat	1
	GPC extracts are confirmed by GC/EI/MS	0.02 ppm	NE	Red meat/fat	
Endosulfan II	GPC plus GLC	0.02 ppm	0.20 ppm	All/fat	1
Endrin	GPC plus GLC	0.03 ppm	0.05 ppm	All/fat	1
	Extracts from GPC or Mills are confirmed by GC/MS	0.05 ppm	NE	All/fat, PP	1
Erythromycin	Antibiotic screen test (Swab): ability of tissue fluids containing antimicrobial activity to inhibit microbial growth	25 ppb	NE	All/kidney	11
	Microbiological assay procedure: ability of tissue fluids containing antimicrobial activity to inhibit microbial growth	25 ppb	NE	All/kidney, liver, muscle	17
Ethion and oxygen analog	Tissue extracts are quantified by GLC with flame photometric or nitrogen-phosphorous flame ionization detector	0.10 ppm	0.15 ppm	All/liver, muscle	1
	Tissue extracts are quantified by GLC with flame photometric or nitrogen-phosphorous flame ionization detector	NE	NE	All/liver muscle	1
Ethylene dibromide	Residue is co-distilled from aqueous suspension and quantified by GLC	0.5 ppb	1.0 ppb	All/fat	1
	MS by NICI to determine bromine	1 ppb	NE	All/fat	1
Fenbendazole	Extraction with organic solvents followed by HPLC with UV detection; extracts derivatized and confirmed by GC/EI/MS		0.5 ppb	All/ liver, muscle	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Fenbendazole (continued)	Tissue extracts are quantified by HPLC	200 ppb	400 ppb	Cattle, calf/ liver	18
	Quantification extract purified by TLC, derivatized and identified by HPLC fluorescence	200 ppb	NE	Cattle, calf/ liver	18
Fenitrothion	Tissue extracts are quantified by GLC with flame photometric or nitrogen-phosphorous flame ionization detector	0.10 ppm	0.15 ppm	All/liver, muscle	1
Flucythrinate	Organic solvent extracts are quantified as the sum of both isomers by GC/EC; extracts are confirmed by GC/EI/MS			All/fat	1
	Solvent extraction followed by a competitive ELISA determination		NE	All/fat	1
Gasoline	Fat from product is heated in a sealed vial and gasoline components are identified by pattern recognition using GC/flame ionization detection	0.1 ppm	1.0 ppm	Canned meat	1
Gentamicin sulfate	Tissue extracts are screened by E-Z Screen	50 ppb	NE	All/muscle, liver, kidney	19
	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	NE	NE	Swine/kidney	20
	Extraction followed by detection by HPLC with fluorescence detector	0.2 ppm	0.4 ppm	Swine/kidney	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Halofuginone	Tissue extracts are quantified by HPLC-UV	0.05 ppm	0.05 ppm	Chicken/liver	1
	Tissue extracts are confirmed by GC/MS/MS	0.05 ppm	0.05 ppm	Chicken/liver	21
HCB	GPC plus GLC	0.01 ppm	0.01 ppm	All/fat	1
	Extracts from GPC are confirmed by GC/ MS	0.01 ppm	NE	All/fat, PP	1
Heptachlor and heptachlor epoxide	GPC plus GLC	0.01 ppm	0.05 ppm	All/fat	1
	Extracts from GPC are confirmed by GC/MS		0.01 ppm, Heptachlor; 0.1 ppm, Heptachlor Epoxide	All/fat, PP	1
Hetacillin, Potassium	Antibiotic screen test (Swab): ability of tissue fluids containing anti-microbial activity to inhibit microbial growth	NE	NE	All/kidney	14
5-Hydroxy-thiabendazole	Extraction with organic solvents followed by HPLC with UV detection; extracts confirmed by derivatized GC/EI/MS	0.05 ppm	50 ppb	Red meat/liver, muscle	1
Hygromycin B	Antibiotic screen test (Swab): ability of tissue fluids containing antimicrobial activity to inhibit microbial growth	5.00 ppm	NE	All/kidney	14
Ipronidazole and hydroxy metabolite	Tissue extracts are quantified by HPLC/UV	1.0 ppb	NE	Turkey, swine/ muscle	1
	Tissue extracts from HPLC are confirmed by GC/NICI/MS	1.0 ppb	NE	Turkey, swine/ muscle	1
Iron	Dry ashed tissue is dissolved and quantified by ICP	0.009 mg/l	0.14 μg/ml	All/kidney, liver, muscle	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Ivermectin	Tissue extracts are quantified by HPLC fluorescence	2 ppb	7.5 ppb	Red meat/liver, muscle	1
	APCI/MS to confirm Ivermectin and Doramectin	7.5 ppb	15 ppb	Red meat/liver, muscle	1
Lasalocid	Tissue extracts are quantified by HPLC fluorescence detector	0.025 ppm	0.35 ppm	Cattle/liver Poultry/fat, skin	1 22
	Tissue extraction followed by bioautography	0.005 ppm	0.01 ppm	Poultry/fat, skin	22
	GC pyrolysis of the HPLC extract with MS identification of the fragments	0.2 ppm	NE	Cattle/liver Poultry/fat, skin	22
Lead	Dry ashed tissue is dissolved and quantified by ICP		0.05 μg/ml	All/kidney, liver, muscle	1
	Dry ashed tissue is quantified by anodic stripping voltammetry	1.0 ppb	NE	Poultry/kidney, liver	7
Levamisole	Tissue extracts are quantified by GLC flame photometric detection	0.05 ppm	0.1 ppm	Red meat/liver, muscle	1
	Tissue extracts are subjected to GC/MS	0.05 ppm	NE	Red meat/liver, muscle	1
Lincomycin hydrochloride	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	0.10 ppm	0.10 ppm	Poultry, swine/all	23
Lindane	GPC plus GLC	0.01 ppm	0.01 ppm	All/fat	1
	Extracts from GPC or Mills are confirmed by GC/MS		0.01 ppm	All/fat, PP	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Linuron	GPC plus GLC	0.25 ppm	0.50 ppm	All/fat	1
	Extracts are confirmed by GC/EI/MS	0.05 ppm	0.50 ppm	Red meat/fat	1
Lysergic acid diethylamide	Tissue extracts are spotted for TLC and detected with specific chromagenic reagent	NE	NE	All/kidney, liver, muscle	1
Macrolide antibiotics	Qualitative identification by APCI-MS/MS full Scan			All/kidney, liver, muscle	1
Malathion	Tissue extracts were quantified by GLC with flame photometric or nitrogen-phosphorous flame ionization detector	0.10 ppm	0.2 ppm	All/liver, muscle	1
Manganese	Dry ashed tissue is dissolved and quantified by ICP		0.002 μg/ml	All/kidney, liver, muscle	1
Mebendazole	Extraction with organic solvents followed by HPLC with UV detection; extracts are confirmed by GC/EI/MS	0.05 ppm	50 ppb	Red meat/liver, muscle, processed products	1
Melengestrol acetate (MGA)	Tissue extract is column chromatographed on Florisil and quantified by GLC	5.0 ppb	10.0 ppb	Cattle/fat	1
	Extracts are derivatized with HFB and confirmed by GC/EI/MS	5.0 ppb	10.0 ppb	Cattle/fat	1
Mercury	Tissue is digested in acid. Mercury is reduced to its vapor and quantified by flameless AAS	0.01 ppm	0.02 ppm	All/kidney, liver, muscle	1
Methanearsonic acid	Dry ashed tissue is dissolved and reacted to produce arsine gas, which is quantified by AAS	0.05 ppm	NE	All/kidney, liver, muscle	1
	The same as above, but arsine gas reacts to form blue complex for colori - metric quantification	0.05 ppm	0.20 ppm	All/kidney, liver, muscle	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Methomyl	GPC plus HPLC with post-column fluorescence detection	5 ppb	10 ppb	All/liver	1
Methoxychlor	GPC plus GLC	0.15 ppm	0.50 ppm	All/fat	1
	Extracts from GPC are confirmed by GC/MS	0.15 ppm	0.15 ppm	All/fat,	1
Methyl parathion	Tissue extracts are quantified by GLC with flame photometric or nitrogen-phosphorous flame ionization detector	0.10 ppm	0.20 ppm	All/liver, muscle	1
Monensin	Tissue extract is partitioned by TLC and semi-quantified by inhibition of micro-organism growth		NE	Poultry/fat	1
Morantel tartrate	Tissue extract is hydrolyzed and a derivative is quantified by GLC	0.25 ppm 0.50 ppm	0.50 ppm 0.50 ppm	Cattle/liver Cattle/muscle	1 1
	Identification of a structurally significant hydrolyzed fragment by GC/MS	0. 25 ppm	NE	Cattle/liver, muscle	24
Narasin	Tissue extracts are spotted on TLC and quantified with a bio-autographic overlay		NE	Cattle, poultry/liver, kidney, fat	1
Neomycin	Antibiotic screen test (Swab): ability of tissue fluids containing anti-microbial activity to inhibit microbial growth	0.25 ppm	NE	All/kidney	14
	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	0.25	NE	All/kidney, liver, muscle	25

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Nequinate	Zymark Pytechnology System: Tissue extracts are screened by HPLC/UV	NE	NE	Cattle/liver, muscle	13
Nicarbazin	Tissues are extracted with ethyl acetate; the dinitro-carbanilide moiety is quantified by HPLC-UV. Extracts verified by photodiode array detection	0.1 ppm	0.4 ppm	Chicken/liver muscle	1
Nickel	Dry ashed tissue is dissolved and quantified by ICP	0.015 mg/l	0.014 μg/ml	All/kidney, liver, muscle	1
Nonachlor	GPC plus GLC	0.03 ppm	0.15 ppm	All/fat	1
	Extracts from GPC are confirmed by GC/MS	0.05 ppm	NE	All/fat, PP	
Novobiocin	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	0.125 ppm	NE	All/kidney, liver, muscle	4
	Zymark Pytechnology System: organic solvent extraction followed by HPLC/UV detection.	0.50 ppm	NE	All/kidney, liver, muscle	1
	Manual system organic solvent extraction followed by HPLC/UV detection	0.50 ppm	1.0 ppm	All/kidney, liver, muscle	1
Oleandomycin	Antibiotic screen test (Swab): ability of tissue fluids containing antimicrobial activity to inhibit microbial growth	0.25 ppm	NE	All/kidney	14

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Oxfendazole	Extraction with organic solvents followed by HPLC with UV detection; extracts are derivatized and confirmed by GC/EI/MS	0.05 ppm	50 ppb	Red meat/liver, muscle, PP	1
Oxytetracycline hydrochloride	Antibiotic screen test (Swab): ability of tissue fluids containing anti-microbial activity to inhibit microbial growth	0.08 ppm	NE	All/kidney	14
	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	0.08 ppm	0.08 ppm	All/kidney, liver muscle	14
	Extraction using C18 columns followed by HPLC with UV detection	0.05 ppm	0.10 ppm	All/kidney, liver muscle	1 (Guide-book draft)
Parathion	Tissue extracts are quantified by GLC with flame photometric or nitrogen-phosphorous flame ionization detector	NE	NE	All/liver, muscle	
PBB's	GPC cleanup followed by GLC/ECD and MS/EI	0.05 ppm	NE	All/fat	26
PCB's (reported as Aroclor 1242, 1248, 1254, 1260, etc.)	Column chromatography plus GLC.	0.30 ppm	0.50 ppm	All/fat, PP	1
	GPC plus GLC	0.30 ppm	0.50 ppm	All/fat	
Penicillin, procaine and procaine G	Antibiotic screen test (Swab): ability of tissue containing anti-microbial activity to inhibit microbial growth.	12.5 ppb	NE	All/kidney	14

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Pentachloroanisole	GPC plus GC	NE	NE	Poultry/fat	
	Extracts confirmed by GC/EI/MS	NE	NE	Poultry/fat	
Pentachlorophenol (PCP)	Tissue extracts for GLC are confirmed by GC/MS.	0.03 ppm	50 ppb	All/liver, muscle	1
	Extracts confirmed by GC/EI/MS	NE	NE	Poultry/fat	1
Permethrin (cis & trans)	Solvent extraction followed by a competitive ELISA determination.	1.0 ppb	50 ppb	All/fat	1
	Organic solvent extracts are quantified as the sum of both isomers; extracts are confirmed by GC/EI/MS	1.0 ppb	50 ppb	All/fat	1
Phencyclidine	Tissue extracts are spotted for TLC with specific chromogenic agent	NE	NE	All/kidney, liver, muscle	1
Phenothrin	Solvent extraction followed by a competitive ELISA determination		NE	All/fat	1
Phosalone	GPC plus GLC	0.01 ppm	0.05 ppm	All/fat	1
	GPC extracts are confirmed by GC/EI/MS	0.02 ppm	0.04 ppm	Red meat/fat	1
Propazine	Fat extracted using C18 columns and quantified by capillary GC detector with nitrogen-phosphorous flame ionization detector.	5 ppb	10 ppb	All/fat	1
	Extracts confirmed by GC/MS	5 ppb	10 ppb	All/fat	1
Promecarb	GPC plus HPLC with post- column fluorescence detection. Extracts are subjected to reverse phase chromatography, derivatized and confirmed by GC/MS	5 ppb	10 ppb	All/liver	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Propoxur	GPC plus HPLC with post- column fluorescence detection. Extracts are subjected to reverse phase chromatography, derivatitized and confirmed by GC/MS	5 ppb	10 ppb	All/liver	1
Pyrantel tartrate	Tissue extract is hydrolyzed and a derivative is quantified by GLC	0.25 ppm	0.50 ppm	Swine/liver, muscle	1
	Identification of a structurally significant hydrolyzed fragment by GC/MS	0.25 ppm	NE	Swine/liver, muscle	27
Ronnel	GPC plus GLC	0.02 ppm	0.05 ppm	All/fat	1
	Extracts are confirmed by GC/EI/MS	0.01 ppm (poultry) 0.10 ppm (red meat)	0.05 ppm 0.05 ppm	Poultry, red meat/fat	1
Roxarsone	Dry ashed tissue is dissolved and reacted to produce arsine gas, which is quantified by AAS.	0.05 ppm	NE	All/kidney, liver muscle	1
	Dry ashed tissue is dissolved and reacted to produce arsine gas, which reacts to form blue complex for colorimetric quantification	0.05 ppm	0.20 ppm	All/kidney, liver, muscle	1
Simazine	Fat extracted using C18 columns and quantified by capillary GC with nitrogen-phosphorous detector.	5 ppb	10 ppb	All/fat	1
	Extracts confirmed by GC/MS	5 ppb	NE	All/fat	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Simazine	Fat extracted using C18 columns and quantified by capillary GC with nitrogen-phosphorous detector.	5 ppb	10 ppb	All/fat	1
	Extracts confirmed by GC/MS	5 ppb	NE	All/fat	1
Spectinomycin hydrochloride	Microbiological assay: tissue extracts are quantified using a turbidimetric assay.	2.8 ppm	NE	All/kidney, liver, muscle	28
Streptomycin	Antibiotic screen test (Swab): ability of tissue fluids containing antimicrobial activity to inhibit microbial growth	0.25 ppm	NE	All/kidney	14
	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	0.25 ppm	NE	All/kidney, liver, muscle	30
Styrene	Tissues are subjected to GC/MS head space analysis.		NE	All/kidney, liver, muscle fat, PP	1
Sulfachloro-pyridazine	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry	0.02 ppm	0.05 ppm	Red meat/liver, muscle	1
Sulfadiazine	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry	0.02 ppm	0.05 ppm	Red meat/liver, muscle	1
	Extraction followed by GC/CI and EI/MS	0.05 ppm	0.05 ppm	Red meat/liver, muscle	1
Sulfadimethoxine	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry	0.02 ppm	0.05 ppm	All/liver, muscle	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Sulfadoxine	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry.	0.05 ppm	0.05 ppm	Red meat/liver, muscle	1
	Tissue extracts are confirmed by GC/EI/MS	NE	0.05 ppm	Red meat/liver, muscle	1
Sulfaethoxy-pyridazine	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry	0.02 ppm	0.05 ppm	Red meat/liver, muscle	1
Sulfamethazine	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry.	0.02 ppm	0.05 ppm	All/liver, muscle	1
	Tissue extracts are confirmed by GC/EI/MS	0.05 ppm	0.05 ppm	All/liver, muscle	1
	Tissue extracts are screened by E-Z Screen	50 ppb	NE	All/liver	1
Sulfamethoxy-pyridazine	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry	0.02 ppm	0.05 ppm	Red meat/liver, muscle	1
Sulfaphenazole	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry	0.05 ppm	0.05 ppm	Red meat/liver, muscle	1
	Tissue extracts are confirmed by GC/EI/MS	NE	0.05 ppm	Red meat/liver, muscle	1
Sulfapyridine	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry.	0.02 ppm	0.05 ppm	All/liver, muscle	1
	Tissue extracts are confirmed by GC/EI/MS	0.05	0.05 ppm	All/liver, muscle, PP	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Sulfaquinoxaline	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry.	12.5 ppb	0.05 ppm	Poultry/liver, muscle	1
	Tissue extracts are confirmed by GC/EI/MS	25 ppb	0.05 ppm	Poultry/liver, muscle	1
Sulfathiazole	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry	0.02 ppm	0.05 ppm	Red meat/liver, muscle	1
	Tissue extracts are confirmed by GC/EI/MS	0.05 ppm	0.05 ppm	Red meat/liver, muscle, PP	1
Sulfatroxazole	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry.	0.05 ppm	0.05 ppm	Red meat/liver, muscle	1
	Tissue extracts are confirmed by GC/EI/MS	NE	0.05 ppm	Red meat/liver, muscle	1
Sulfisoxazole	TLC fluorescence: tissue extracts are partitioned by TLC and quantified by densitometry.	0.05 ppm	0.05 ppm	Red meat/liver, muscle	1
	Tissue extracts are confirmed by GC/EI/MS	NE	0.05 ppm	Red meat/liver, muscle	1
TDE (metabolite of DDT reported as DDT)	GPC plus GLC	0.03 ppm	0.15 ppm	All/fat	1
	Extracts from GPC are confirmed by GC/MS	0.02 ppm	0.04 ppm	All/fat, PP	1
Terbuthylazine	Fat extracted using C18 columns and quantified by capillary GC with nitrogen-phosphorous detector Extracts confirmed by GC/MS	5 ppb	10 ppb	All/fat	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Terpene polychlorinates (Strobane)	Micro alumina assay: column chromatography plus GLC	0.50 ppm	1.0 ppm	All/fat	1
	Mills method: Florisil column chromatography plus GLC	0.50 ppm	1.0 ppm	All/fat	1
Tetracycline hydrochloride	Antibiotic screen test (Swab): ability of tissue chloride fluids containing antimicrobial activity to inhibit microbial growth	0.08 ppm	NE	All/kidney	14
	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth.	0.08 ppm	NE	All/kidney, liver, muscle	29
Tetracycline hydrochloride	Extraction using C18 columns followed by HPLC with UV detection.	0.05 ppm	0.10 ppm	All/kidney, liver, muscle	1 (Guide-book draft)
Thiabendazole	pH extraction with organic solvents followed by HPLC with UV detection; extracts derivatized and confirmed by GC/EI/MS	0.05 ppm	50 ppb	Red meat/PP, liver, muscle	1
Tiamulin	Organic solvent extraction followed by GC of the 8-hydroxymutilin metabolite.	0.2 ppm	NE	Swine/liver	30
	Extracts confirmed by GC/MS	NE	NE	Swine/liver	30

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Tilmicosin	Antibiotic screen test (SWAB): ability of tissue chloride fluids containing antimicrobial activity to inhibit microbial growth.	NE	NA	All/kidney	28
	Microbial assay procedure: ability of tissue extract containing antimicrobial activity to inhibit microbial growth.	NE	NA	All/kidney	29
	Liquid-liquid extraction with quantitation by ion pairing HPLC/UV		0.3 ppm M 0.6 ppm L	Beef/liver, kidney, muscle	1 (Guidebook draft)
Tin	Tissue is dry ashed and dissolved and quantified by AAS (used to screen for organotin compounds)	0.02 ppm	0.1 ppm	All/kidney, liver, muscle	1
Toxaphene	GPC plus GLC	0.50 ppm	1.0 ppm	All/fat	1
	Tissue extracts are screened by E-Z screen.	50 ppm	NE	All/muscle, liver, kidney	9
Tylosin	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	0.2 :g/mL	NA	Cattle/muscle	31
Virginiamycin	Microbiological assay procedure: ability of tissue extracts containing antimicrobial activity to inhibit microbial growth	0.64 ppm	NA	Swine/kidney, liver, muscle	31
	Organic solvent extraction followed by HPLC/UV quantification	0.1 ppm	0.2 ppm	All/kidney, liver, muscle	1

Compound	Method Description	LDL/MIC	MPL	Species/Tissues	Reference
Zeranol and metabolite taleranol	Extraction followed by radioimmunoassay		NE	Cattle/liver, muscle	1
	Solid phase extraction using an internal standard followed by polymethyl-silation for GC/MS quantification and confirmation		NE	Cattle, sheep/liver, muscle	1
Zinc	Dry ashed tissue is dissolved and quantified by ICP	0.006 mg/l	0.006 μg/ml	All/kidney, liver, muscle	1

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