

United States Department of Agriculture

Food Safety and Inspection Service

CLG-MEGA1.01

**Screening of Veterinary Drug and Pesticides
Residues by UHPLC-MS/MS**

This method describes the laboratory procedure to screen 107 veterinary drugs from 15 drug classes and 81 pesticides from 8 pesticide classes in 6 animal species (bovine, porcine, poultry, *Siluriformes*, caprine, and ovine) and 3 egg products (egg white, egg yolk, and whole egg).

Executive Summary

This multi-residue method is used to screen for 107 veterinary drug and 81 pesticide residues. These residues include 15 drug classes (analgesics/anti-inflammatories, avermectins, beta-agonists, beta-lactams, benzimidazoles, fluoroquinolones, general drugs, hormones, ionophores, macrolides, nitroimidazoles, phenicols, sulfas, tetracyclines, and tranquilizers) and 8 pesticide classes (carbamates, conazoles/triazoles, halogenated pesticides, neonicotinoids, organophosphates, general pesticides, pyrethroids, and triazines). The method is applicable for screening analysis in muscle tissues from 6 animal species (bovine, porcine, poultry, *Siluriformes*, caprine, and ovine) and 3 egg products (egg white, egg yolk, and whole egg). The method's key performance characteristics include:

- High-throughput screening of various classes of veterinary drug residues and pesticides
- Utilization of multiple instrument platforms to improve robustness and flexibility.

The range of minimum levels of applicability (MLA) or lowest levels at which an FSIS method has been successfully validated for a residue class in each matrix for this method are found in the table below. The MLAs for each matrix and individual analyte are found in Tables 33-36.

Analyte	Range of MLAs (ppb)	
	Muscle	Egg Products
Veterinary Drug Class		
Analgesics	5-20	5-50
Avermectins	7.5-25	7.5-100
Beta-Agonists	3-12	3
Beta-Lactams	5-50	5-200
Benzimidazoles	10-200	10-200
Fluoroquinolones	25-50	25-50
General Drugs	15-350	15-350
Hormones	12-50	12-50
Macrolides	25-1000	25-1000
Nitroimidazoles	1-200	1-50
Phenicols	3-150	3-150
Sulfas	50-200	50
Tetracyclines	25-1000	500-2000
Tranquilizers	1-20	1-20
Ionophores	2-20	2-20
Pesticide Class		
Carbamates	20-100	10-1000
Conazoles/Triazoles	5-15	5-15
Halogenated Pesticides	5-50	5-50
Neonicotinoids	5-20	5-20
Organophosphates	5-500	5-500
General Pesticides	5-500	5-500
Pyrethroids	7.5-500	7.5-500
Triazines	10-15	10-15

Notice of Change

An editorial revision was made to correct typographical errors that were found in the first version of the method. The revisions do not affect the purpose or procedure of the method. No revisions were made to the flow chart found in CLG-MEGA1 Appendix 1.

Table of Contents

Executive Summary	1
Notice of Change	2
Introduction	4
Materials and Reagents	6
Equipment	6
Instrumentation	7
Reagents	7
Reference Materials	8
Extraction and Analysis	12
Solution Preparation.....	12
Standard Preparation.....	14
Acetonitrile Mix.....	14
Beta-Lactam Mix	21
Pesticide Mix	23
Internal Standard Mix	28
Sample Preparation	29
MEGA Extraction	30
Instrumental Analysis	32
Reporting of Results	50
Decision Criteria	50
Minimum Level of Applicability	51
Safety Hazards	63
References	64
Contact Information and Inquiries	64

Safety Precautions

The personnel performing the analysis are to read the Safety Data Sheets for the standards and reagents used in this method. The hazards and recommended safe procedures for use are listed in Table 37. Follow all applicable federal, state, and local regulations regarding the disposal of chemicals listed in this method.

Introduction

Veterinary drugs are widely used in agriculture to raise healthy animals, treat diseases, and increase growth and feed efficiency. The Food and Drug Administration (FDA) through the [Federal Food, Drug, and Cosmetic Act](#) has the authority to approve and regulate the use of veterinary drugs. FDA establishes and publishes regulations setting tolerances for residues of veterinary drugs.

Pesticides are used in agriculture to prevent, mitigate, destroy, or repel pests. The Environmental Protection Agency (EPA) regulates the approval and use of pesticides under the [Federal Insecticide, Fungicide, and Rodenticide Act](#). The EPA establishes and publishes regulations setting tolerances for residues of pesticides.

The National Residue Program (NRP) is an interagency program that is designed to identify, prioritize, and analyze residues in meat, poultry, and egg products. The Food Safety and Inspection Service (FSIS) administers the NRP by collecting and testing samples of domestic and imported meat (including *Siluriformes* fish products), poultry, and egg products for veterinary drugs and pesticides to verify that these products are below tolerances and are safe, wholesome, and accurately labeled. FSIS publishes an [Annual Sampling Plan](#) to provide information on the process of sampling meat, poultry, and egg products for veterinary drugs of public health concern. The NRP is monitored and modified annually to set priorities based on data analyses that identify trends in detected residues.

Method Overview:

CLG-MEGA1 is used for screening of 107 veterinary drugs in 15 drug classes. The number of veterinary drugs in those classes are listed as follows: 8 analgesics/anti-inflammatories, 5 β -agonists, 7 benzimidazoles, 6 avermectins, 10 β -lactam/cephalosporins, 8 fluoroquinolones, 3 hormones, 11 macrolides, 4 ionophores, 4 general drugs, 7 nitroimidazoles, 16 sulfonamides, 4 tetracyclines, 3 phenicols, and 11 tranquilizers/sedatives. It is also used for screening 81 pesticides in 8 pesticide classes. The number of pesticides in those classes are listed as follows: 7 carbamates, 4 conazoles/triazoles, 9 halogenated pesticides, 4 neonicotinoids, 18 organophosphates, 31 general pesticides, 5 pyrethroids, and 3 triazines. The method is applicable for the analysis of veterinary drug residues and pesticides in bovine, poultry, porcine, caprine, ovine, and *Siluriformes* (catfish) muscle tissue, as well as liquid egg products. CLG-MEGA1 was initially developed by the Agricultural Research Service (ARS) in collaboration with the FDA, and FSIS has further optimized the method.

Chemical residues from veterinary drugs and pesticides are extracted from tissue using a solution of acetonitrile and water. The extracted veterinary drug residues and pesticides are examined using ultra-high performance liquid chromatography with a tandem mass spectrometer (UHPLC-MS-MS) under electrospray ionization (ESI) conditions.

KEY DEFINITIONS

UHPLC-MS-MS: An analytical technique where there is a physical separation of target compounds followed by their mass-based detection.

This method is to be performed using the standards and solutions for the respective analyte(s) of interest. Only applicable standards and solutions are necessary for reporting results.

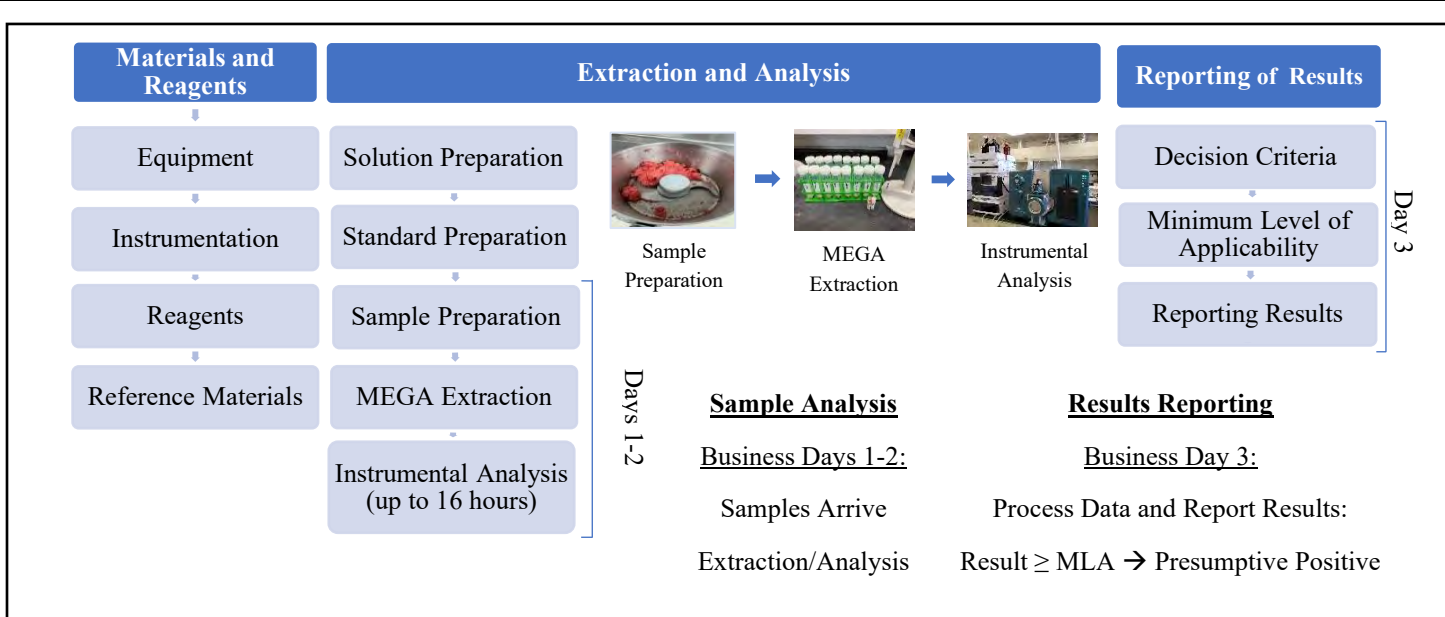


Figure 1: Overview and timeframe of CLG-MEGA1. Materials and reagents are obtained and utilized to prepare solutions and standards. The samples arrive at laboratory, are prepared into a homogenized mixture, weighed, extracted, and analyzed by UHPLC-MS/MS on business days 1-2. Screening results are reported on business day 3. This figure represents the best-case scenarios, but analyses may take longer. Photos courtesy of Hue Quach, USDA-FSIS, Ryan Matsuda, USDA-FSIS, and Sam Zipperer, USDA-FSIS.

Decision Criteria

A sample is considered negative if the results are less than the Minimum Level of Applicability (MLA). A sample is considered a presumptive positive if the results are greater than or equal to the MLA. Screened positive results will require further analysis through additional methods to determine if the sample is violative.

KEY DEFINITIONS
MLA: Lowest level at which an FSIS method has been successfully validated for a residue in each matrix. Full definition is on the [CLG website](#).

Disclosure Statement

FSIS does not specifically endorse any test products listed in this method. FSIS acknowledges that equivalent equipment, reagents, or solutions may be suitable for laboratory use. The FSIS laboratory system uses method performance requirements when evaluating the equivalence of alternative equipment, reagents, or solutions for a given analyte and sample matrix pair. Significant equivalence changes would require FSIS laboratory leadership approval.

Materials and Reagents

Equipment

Table 1: Equipment Required to Perform CLG-MEGA1

Equipment	Supplier and Part Number	Purpose
Pulsating vortex platform shaker	General lab supplier	Facilitates extraction of residue from the sample.
Centrifuge capable of ~ 4700 RCF	General lab supplier	Separates the solid sample material from the extraction solution.
Analytical Balance	General lab supplier	Record weight of standard reagent. Minimum readability ± 0.0001 g.
Top Loading Balance	General lab supplier	Record weight of quality controls and samples. Minimum readability ± 0.01 g.
Centrifuge tubes Polypropylene (PP), 50 mL	General lab supplier	Contain sample material and extraction vessel.
LC vials with screw cap lids - Amber glass, 4 mL	General lab supplier	Store standard solutions.
Plastic screw cap vials - Polypropylene, 4 mL	General lab supplier	Store standard solutions.
Screw top, amber glass, autosampler vials, PTFE septa, 2 mL	General lab supplier	Store extract.
Food Processor	Robot Coupe USA Inc.	Homogenize sample.
Freezer, -10 °C	General lab supplier	Store samples, standards, and reagents.
Repeating pipettes and tips, 2 μL - 20 μL, 20 μL - 1000 μL, 500 μL - 2500 μL	General lab supplier	Dispense standards and reagents.
Bottle-Top dispensers, 1 mL - 5 mL, 2 mL and 10 mL	General lab supplier	Add solutions.

Instrumentation

Table 2: Instrumentation

Instrument	Supplier and Model Number	Purpose
1290 Infinity UHPLC with SCIEX QTrap 6500+ mass spectrometer	Agilent/SCIEX	Sample extract analysis
1290 Infinity UHPLC with SCIEX 7500 QTrap Activated mass spectrometer	Agilent/SCIEX	Sample extract analysis
Kinetex core-shell C18 column, 50 × 3 mm, 1.7 μm	Phenomenex, 00B-4475-Y0	Sample extract analysis
SecurityGuard ULTRA Cartridges, UHPLC C18 3.0 mm ID Columns	Phenomenex, AJ0-8775	Sample extract analysis
SecurityGuard ULTRA Holder, for UHPLC Columns 2.1 to 4.6 mm ID	Phenomenex, AJ0-9000	Sample extract analysis

Reagents

Table 3: Reagents

Reagent	Supplier and Part Number
Acetonitrile (ACN) - LC-MS Grade	General lab supplier
Formic acid - LC-MS Grade	General lab supplier
Water – Resistivity of > 18 MΩ-cm	House system
Sodium hydroxide (NaOH) - pellets	General lab supplier
Methanol (MeOH)	General lab supplier
Dimethyl sulfoxide (DMSO)	General lab supplier
Acetone	General lab supplier

Reference Materials		
---------------------	--	--

Table 4: Veterinary Drug Residue Reference Materials		
--	--	--

Standard	Supplier	Catalog Number
2-Amino-flubendazole	LGC Standards	DRE-C10202370
2-Aminosulfone albendazole	LGC Standards	DRE-C10065200
2-Quinoxaline carboxylic acid (QCA)	LGC Standards	DRE-C16713000
Abamectin	LGC Standards	DRE-CA10001000
Acepromazine	LGC Standards	DRE-C10010300
Albendazole	LGC Standards	DRE-C10065000
Amoxicillin	LGC Standards	DRE-C10242500
Ampicillin	LGC Standards	DRE-C10243080
Azaperone	LGC Standards	DRE-C10340510
Butorphanol	Cayman Chemical	23812
Carazolol	LGC Standards	DRE-C10968000
Carbadox	LGC Standards	DRE-C10968300
Cefazolin	MilliporeSigma	PHR1291
Chloramphenicol	LGC Standards	DRE-C11120000
Chlorpromazine	LGC Standards	DRE-C11575000
Chlortetracycline	MilliporeSigma	PHR1520
Cimaterol	LGC Standards	DRE-C11666350
Ciprofloxacin	MilliporeSigma	PHR1044
Clenbuterol	LGC Standards	DRE-C11668550
Clindamycin	MilliporeSigma	PHR1159
Cloxacillin	MilliporeSigma	PHR1922
Danofloxacin	LGC Standards	DRE-C11960400
DCCD	LGC Standards	TRC-D289905
Desacetyl cephalixin	LGC Standards	TRC-D288970
Desethylene ciprofloxacin	LGC Standards	TRC-D289150
Diclofenac	MilliporeSigma	PHR1144
Dicloxacillin	LGC Standards	DRE-C12560500
Difloxacin	LGC Standards	DRE-C12627000
Dimetridazole	LGC Standards	DRE-C12772000
Dimetridazole - OH	LGC Standards	DRE-C12772050
Dipyrene	LGC Standards	MM0052.00-0250
Doramectin	LGC Standards	DRE-C13083000

Standard	Supplier	Catalog Number
Doxycycline	LGC Standards	DRE-C13084280
Emamectin benzoate	LGC Standards	DRE-C13117000
Enrofloxacin	MilliporeSigma	PHR1513
Erythromycin A	MilliporeSigma	PHR1039
Fenbendazole	MilliporeSigma	PHR1832
Fenbendazole sulphone	LGC Standards	DRE-C15783010
Florfenicol	LGC Standards	DRE-C13665000
Florfenicol amine	LGC Standards	DRE-C13665020
Flubendazole	LGC Standards	DRE-C13678000
Flunixin	LGC Standards	DRE-C13726900
Gamithromycin	LGC Standards	DRE-C13998500
Haloperidol	LGC Standards	DRE-C14059400
Ipronidazole	LGC Standards	DRE-C14370700
Ipronidazole - OH	LGC Standards	DRE-C14370720
Ivermectin	MilliporeSigma	PHR1380
Ketamine	LGC Standards	DRE-C14531000
Ketoprofen	LGC Standards	DRE-C14532100
Lasalocid A	LGC Standards	DRE-C14593000
Levamisole	LGC Standards	DRE-C14629700
Lincomycin	MilliporeSigma	PHR1657
Melengestrol acetate	MilliporeSigma	PHR3035
Meloxicam	MilliporeSigma	PHR1799
Metronidazole	LGC Standards	DRE-C15201000
Metronidazole - OH	LGC Standards	DRE-C15201300
Monensin	MilliporeSigma	PHR2775
Morantel tartrate	MilliporeSigma	PHR3209
Moxidectin	LGC Standards	DRE-CA15335000
Nafcillin	US Pharmacopeia	1450007
Narasin	US Pharmacopeia	1457458
Norfloxacin	LGC Standards	DRE-C15648000
Orbifloxacin	LGC Standards	DRE-C15742000
Oxacillin	LGC Standards	DRE-C15755100
Oxyphenylbutazone	LGC Standards	DRE-C15810000
Oxytetracycline	LGC Standards	DRE-C15820000

Standard	Supplier	Catalog Number
Penicillin G	LGC Standards	DRE-C15935000
Phenylbutazone	LGC Standards	DRE-C16056500
Pirlimycin	LGC Standards	TRC-P509300
Prednisone	LGC Standards	DRE-C16286550
Promethazine	LGC Standards	DRE-C16355000
Propionylpromazine	LGC Standards	DRE-C16494500
Ractopamine	LGC Standards	DRE-C16805000
Ronidazole	LGC Standards	DRE-C16815500
Salbutamol	LGC Standards	DRE-C16903000
Salinomycin	MilliporeSigma	46729
Sarafloxacin	LGC Standards	DRE-C16908000
Selamectin	US Pharmacopeia	1611729
Sulfachloropyridazine	LGC Standards	DRE-C16990100
Sulfadiazine	LGC Standards	DRE-C16990500
Sulfadimethoxine	LGC Standards	DRE-C16990550
Sulfadoxine	LGC Standards	DRE-C16990600
Sulfaethoxypyridazine	LGC Standards	DRE-C16990650
Sulfamerazine	LGC Standards	DRE-C16995100
Sulfamethazine	LGC Standards	DRE-C16996500
Sulfamethizole	LGC Standards	DRE-C16998000
Sulfamethoxazole	LGC Standards	DRE-C16998100
Sulfamethoxypyridazine	LGC Standards	DRE-C16998150
Sulfanilamide	LGC Standards	DRE-C17000000
Sulfanitran	LGC Standards	DRE-C17000050
Sulfapyridine	LGC Standards	DRE-C17000100
Sulfaquinoxaline	LGC Standards	DRE-C16990000
Sulfathiazole	LGC Standards	DRE-C17000200
Sulfisoxazole	LGC Standards	DRE-C17000450
Taleranol (B-Zearalanol)	LGC Standards	DRE-C17947330
Tetracycline	LGC Standards	DRE-C17396150
Thiabendazole	LGC Standards	DRE-C17450000
Tildipirosin	LGC Standards	DRE-C17580000
Tilmicosin	LGC Standards	DRE-C17582000
Tolfenamic acid	LGC Standards	DRE-C17591000

Standard	Supplier	Catalog Number
Triflupromazine	LGC Standards	DRE-C17848000
Tulathromycin A	LGC Standards	DRE-C17895290
Tylosin	MilliporeSigma	PHR2652
Tylvalosin	LGC Standards	TRC-T634280
Virginiamycin	MilliporeSigma	V2753
Xylazine	MilliporeSigma	PHR3263
Zilpaterol	LGC Standards	DRE-C17949010

When possible, reference materials are to be purchased from manufacturers accredited to ISO Standard 17034.

Purity and counterions are to be taken into account when calculating standard concentrations. In-house prepared standards are to be assigned an expiration date that is no later than the stability stated in the method.

Table 5: Pesticides Reference Materials

Standard	Supplier	Catalog Number
Custom Pesticide Standard	AccuStandard, Inc.	S-104805-01-4X
Custom Pesticide Standard	AccuStandard, Inc.	S-104805-02-4X
Custom Pesticide Standard	AccuStandard, Inc.	S-104805-03-4X
Custom Pesticide Standard	AccuStandard, Inc.	S-104805-04
Pyrethrin	AccuStandard, Inc.	P-187S-CN
Spirodiclofen	AccuStandard, Inc.	P-938S-CN

When possible, reference materials are to be purchased from manufacturers accredited to ISO Standard 17034.

Purity and counterions are to be taken into account when calculating standard concentrations. In-house prepared standards are to be assigned an expiration date that is no later than the stability stated in the method.

Table 6: Internal Standard Reference Materials

Standard	Supplier	Catalog Number
Flunixin-d₃	LGC Standards	DRE-C13727010
Penicillin G-d₇	LGC Standards	TRC-B288600
Sulfamethazine phenyl-¹³C₆	LGC Standards	DRE-C16996502
Ethoprophos	AccuStandard, Inc.	P-129S-A-10X
Atrazine-d₅	LGC Standards	DRE-C10330100

When possible, reference materials are to be purchased from manufacturers accredited to ISO Standard 17034.

Purity and counterions are to be taken into account when calculating standard concentrations. In-house prepared standards are to be assigned an expiration date that is no later than the stability stated in the method.

Extraction and Analysis

Solution Preparation

Table 7: Solutions

Solution	Procedure
80:20 Acetonitrile/Water (v/v)	<ol style="list-style-type: none"> 1) Measure 800 mL of acetonitrile using a graduated cylinder and transfer to a container. 2) Measure 200 mL of water using a graduated cylinder and add to the container containing the acetonitrile. 3) Mix solution and transfer to a glass dispenser bottle. 4) Store at room temperature. <p><i>Solution expires 1 year after preparation.</i></p>
50:50 Acetonitrile/Methanol (ACN/MeOH) (v/v)	<ol style="list-style-type: none"> 1) Measure 50 mL of acetonitrile using a graduated cylinder and transfer to a container. 2) Measure 50 mL of methanol using a graduated cylinder and add to the container containing the acetonitrile. 3) Mix solution and transfer to a glass dispenser bottle. 4) Store at room temperature. <p><i>Solution expires 1 year after preparation.</i></p>
50:50 Methanol/Water (v/v)	<ol style="list-style-type: none"> 1) Measure 500 mL of methanol using a graduated cylinder and transfer to a container. 2) Measure 500 mL of water using a graduated cylinder and add to the container containing the methanol. 3) Mix solution and transfer to a glass dispenser bottle. 4) Store at room temperature. <p><i>Solution expires 1 year after preparation.</i></p>
3% DMSO in Acetonitrile (v/v)	<ol style="list-style-type: none"> 1) Measure 3 mL of DMSO and add to a 100 mL volumetric flask. 2) Dilute to volume with acetonitrile. 3) Mix and transfer solution to a storage vessel. 4) Store at room temperature. <p><i>Solution expires 1 year after preparation.</i></p>
6% DMSO in Acetonitrile (v/v)	<ol style="list-style-type: none"> 1) Measure 6 mL of DMSO and add to a 100 mL volumetric flask. 2) Dilute to volume with acetonitrile. 3) Mix and transfer solution to a storage vessel. 4) Store at room temperature. <p><i>Solution expires 1 year after preparation.</i></p>

12.5% DMSO in Acetonitrile (v/v)

- 1) Measure 12.5 mL of DMSO and add to a 100 mL volumetric flask.
- 2) Dilute to volume with acetonitrile.
- 3) Mix and transfer solution to a storage vessel.
- 4) Store at room temperature.

Solution expires 1 year after preparation.

12.5% DMSO in Methanol (v/v)

- 1) Measure 12.5 mL of DMSO and add to a 100 mL volumetric flask.
- 2) Dilute to volume with methanol.
- 3) Mix and transfer solution to a storage vessel.
- 4) Store at room temperature.

Solution expires 1 year after preparation.

0.03 M Sodium Hydroxide

- 1) Add 0.12 g of NaOH to a 100 ml volumetric flask containing 80 mL of water.
- 2) Mix and allow solution to cool.
- 3) Adjust to final volume using water.
- 4) Store in a plastic container.
- 5) Store at room temperature.

Solution expires 1 year after preparation.

UHPLC Aqueous Mobile Phase (0.1% Formic acid in water)

- 1) Add 1.0 mL of formic acid to a 1 L volumetric flask.
- 2) Dilute to volume with water.
- 3) Mix and transfer to the glass aqueous reservoir of the LC.
- 4) Store at room temperature.

Solution expires 1 year after preparation.

UHPLC Organic Mobile Phase (0.1% Formic acid in acetonitrile)

- 1) Add 1.0 mL of formic acid into a 1 L volumetric flask.
- 2) Dilute to volume using acetonitrile.
- 3) Mix and transfer to the glass organic reservoir of the LC.
- 4) Store at room temperature.

Solution expires 1 year after preparation.

Standard Preparation

Veterinary drug residue standards are prepared and defined as Acetonitrile Mix or Beta-Lactam Mix based on their preparation solvents. Pesticides standards are prepared and defined as Pesticide Mix. Internal standards are prepared and defined as Internal Standard Mix.

Acetonitrile Mix

Table 8: Acetonitrile Mix Single-Analyte Stock Standard Preparation

Solution	Procedure
Single-analyte Stock Standard Solutions for Acetonitrile Mix	<ol style="list-style-type: none"> 1) For each stock solution, calculate the amount of base material needed (e.g., accounting for purity and/or water and counterion content) to prepare the concentration listed in Table 9 using the appropriate solvent listed. 2) Other concentrations are to be used based on two criteria: <ol style="list-style-type: none"> a) Solubility of the drug in the solvent b) Cost and availability of the drug 3) Transfer to storage container. 4) Store in freezer at $< -10\text{ }^{\circ}\text{C}$. <p style="text-align: center;"><i>Solution expires 6 months after preparation.</i></p>

Table 9: Acetonitrile Mix Single-Analyte Stock Standard Concentration

Standard Analyte	Solvent used	Stock Standard Solution Concentration (ng/ μL)
Acetonitrile Mix Stock Standards		
2-Amino-flubendazole	12.5% DMSO in Acetonitrile	1000
2-Aminosulfone albendazole	12.5% DMSO in Methanol	1000
2-Quinoxaline carboxylic acid (QCA)	Purchased	15
Abamectin	Acetonitrile	1000
Acepromazine	Acetonitrile	1000
* Albendazole	12.5% DMSO in Acetonitrile	1000
Azaperone	Methanol	1000
Butorphanol	Methanol	1000
Carazolol	Acetonitrile	1000
Carbadox	100% DMSO	1000
Cefazolin	Water	400
Chloramphenicol	Acetonitrile	1000
Chlorpromazine	Acetonitrile	1000
Chlortetracycline	Methanol	500
Cimaterol	Acetonitrile	1000
Ciprofloxacin	0.03 M NaOH	1000
* Clenbuterol	Acetonitrile	1000

The stock standard solutions identified with an asterisk (*) symbol may require gentle heating at the time of preparation and before preparation of mixed working standards to aid in the dissolution of material.

Standard Analyte	Solvent used	Stock Standard Solution Concentration (ng/ μ L)
* Clindamycin	Acetonitrile	1000
Danofloxacin	0.03 M NaOH	1000
Desethylene ciprofloxacin	0.03 M NaOH	300
Diclofenac	6% DMSO in Acetonitrile	1000
Difloxacin	50% ACN/MeOH	500
Dimetridazole	Acetonitrile	1000
Dimetridazole - OH	Acetone	1000
Dipyrrone	Methanol	1000
Doramectin	Acetonitrile	1000
Doxycycline	Methanol	1000
Emamectin benzoate	Acetonitrile	1000
Enrofloxacin	Acetonitrile	500
Erythromycin A	Acetonitrile	1000
* Fenbendazole	12.5% DMSO in Methanol	1000
* Fenbendazole sulphone	12.5% DMSO in Acetonitrile	1000
Florfenicol	Acetonitrile	1000
Florfenicol amine	Acetonitrile	1000
* Flubendazole	12.5% DMSO in Acetonitrile	1000
Flunixin	Methanol	1000
Gamithromycin	Acetonitrile	500
Haloperidol	Methanol	1000
Ipronidazole	Acetonitrile	250
Ipronidazole - OH	Acetonitrile	250
Ivermectin	Acetonitrile	1000
Ketamine	Purchased	1000
Ketoprofen	Acetonitrile	1000
Lasalocid A	Purchased	100
Levamisole	12.5% DMSO in Methanol	1000
Lincomycin	50% ACN/MeOH	500
Melengestrol acetate	Acetonitrile	1000
Meloxicam	Acetonitrile	1000
Metronidazole	Acetonitrile	1000
Metronidazole - OH	Acetonitrile	500
Monensin	Methanol	1000
Morantel tartrate	Water	1000
Moxidectin	Acetonitrile	1000
Narasin	Methanol	1000
* Norfloxacin	Acetonitrile	1000
* Orbifloxacin	Methanol	500
Oxyphenylbutazone	Acetonitrile	1000
Oxytetracycline	Methanol	1000
Phenylbutazone	Acetonitrile	1000
Pirlimycin	50% ACN/MeOH	1000

The stock standard solutions identified with an asterisk (*) symbol may require gentle heating at the time of preparation and before preparation of mixed working standards to aid in the dissolution of material.

Standard Analyte	Solvent used	Stock Standard Solution Concentration (ng/ μ L)
Prednisone	Methanol	1000
Promethazine	Acetonitrile	1000
Propionylpromazine	Acetonitrile	1000
Ractopamine	Water	1000
Ronidazole	Acetonitrile	1000
* Salbutamol	Acetonitrile	1000
Salinomycin	Acetonitrile	1000
Sarafloxacin	Methanol	1000
Selamectin	Acetonitrile	1000
Sulfachloropyridazine	Acetonitrile	1000
* Sulfadiazine	Acetonitrile	1000
Sulfadimethoxine	Acetonitrile	1000
Sulfadoxine	Acetonitrile	1000
Sulfaethoxypyridazine	Acetonitrile	1000
Sulfamerazine	Acetonitrile	1000
Sulfamethazine	Acetonitrile	1000
Sulfamethizole	Acetonitrile	1000
Sulfamethoxazole	Acetonitrile	1000
Sulfamethoxypyridazine	Acetonitrile	1000
Sulfanilamide	Acetonitrile	500
Sulfanitran	Acetonitrile	500
Sulfapyridine	Acetonitrile	1000
* Sulfaquinoxaline	Acetonitrile	500
Sulfathiazole	Acetonitrile	1000
Sulfisoxazole	Acetonitrile	1000
Taleranol (B-Zearalanol)	Methanol	1000
Tetracycline	Methanol	500
Thiabendazole	Methanol	1000
Tildipirosin	Methanol	1000
Tilmicosin	Acetonitrile	1000
Tolfenamic acid	Acetonitrile	1000
Triflupromazine	3% DMSO in Acetonitrile	1000
Tulathromycin A	Acetonitrile	1000
Tylosin	Acetonitrile	1000
Tyvalosin	Methanol	1000
Virginiamycin	Methanol	250
Xylazine	Acetonitrile	1000
Zilpaterol	Water	1000

The stock standard solutions identified with an asterisk (*) symbol may require gentle heating at the time of preparation and before preparation of mixed working standards to aid in the dissolution of material.

Table 10: Intermediate Acetonitrile Mix Standard Solutions Preparation

Solution	Procedure
Intermediate standard solutions	<ol style="list-style-type: none"> 1) Prepare individual intermediate standard solutions as described for the analytes in Table 11 using 10 mL volumetric flasks. 2) Calculate the volume based on the actual single analyte stock standard concentration to prepare at the concentration listed in Table 11 using the appropriate solvent listed. 3) Transfer to storage container. 4) Store in freezer at <-10 °C. <p><i>Solution expires 6 months after preparation.</i></p>

Table 11: Intermediate Acetonitrile Mix Standard Solution Concentration

Standard Analyte	Stock Standard Solution Concentration (ng/μL)	Stock Standard Solution Volume (μL)	Solvent Used	Intermediate Standard Solution concentration (ng/μL)
Azaperone	1000	1000	Methanol	100
Butorphanol	1000	1000	Methanol	100
Carazolol	1000	1000	Acetonitrile	100
Chloramphenicol	1000	500	Acetonitrile	50
Chlorpromazine	1000	1000	Acetonitrile	100
Cimaterol	1000	500	Acetonitrile	50
Dimetridazole	1000	1000	Acetonitrile	100
Haloperidol	1000	1000	Methanol	100
Metronidazole	1000	1000	Acetonitrile	100
Promethazine	1000	1000	Acetonitrile	100
Propionylpromazine	1000	1000	Acetonitrile	100
Ractopamine	1000	500	Water	50
Ronidazole	1000	1000	Acetonitrile	100
Salbutamol	1000	500	Acetonitrile	50
Triflupromazine	1000	1000	Acetonitrile	100
Xylazine	1000	1000	Acetonitrile	100
Zilpaterol	1000	1000	Water	100

Table 12: Acetonitrile Mix Working Solution Preparation

Working Solution	Procedure
Acetonitrile Mix Working Solution	<ol style="list-style-type: none"> 1) Calculate the volume of stock or intermediate stock solution required to give the working standard concentration listed for each analyte in Table 13. 2) Pipet the calculated volume of stock into a 100 mL volumetric flask. 3) Dilute to 100 mL volume with acetonitrile. 4) Cap the flask and mix. 5) Transfer to 4 mL amber glass LC vials with screw cap lids. 6) Store in freezer at <-10 °C. <p><i>Solution expires 6 months after preparation.</i></p>

Table 13: Acetonitrile Mix Working Standard Solution Concentration

Standard Analyte	Stock or Intermediate Standard Solution Concentration (ng/μL)	Stock or Intermediate Standard Solution Volume (μL)	Acetonitrile Mix Working Standard Solution Concentration (ng/μL)
2-Amino-flubendazole	1000	50	0.5
2-Aminosulfone albendazole	1000	125	1.25
2-Quinoxaline carboxylic acid (QCA)	15	5000	0.75
Abamectin	1000	125	1.25
Acepromazine	1000	20	0.2
Albendazole	1000	125	1.25
Azaperone	100	50	0.05
Butorphanol	100	50	0.05
Carazolol	100	50	0.05
Carbadox	1000	75	0.75
Chloramphenicol	50	300	0.15
Chlorpromazine	100	50	0.05
Chlortetracycline	500	10000	50
Cimaterol	50	300	0.15
Ciprofloxacin	1000	125	1.25
Clenbuterol	1000	15	0.15
Clindamycin	1000	250	2.5
Danofloxacin	1000	125	1.25
Desethylene ciprofloxacin	300	417	1.25
Diclofenac	1000	25	0.25

Standard Analyte	Stock or Intermediate Standard Solution Concentration (ng/ μ L)	Stock or Intermediate Standard Solution Volume (μ L)	Acetonitrile Mix Working Standard Solution Concentration (ng/ μ L)
Difloxacin	500	250	1.25
Dimetridazole	100	50	0.05
Dimetridazole - OH	1000	250	2.5
Dipyron	1000	125	1.25
Doramectin	1000	37.5	0.375
Doxycycline	1000	125	1.25
Emamectin benzoate	1000	37.5	0.375
Enrofloxacin	500	250	1.25
Erythromycin A	1000	250	2.5
Fenbendazole	1000	1000	10
Fenbendazole sulphone	1000	1000	10
Florfenicol	1000	500	5
Florfenicol amine	1000	750	7.5
Flubendazole	1000	50	0.5
Flunixin	1000	62.5	0.625
Gamithromycin	500	500	2.5
Haloperidol	100	50	0.05
Ipronidazole	250	20	0.05
Ipronidazole - OH	250	20	0.05
Ivermectin	1000	37.5	0.375
Ketamine	1000	100	1
Ketoprofen	1000	25	0.25
Lasalocid A	100	100	0.1
Levamisole	1000	250	2.5
Lincomycin	500	500	2.5
Melengestrol acetate	1000	100	1
Meloxicam	1000	50	0.5
Metronidazole	100	50	0.05
Metronidazole - OH	500	40	0.2
Monensin	1000	100	1
Morantel tartrate	1000	1750	17.5
Moxidectin	1000	37.5	0.375
Narasin	1000	100	1
Norfloxacin	1000	125	1.25
Orbifloxacin	500	250	1.25
Oxyphenylbutazone	1000	250	2.5
Oxytetracycline	1000	2500	25

Standard Analyte	Stock or Intermediate Standard Solution Concentration (ng/μL)	Stock or Intermediate Standard Solution Volume (μL)	Acetonitrile Mix Working Standard Solution Concentration (ng/μL)
Phenylbutazone	1000	250	2.5
Pirlimycin	1000	1250	12.5
Prednisone	1000	250	2.5
Promethazine	100	50	0.05
Propionylpromazine	100	50	0.05
Ractopamine	50	300	0.15
Ronidazole	100	50	0.05
Salbutamol	50	300	0.15
Salinomycin	1000	100	1
Sarafloxacin	1000	125	1.25
Selamectin	1000	37.5	0.375
Sulfachloropyridazine	1000	250	2.5
Sulfadiazine	1000	250	2.5
Sulfadimethoxine	1000	250	2.5
Sulfadoxine	1000	250	2.5
Sulfaethoxypyridazine	1000	250	2.5
Sulfamerazine	1000	250	2.5
Sulfamethazine	1000	250	2.5
Sulfamethizole	1000	250	2.5
Sulfamethoxazole	1000	250	2.5
Sulfamethoxypyridazine	1000	250	2.5
Sulfanilamide	500	500	2.5
Sulfanitran	500	500	2.5
Sulfapyridine	1000	250	2.5
Sulfaquinoxaline	500	500	2.5
Sulfathiazole	1000	250	2.5
Sulfisoxazole	1000	100	1
Taleranol (B-Zearalanol)	1000	60	0.6
Tetracycline	500	5000	25
Thiabendazole	1000	250	2.5
Tildipirosin	1000	2500	25
Tilmicosin	1000	300	3
Tolfenamic acid	1000	125	1.25
Triflupromazine	100	50	0.05
Tulathromycin A	1000	5000	50
Tylosin	1000	500	5
Tylvalosin	1000	125	1.25

Standard Analyte	Stock or Intermediate Standard Solution Concentration (ng/ μ L)	Stock or Intermediate Standard Solution Volume (μ L)	Acetonitrile Mix Working Standard Solution Concentration (ng/ μ L)
Virginiamycin	250	1000	2.5
Xylazine	100	50	0.05
Zilpaterol	100	150	0.15

Beta-Lactam Mix

Table 14: Beta-Lactam Mix Single-Analyte Stock Standard Preparation

Solution	Procedure
Single-analyte Stock Standard Solutions for Beta-Lactam Mix	<ol style="list-style-type: none"> 1) For each stock solution, calculate the amount of base material needed (e.g., accounting for purity and/or water and counterion content) to prepare at the concentration listed in Table 15 using the appropriate solvent listed. 2) Other concentrations are to be used based on two criteria: <ol style="list-style-type: none"> a) Solubility of the drug in the solvent b) Cost and availability of the drug 3) Transfer to storage container. 4) Store in freezer at <-10 °C. <p><i>Solution expires 2 months after preparation.</i></p>

Table 15: Beta-Lactam Mix Single-Analyte Stock Standard Concentration

Standard Analyte	Solvent used	Stock Standard Solution Concentration (ng/ μ L)
Amoxicillin	Water	350
Ampicillin	Water	250
Cefazolin	Water	400
Cloxacillin	Water	200
* DCCD	Water	300
Desacetyl cephalixin	Water	250
Dicloxacillin	Water	200
Nafcillin	Water	300
Oxacillin	Water	200
Penicillin G	Water	250

The stock standard solutions identified with an asterisk (*) symbol may require gentle heating at the time of preparation and before preparation of mixed working standards to aid in the dissolution of material

Table 16: Beta-Lactam Mix Working Solution Preparation

Working Solution	Procedure
Beta-Lactam Mix working solution	<ol style="list-style-type: none"> 1) Calculate the volume of stock solution required to give the working standard concentration listed for each analyte in Table 17. 2) Pipet the calculated volume of stock to a 25 mL volumetric flask. 3) Dilute to 25 mL volume with water. 4) Cap flask and mix. 5) Transfer to 4 mL polypropylene vials with screw cap lids. 6) Store in freezer at <-10 °C <p><i>Solution expires 2 months after preparation</i></p>

Table 17: Beta-Lactam Mix Working Standard Solution Concentration

Standard Analyte	Stock Standard Solution Concentration (ng/μL)	Stock Standard Solution Volume (μL)	Beta-Lactam Mix Working Standard Solution Concentration (ng/μL)
Amoxicillin	350	71.4	1
Ampicillin	250	25	0.25
Cefazolin	400	156.3	2.5
Cloxacillin	200	31.3	0.25
DCCD	300	208	2.5
Desacetyl cephalosporin	250	250	2.5
Dicloxacin	200	312.5	2.5
Nafcillin	300	208.3	2.5
Oxacillin	200	312.5	2.5
Penicillin G	250	125	1.25

Pesticide Mix

Pesticide standards are prepared using custom mixes (S-104805-01-4X, S-104805-02-4X, S-104805-03-4X, S-104805-04) and commercially prepared single standards (Pyrethrin and Spirodiclofen).

Table 18 lists the concentrations for the Pesticide Mix Stock Standard Concentrations.

Table 18: Pesticide Mix Single Analyte Stock Standard Concentration

Standard Analyte	Solvent used	Stock Standard Solution Concentration (ng/μL)
Acephate	Acetonitrile	80
Acetamiprid	Acetonitrile	10
Alachlor	Acetonitrile	20
Aldicarb	Acetonitrile	20
Aldicarb sulfone	Acetonitrile	20
Atrazine	Acetonitrile	20
Azinphos methyl	Acetonitrile	20
Atrazine desethyl	Acetonitrile	20
Azoxystrobin	Acetonitrile	10
Benoxacor	Acetonitrile	10
Boscalid	Acetonitrile	20
Buprofezin	Acetonitrile	50
Carbaryl	Acetonitrile	50
Carbofuran	Acetonitrile	50
Carbofuran-3-hydroxy	Acetonitrile	50
Carboxin	Acetonitrile	15
Carfentrazone ethyl	Acetonitrile	100
Chlorpyrifos	Acetonitrile	15
Chlorpyrifos methyl	Acetonitrile	10
Clofentezine	Acetonitrile	50
Clothianidin	Acetonitrile	10
Coumaphos O	Acetonitrile	200
Coumaphos S	Acetonitrile	250
Diazinon	Acetonitrile	20
Dichlorvos (DDVP)	Acetonitrile	10
Difenoconazole	Acetonitrile	30
Diiflubenzuron	Acetonitrile	50
Dimethoate	Acetonitrile	20
Diuron	Acetonitrile	250
Ethion	Acetonitrile	20
Fenoxaprop ethyl	Acetonitrile	50
Fenpyroximate	Acetonitrile	10
Fipronil	Acetonitrile	10
Fipronil desulfinyl	Acetonitrile	10
Fipronil sulfide	Acetonitrile	10
Fipronil sulfone	Acetonitrile	10
Fluridone	Acetonitrile	50
Fluroxypyr-1-methylheptyl-ester	Acetonitrile	10
Fluvalinate	Acetonitrile	15
Hexazinone	Acetonitrile	100
Hexythiazox	Acetonitrile	50

Standard Analyte	Solvent used	Stock Standard Solution Concentration (ng/μL)
Imazalil	Acetonitrile	10
Imidacloprid	Acetonitrile	20
Indoxacarb	Acetonitrile	30
Linuron	Acetonitrile	100
Malathion	Acetonitrile	100
Metalaxyl	Acetonitrile	50
Methamidophos	Acetonitrile	20
Methomyl	Acetonitrile	60
Methoxyfenozide	Acetonitrile	10
Metolachlor	Acetonitrile	20
Metribuzin	Acetonitrile	10
Myclobutanil	Acetonitrile	10
Norflurazon	Acetonitrile	20
Omethoate	Acetonitrile	20
Phenothrin	Acetonitrile	40
Piperonyl butoxide	Acetonitrile	100
Pirimiphos methyl	Acetonitrile	10
Prallethrin	Acetonitrile	250
Profenofos	Acetonitrile	20
Pronamide	Acetonitrile	10
Propachlor	Acetonitrile	20
Propanil	Acetonitrile	50
Propargite	Acetonitrile	10
Propetamphos	Acetonitrile	15
Propiconazole	Acetonitrile	10
Pyraclostrobin	Acetonitrile	50
Pyridaben	Acetonitrile	50
Pyriproxyfen	Acetonitrile	100
Resmethrin (cis& trans)	Acetonitrile	250
Simazine	Acetonitrile	30
Sulprofos	Acetonitrile	50
Tebufenozide	Acetonitrile	50
Tetraclorvinphos	Acetonitrile	50
Tetraconazole	Acetonitrile	20
Thiamethoxam	Acetonitrile	20
Thiobencarb	Acetonitrile	50
Tribufos (DEF 6)	Acetonitrile	10
Trifloxystrobin	Acetonitrile	40

Table 19: Pesticide Mix Working Solution Preparation

Working Solution	Procedure
Pesticide Working solution	<ol style="list-style-type: none"> 1) Pipet 5.26 mL of Pyrethrin stock standard into a 200 mL volumetric flask. 2) Pipet 1mL of Spirodiclofen stock standard into the same flask. 3) Pipet 10 mL of S-104805-04 into the same volumetric flask. 4) Pipet 2.5 mL of S-104805-01-4X into the same volumetric flask. 5) Pipet 2.5 mL of S-104805-02-4X into the same volumetric flask. 6) Pipet 2.5 mL of S-104805-03-4X into the same volumetric flask. 7) Dilute to 200 mL volume with acetonitrile. 8) Cap flask and mix. 9) Transfer to a 250 mL glass storage container. 10) Store in freezer at <-10 °C <p><i>Solution expires 1 year after preparation</i></p>

Table 20: Pesticide Mix Working Solution Concentration

Standard Analyte	Stock Standard Solution Concentration (ng/μL)	Pesticide Mix Working Standard Solution Concentration (ng/μL)
Acephate	80	1.00
Acetamiprid	10	0.125
Alachlor	20	0.250
Aldicarb	20	0.250
Aldicarb sulfone	20	0.250
Atrazine	20	0.250
Atrazine desethyl	20	0.250
Azinphos methyl	20	0.250
Azoxystrobin	10	0.125
Benoxacor	10	0.125
Boscalid	20	0.250
Buprofezin	50	0.625
Carbaryl	50	0.625
Carbofuran	50	0.625
Carbofuran-3-hydroxy	50	0.625
Carboxin	15	0.188

Standard Analyte	Stock Standard Solution Concentration (ng/ μ L)	Pesticide Mix Working Standard Solution Concentration (ng/ μ L)
Carfentrazone ethyl	100	1.25
Chlorpyrifos	15	0.188
Chlorpyrifos methyl	10	0.125
Clofentezine	50	0.625
Clothianidin	10	0.125
Coumaphos O	200	10.0
Coumaphos S	250	12.50
Diazinon	20	0.250
Dichlorvos (DDVP)	10	0.125
Difenoconazole	30	0.375
Diflubenzuron	50	0.625
Dimethoate	20	0.250
Diuron	250	12.50
Ethion	20	0.250
Fenoxaprop ethyl	50	0.625
Fenpyroximate	10	0.125
Fipronil	10	0.125
Fipronil desulfinyl	10	0.125
Fipronil sulfide	10	0.125
Fipronil sulfone	10	0.125
Fluridone	50	0.625
Fluroxypyr-1-methylheptyl-ester	10	0.125
Fluvalinate	15	0.188
Hexazinone	100	1.25
Hexythiazox	50	0.625
Imazalil	10	0.125
Imidacloprid	20	0.250
Indoxacarb	30	0.375
Linuron	100	1.25
Malathion	100	1.25
Metalaxyl	50	0.625
Methamidophos	20	0.250
Methomyl	60	0.750
Methoxyfenozide	10	0.125
Metolachlor	20	0.250
Metribuzin	10	0.125
Myclobutanil	10	0.125
Norflurazon	20	0.250

Standard Analyte	Stock Standard Solution Concentration (ng/μL)	Pesticide Mix Working Standard Solution Concentration (ng/μL)
Omethoate	20	0.250
Phenothrin	40	0.500
Piperonyl butoxide	100	1.25
Pirimiphos methyl	10	0.125
Prallethrin	250	12.5
Profenofos	20	0.250
Pronamide	10	0.125
Propachlor	20	0.250
Propanil	50	0.625
Propargite	10	0.125
Propetamphos	15	0.188
Propiconazole	10	0.125
Pyraclostrobin	50	0.625
Pyrethrin I	56.71	1.5
Pyridaben	50	0.625
Pyriproxyfen	100	1.25
Resmethrin (cis& trans)	250	12.5
Simazine	30	0.375
Spirodiclofen	101	0.505
Sulprofos	50	0.625
Tebufenozide	50	0.625
Tetrachlorvinphos	50	2.50
Tetraconazole	20	0.25
Thiamethoxam	20	0.25
Thiobencarb	50	2.50
Tribufos (DEF6)	10	0.125
Trifloxystrobin	40	0.500

Internal Standard Mix

Table 21: Internal Standard Mix Single-Analyte Stock Standard Preparation

Solution	Procedure
Single-analyte Stock Standard Solutions for Internal Mix	<ol style="list-style-type: none"> 1) For each stock solution, calculate the amount of base material needed (ex. accounting for purity and/or water and counterion content) to prepare at the concentration listed in Table 22 using the appropriate solvent listed. 2) Other concentrations are to be used based on two criteria: <ol style="list-style-type: none"> a) Solubility of the drug in the solvent b) Cost and availability of the drug 3) Mix well and transfer to glass storage container. 4) Store in freezer at <-10 °C <p style="text-align: center;"><i>Solution expires 2 months after preparation</i></p>

Table 22: Internal Standard Mix Single-Analyte Stock Standard Concentration

Standard Analyte	Solvent used	Stock Standard Solution Concentration (ng/μL)
Atrazine-d ₅	Acetonitrile	500
Flunixin-d ₃	Methanol	1000
Penicillin G-d ₇	Water	500
Sulfamethazine phenyl- ¹³ C ₆	Acetonitrile	1000
Ethoprosfos*	Acetone	1000

*Ethoprosfos is a commercially prepared standard

Table 23: Internal Standard Mix Working Solution Preparation

Working Solution	Procedure
Internal Mix working solution	<ol style="list-style-type: none"> 1) Calculate the volume of stock solution required to give the working standard concentration listed for each analyte in Table 24. 2) Pipet the calculated volume of stock into a 50 mL volumetric flask. 3) Dilute to 50 mL volume with acetonitrile. 4) Cap flask and mix. 5) Transfer to a glass storage container for use. 6) Store in freezer at <-10 °C. <p style="text-align: center;"><i>Solution expires 2 months after preparation.</i></p>

Table 24: Internal Standard Mix Working Solution Concentration

Standard Analyte	Stock Standard Solution Concentration (ng/ μ L)	Stock Standard Solution Volume (μ L)	Internal Standard Mix Working Standard Solution Concentration (ng/ μ L)
Atrazine-d ₅	500	500	5
Flunixin-d ₃	1000	250	5
Penicillin G-d ₇	500	500	5
Sulfamethazine phenyl- ¹³ C ₆	1000	250	5
Ethoprophos	1000	250	5

If preparing an External Calibration Curve for system suitability, use Table 25 to prepare external standards. External Calibration Curve solutions are prepared the day of use.

Table 25: Preparation of External Standards

Level	Acetonitrile Standard Mix Volume (μ L)	Beta Lactam Standard Mix Volume (μ L)	Pesticides Standard Mix Volume (μ L)	Internal Standard Mix Volume (μ L)	80:20 Acetonitrile/Water Volume (μ L)
1/2 X	4	4	4	3	985
1 X	8	8	8	3	973
2 X	16	16	16	3	949

Sample Preparation

Samples must be kept cold before and during shipping to the laboratory. Once received at the laboratory, samples must be frozen (≤ -10 °C) prior to grinding if they cannot be prepared on the day of receipt. Once frozen, temper (partially thaw) the sample while keeping it as cold as possible. As shown in Figure 2, trim away fat and connective tissue from the tissue. As shown in Figure 3, grind the tissue in a blender or vertical cutter-mixer until homogeneous. Store samples frozen (≤ -10 °C) prior to analysis.

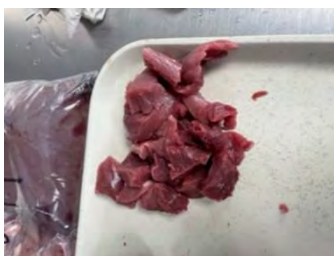


Figure 2: Prepared lean muscle sample with connective tissue removed. Photo courtesy of Hue Quach, USDA-FSIS.



Figure 3: Homogenized sample. Photo courtesy of Hue Quach, USDA-FSIS.



Figure 4: Liquid egg samples. Photo courtesy of Hue Quach USDA-FSIS.

Liquid egg products require no sample preparation. An example of a liquid egg product is shown in Figure 4.

MEGA Extraction

Samples

Weigh 2.0 ± 0.1 g of homogenized samples or egg products into labeled 50 mL polypropylene centrifuge tubes, as shown in Figure 5.

KEY DEFINITIONS

Negative control (Blank): A sample that is negative of all analytes.

Matrix matched control: A sample prepared with the addition of analytes post-extraction to have a specified concentration level.

Recovery (positive control): A sample is prepared by the addition of analytes prior to extraction that have a concentration level comparable to MLA. Samples are compared to the recovery.

QUALITY CONTROL

- 1) Weigh seven 2 g portions of blank porcine tissue for muscle screening or blank whole egg for liquid egg product screening into 50 mL polypropylene centrifuge tubes. One for the blank (negative control), one each for the 1/2 X, 1 X, and 2 X recoveries (positive control), and one each for the 1/2 X, 1 X, and 2 X matrix matched controls. Weigh one additional portion for a check sample, if necessary.
- 2) Prepare recoveries using the solutions and volumes in Table 26.
- 3) Matrix matched controls are to be spiked during Step 6 of the extraction procedure.

Table 26: Preparation of Recovery Controls

Sample Type	Acetonitrile Standard Mix Volume (μL)	Beta Lactam Standard Mix Volume (μL)	Pesticide Spiking Mix Volume (μL)
1/2 X Recovery	40	40	40
1 X Recovery	80	80	80
2 X Recovery	160	160	160

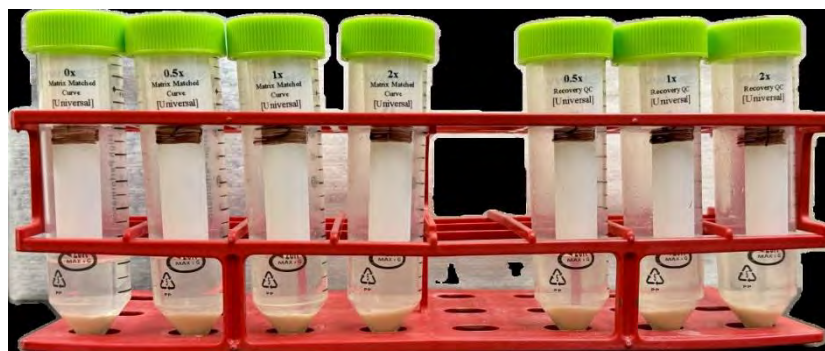


Figure 5: Weighed controls. Photo courtesy of Nicholas Paganella, USDA-FSIS.

Extraction

- 1) Add 25 μL of Internal Standard Mix to all tubes except matrix match control.
- 2) Vortex tubes for 10 seconds (sec) each to mix chemicals with matrix.
- 3) Add 10 mL of 80:20 acetonitrile/water (v/v) to all tubes. Cap tubes well.
- 4) Vortex the tubes using the pulsating vortex platform shaker for 10 minutes (min). Ensure that solvent reaches entire sample, as shown in Figure 6.
- 5) Centrifuge the tubes at 4708 RCF (~4600 RPM) for 10 min.
- 6) Spike matrix matched standards according to Table 27.



Figure 6: Vortexed sample. Photo courtesy of Stephen Kubota, USDA-FSIS.

Table 27: Preparation of Matrix Matched Controls

Sample Type	Acetonitrile Standard Mix Volume (μL)	Beta Lactam Standard Mix Volume (μL)	Pesticides Spiking Mix Volume (μL)	Internal Standard Mix Volume (μL)
1/2 X Matrix Matched Control	40	40	40	25
1 X Matrix Matched Control	80	80	80	25
2 X Matrix Matched Control	160	160	160	25

- 7) Spike directly into the matrix match control 50 mL centrifuge tubes and swirl gently, being careful to avoid disruption of tissue pellet. Repeat step 5 as necessary if the tissue pellet is dispersed.
- 8) Pipette 1000 μL of sample extract to labeled amber glass autosampler vials, as shown in Figure 7.



Figure 7: Extracted samples transferred to vials. Photo courtesy of Ryan Matsuda, USDA-FSIS.

Instrumental Analysis

Chromatographic Parameters

- 1) Mobile phases for MEGA Analysis
 - a. UHPLC Aqueous Mobile Phase (0.1% Formic acid in water)
 - b. UHPLC Organic Mobile Phase (0.1% Formic acid in acetonitrile)
- 2) Flow rate (mL/min): 0.3
- 3) Run time (min): 12.9
- 4) Gradient Program

Table 28: UHPLC Gradient Program

Time (min)	% Aqueous Mobile Phase A	% Organic Mobile Phase B	Gradient
0.00	98	2	None
0.10	98	2	linear
8.00	0	100	linear
10.70	0	100	linear
10.80	98	2	linear
12.90	98	2	linear

- 5) Autosampler Program
 - a. Run time (min): 12.9
 - b. Injection Volume (μL): 1
 - c. Needle Level (mm): 0.20
 - d. Wash Time (sec): 30
 - e. Sample Temperature ($^{\circ}\text{C}$): 10
- 6) Column Oven
 - a. Column Valve Position: To match column location.
 - b. Column Temperature ($^{\circ}\text{C}$): 40

Mass Spectrometry Parameters for SCIEX QTrap 6500+ mass spectrometer

- 1) MS Method Parameters
 - a. Scan Type: Scheduled MRM
 - b. Polarity: Positive/Negative Switching
 - c. MRM Detection Window (sec): 30
 - d. Target Scan Time (sec): 1
 - e. Resolution Q1: Unit
 - f. Resolution Q3: Unit
 - g. Settling Time (msec): 20.0000
 - h. Collision Energy: Variable – see Table 29 and 30 below



Figure 8: 1290 Infinity UHPLC with SCIEX QTrap 6500+ mass spectrometer. Photo courtesy of Sam Zipperer, USDA-FSIS.

- 2) Electrospray Source Parameters
 - a. Curtain Gas: 25
 - b. Collision Gas: Medium
 - c. Temperature (°C): 425
 - d. Source Gas 1: 50
 - e. Source Gas 2: 50
 - f. Ion Spray Voltage ESI+ (V): 5000
 - g. Ion Spray Voltage ESI- (V): -4500

Mass Spectrometry Parameters for SCIEX 7500 QTrap Activated mass spectrometer

- 1) MS Method Parameters
 - a. Scan Type: Scheduled MRM
 - b. Polarity: Positive/Negative Switching
 - c. MRM Detection Window (sec): 20
 - d. Target Scan Time (sec): 1
 - e. Resolution Q1: Unit
 - f. Resolution Q3: Unit
 - g. Settling Time (msec): 20.0000
 - h. Collision Energy: Variable – see Table 31 and 32 below



Figure 9: 1290 Infinity UHPLC with SCIEX 7500 QTrap Activated mass spectrometer. Photo courtesy of Stephen Kubota, USDA-FSIS.

- 2) Electrospray Source Parameters
 - a. Curtain Gas: 25
 - b. Collision Gas: Medium
 - c. Temperature (°C): 425
 - d. Source Gas 1: 50
 - e. Source Gas 2: 50
 - f. Ion Spray Voltage ESI+ (V): 1850
 - g. Ion Spray Voltage ESI- (V): -4500

Table 29: Veterinary Drug Residue Scan Parameters for 1290 Infinity UHPLC with SCIEX QTrap 6500+ mass spectrometer

Analyte	Precursor Ion (m/z)	Retention Time (min)	Declustering Potential (V)	Entrance Potential (V)	First Transition			Second Transition		
					Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Positive ESI Mode										
2-Amino-flubendazole	256.0	3.50	145	9	123.0	37	6	95.0	45	14
2-Aminosulfone albendazole	240.0	2.59	85	10	133.1	40	6	198.0	27	10
2-Quinoxaline carboxylic acid (QCA)	175.0	3.08	63	4	131.0	23	6	102.0	40	12
Abamectin-Na	895.7	8.13	270	2	751.4	57	45	449.3	62	25
Acepromazine	327.1	4.21	72	6	86.0	25	14	221.1	51	12
Albendazole	266.0	4.33	89	9	234.0	27	12	191.0	45	10
Amoxicillin	366.2	2.12	60	3	349.0	12	16	114.0	25	18
Ampicillin	330.1	2.70	59	5	106.0	22	12	160.0	17	8
Azaperone	328.2	3.07	83	6	165.1	28	10	123.0	44	14
Butorphanol	328.2	3.57	66	5	310.1	32	26	157.1	57	26
Carazolol	299.2	3.61	77	7	116.1	26	6	194.1	39	10
Carbadox	263.3	2.88	46	8	229	24	14	130	28	14
Cefazolin	455.0	3.15	35	4	323.0	15	16	156.0	23	8
Chlorpromazine	319.0	4.70	72	6	85.8	25	14	214.0	53	12
Chlortetracycline	479.0	3.46	60	5	444.0	29	20	154.0	37	6
Cimaterol	220.0	2.13	34	6	143.1	31	6	89.0	56	10
Ciprofloxacin	332.1	2.89	99	6	314.1	27	14	288.1	24	14
Clenbuterol	277.0	3.22	37	10	202.8	23	22	132.0	39	12
Clindamycin	425.1	3.50	90	5	377.1	26	20	389.2	22	10
Cloxacillin	436.0	5.19	27	5	277.0	23	14	160.0	16	8
Danofloxacin	358.1	2.96	84	10	340.1	31	16	314.1	25	16
DCCD	549.0	2.35	95	7	183.0	38	10	241.0	29	10
Desacetyl cephalixin	382.3	1.83	66	7	112.0	31	12	124.0	61	14
Desethylene ciprofloxacin	306.1	2.72	51	4	288.1	25	16	217.0	47	20
Diclofenac	296.0	6.17	30	7	215.0	27	12	214.0	45	12

Analyte	Precursor Ion (m/z)	Retention Time (min)	Declustering Potential (V)	Entrance Potential (V)	First Transition			Second Transition		
					Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Dicloxacillin	470.0	5.53	40	7	311.0	19	16	160.0	19	10
Difloxacin	400.1	3.29	91	10	382.0	31	18	356.0	27	18
Dimetridazole	142.0	2.52	38	10	96.0	22	16	81.0	33	12
Dimetridazole - OH	158.1	2.26	36	6	139.9	16	16	111.9	26	12
Dipyron	218.1	2.30	32	7	97.0	17	15	187.1	14	10
Doramectin	921.6	8.49	50	5	777.4	59	40	449.0	64	25
Doxycycline	445.1	2.99	61	3	428.0	26	24	267.0	50	30
Emamectin benzoate	886.5	6.09	56	4	158.1	40	10	82.1	124	2
Enrofloxacin	360.1	3.04	84	8	342.0	29	16	316.0	27	16
Erythromycin A	734.3	4.08	94	8	576.4	28	26	158.1	38	8
Fenbendazole sulphone	332.0	4.33	123	7	300.0	31	16	159.0	49	8
Fenbendazole	300.0	5.00	104	5	268.0	29	14	159.0	47	8
Florfenicol amine	248.0	1.42	34	8	230.1	15	12	130.1	31	6
Florfenicol	358.0	3.78	21	6	241.0	23	12	206.1	34	12
Flubendazole	314.0	4.59	123	6	282.1	31	14	122.8	47	6
Flunixin	297.1	5.55	70	3.5	259.1	39	14	236.1	59	14
Flunixin-d ₃	300.0	5.55	90	7	282.0	33	14	264.1	47	14
Gamithromycin	777.4	3.55	66	5	619.4	45	26	601.4	49	26
Haloperidol	376.1	4.25	101	10	165.1	32	8	123.0	43	6
Iprnidazole	170.0	3.91	65	5	124	24	8	109	32	18
Iprnidazole - OH	186.1	3.21	40	3	106	49	12	168	17	14
Ivermectin	897.3	8.13	290	2	753.6	59	30	329.2	69	16
Ketamine	238.1	2.95	52	4	125.0	35	11	220.0	20	12
Ketoprofen	255.0	5.33	70	9	105.0	28	12	194.0	31	16
Levamisole	205.0	2.57	82	10	178.1	30	8	123.0	38	11
Lincomycin	407.1	2.52	66	2	126.1	33	14	359.0	24	18
Melengesterol acetate	397.2	6.75	88	8	337.3	19	16	279.2	27	14

Analyte	Precursor Ion (m/z)	Retention Time (min)	Declustering Potential (V)	Entrance Potential (V)	First Transition			Second Transition		
					Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Meloxicam	352.0	5.55	66	2	115.0	23	8	141.0	25	6
Metronidazole	172.1	2.27	37	11	128.0	19	14	82.1	31	6
Metronidazole - OH	188.0	2.01	5	3	123.1	16	6	126.0	22	6
Monensin	693.4	9.30	40	8	461.0	68	10	479.0	74	13
Morantel	221.0	3.30	74	6	123.0	47	6	111.0	33	18
Moxidectin	640.4	8.66	50	7	528.3	13	40	498.4	17	30
Nafcillin	415.1	5.30	14	5	199.0	17	11	171.0	48	19
Narasin	787.5	9.51	30	13	431.2	69	7	531.2	65	13
Norfloracin	320.1	2.79	91	6	302.0	27	16	233.0	23	12
Orbifloxacin	396.2	3.10	102	7	352.1	25	20	295.0	32	16
Oxacillin	402.0	4.95	34	8	243.0	19	12	160.0	19	8
Oxyphenylbutazone	325.1	5.40	57	6	204.1	21	8	120.1	23	6
Oxytetracycline	461.1	2.84	56	4	426.1	24	20	443.1	18	20
Penicillin G	335.0	4.44	40	6	160.0	13	10	176.1	16	10
Penicillin G-d ₇	342.2	4.43	40	6	160.0	14	10	183.0	17	10
Phenylbutazone	309.1	6.47	20	4	188.1	23	10	120.1	25	8
Pirlimycin	411.1	3.35	96	2	112.0	32	6	363.1	23	18
Prednisone	359.1	4.17	77	3	147.1	31	8	171.0	39	8
Promethazine	285.0	4.26	50	5	86.0	21	10	71.1	61	12
Propionylpromazine	341.1	4.56	55	2	86.2	25	12	236.0	49	12
Ractopamine	302.1	2.93	48	7	164.1	22	8	121.1	29	10
Ronidazole	201.0	2.46	20	2	140.0	15	15	55.0	23	13
Salbutamol	240.2	2.07	23	10	148.0	23	8	166.1	18	6
Salinomycin	773.3	9.16	50	4	431.1	71	10	531.4	63	8
Sarafloxacin	386.1	3.26	106	6	368.0	31	18	342.1	27	16
Selamectin	770.3	8.86	140	5	626.0	25	26	608.0	29	6
SMZ- ¹³ C ₆	285.0	3.18	56	7	186.1	23	10	124.1	31	8

Analyte	Precursor Ion (m/z)	Retention Time (min)	Declustering Potential (V)	Entrance Potential (V)	First Transition			Second Transition		
					Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Sulfachloropyridazine	285.0	3.58	24	8	156.0	21	8	92.0	37	14
Sulfadiazine	251.0	2.62	25	9	156.0	21	8	92.0	33	11
Sulfadimethoxine	311.1	4.20	69	6	156.0	27	8	108.0	35	18
Sulfadoxine	311.0	3.70	69	6	155.9	25	8	107.8	33	16
Sulfaethoxypyridazine	295.0	3.75	47	6	156.0	25	8	140.1	25	8
Sulfamerazine	265.0	2.95	4	8	156.0	22	8	172.0	22	9
Sulfamethazine	279.0	3.18	41	7	186.0	23	10	124.1	30	6
Sulfamethizole	270.9	3.16	29	8	156.0	19	8	92.0	37	11
Sulfamethoxazole	254.0	3.76	19	8	156.0	21	8	92.0	35	14
Sulfamethoxypyridazine	281.0	3.22	52	6	156.0	22	8	108.0	31	14
Sulfanilamide	173.0	1.56	16	10	93.0	29	10	65.0	40	12
Sulfapyridine	250.1	2.80	65	8	156.0	22	8	184.1	23	10
Sulfaquinoxaline	301.0	4.21	66	6	156.0	23	8	108.0	33	16
Sulfathiazole	256.0	3.75	22	8	92.0	37	14	108.0	31	14
Sulfisoxazole	268.2	3.89	58	10	155.9	18	10	113.0	20	8
Tetracycline	445.1	3.00	52	4	410.0	27	20	427.1	19	23
Thiabendazole	202.1	2.65	105	7	175.0	35	18	131.1	44	14
Tildipirosin	368.0	2.45	65	5	98.1	23	11	464.3	23	28
Tilmicosin	435.4	3.70	66	3	99.0	26	34	696.0	24	8
Tolfenamic acid	262.0	6.80	2	5	244.0	17	14	209.1	38	12
Triflupromazine	353.1	4.95	93	2	86.0	25	14	280.0	35	14
Tulathromycin A	404.0	2.84	45	10	577.2	19	29	158.1	28	8
Tylosin	919.6	4.20	98	6	174.0	47	10	772.0	41	32
Tyvalosin	1042.5	5.28	90	7	174.0	48	12	814.0	45	20
Virginiamycin M1	526.2	4.99	75	4	355.1	25	16	337.0	29	16
Xylazine	221.1	3.17	90	6	90.0	35	14	164.0	33	8
Zilpaterol	262.1	2.07	47	11	185.1	32	10	157.0	41	8

Analyte	Precursor Ion (m/z)	Retention Time (min)	Declustering Potential (V)	Entrance Potential (V)	First Transition			Second Transition		
					Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Negative ESI Mode										
Chloramphenicol	321	3.96	-50	-10	151.9	-22	-7	194	-18	-9
Lasalocid A	589.4	8.7	-85	-10	234.9	-40	-13	173	-54	-11
Sulfantran	334	4.76	-172	-3	135.9	-38	-7	270	-34	-13
Zearalanol	321	4.94	-130	-2	277	-28	-15	303.1	-28	-21

Table 30: Pesticide Residue Scan Parameters for 1290 Infinity UHPLC with SCIEX QTrap 6500+ mass spectrometer

Analyte	Precursor Ion (m/z)	Retention Time (min)	Declustering Potential (V)	Entrance Potential (V)	First Transition			Second Transition		
					Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Positive ESI Mode										
Acephate	184.1	2.01	30	10	125	23	15	143	11	15
Acetamiprid	223	3.75	71	10	126	27	6	90	51	6
Alachlor	269.8	6.48	27	10	237.8	11	5	161.9	19	5
Aldicarb.Na	213	4.24	90	10	89	21	10	116	17	12
Aldicarb sulfone	223	2.72	41	10	148	13	8	86	19	4
Atrazine	216	5.16	66	10	174	25	10	96	33	6
Atrazine desethyl	187.9	3.58	35	10	146	20	5	104	28	5
Atrazine-d ₅	221.1	5.14	110	5	179.1	26	10	137.1	31	10
Azinphos methyl	318	5.87	35	8	261.1	11	15	132.1	21	12
Azoxystrobin	404	6.02	91	10	372	21	16	344	35	16
Benoxacor	259.7	6	22	10	148.9	17	5	120	35	5
Boscalid	343	6.12	90	10	307	27	6	272	43	12
Buprofezin	306	6.7	56	10	201	17	10	106	33	6
Carbaryl	202	5.05	91	10	127	37	6	145	15	6
Carbofuran	222	4.86	41	10	165	17	10	123	29	8
Carbofuran-3-hydroxy	238.1	3.48	40	10	181.1	15	20	163.1	20	15
Carboxin	236	5.17	71	10	143	24	4	87	31	6

Analyte	Precursor Ion (m/z)	Retention Time (min)	Declustering Potential (V)	Entrance Potential (V)	First Transition			Second Transition		
					Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Carfentrazone ethyl	412	6.7	70	8	365.9	24	26	345.9	15	15
Chlorpyrifos	350	7.8	60	5	198	22	26	96.9	45	15
Chlorpyrifos methyl	321.9	7.17	60	7	125	24	15	289.9	21	15
Clofentezine	303	7.07	56	10	138	19	8	102	51	4
Clothianidin	250	3.43	71	10	169	19	4	132	23	6
Coumaphos O	347	5.6	45	10	291	22	5	211	34	5
Coumaphos S	362.8	7	151	10	227	35	4	306.9	23	8
Diazinon	305	7.02	80	10	169	29	8	153	27	8
Dichlorvos	220.9	4.55	55	10	109	23	6	145	17	12
Difenoconazole	406	6.76	72	10	251	33	14	337	23	16
Diflubenzuron	311	6.29	71	140	158	21	8	141	45	8
Dimethoate	230	3.67	56	10	199	13	8	125	29	6
Diuron	233	5.22	80	10	72	21	12	46	37	8
Ethion	384.7	7.82	22	10	198.8	11	5	171	25	5
Ethoprophos	243.1	6.2	23	10	173	22	5	215	17	12
Fenoxaprop ethyl	361.9	7.3	12	10	287.7	19	5	121	32	5
Fenpyroximate	422.1	7.93	100	5	366.2	24	20	215.1	38	15
Fluridone	330	5.75	22	10	309.2	33	5	290.1	35	5
Fluroxypyr-1-methylheptyl-ester	367	7.96	20	10	255	10	5	209	22	5
Fluvalinate	503	8.43	180	6	208	17	15	181	40	15
Hexazinone	252.9	4.2	32	10	170.8	17	5	70.9	33	5
Hexythiazox	353	7.8	81	10	228	21	10	168	33	8
Imazalil	297	4.42	55	10	159	37	8	255	21	12
Imidacloprid	256	3.56	60	10	175	23	12	209	27	10
Indoxacarb	528	7.2	55	10	249	23	13	293	19	6
Linuron	249	5.89	63	10	160	25	6	182	23	8
Malathion	331	6.4	78	10	127	17	6	99	31	4

Analyte	Precursor Ion (m/z)	Retention Time (min)	Declustering Potential (V)	Entrance Potential (V)	First Transition			Second Transition		
					Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Metalaxyl	280	5.14	66	10	220	19	4	192	25	8
Methamidophos	141.9	1.71	107	10	94	19	12	125.1	19	4
Methomyl	163	2.9	76	10	88	13	8	106	13	8
Methoxyfenozide	369	6.3	80	10	149	23	12	313	11	4
Metolachlor	283.8	6.45	140	10	252.2	21	25	176.2	35	20
Metribuzin	214.8	4.65	32	10	186.7	19	5	83.9	21	5
Myclobutanil	289.1	5.99	28	10	70.1	18	5	125.1	30	5
Norflurazon	304	5.3	76	10	284	33	44	160	41	28
Omethoate	214	2.22	51	10	183	15	10	125	29	8
Phenothrin (sumithrin)	351.1	8.57	60	7	183.1	30	20	305.2	16	20
Piperonyl butoxide	356	7.54	76	10	177	21	8	119	47	6
Pirimiphos methyl	305.9	6.96	12	10	107.8	33	5	163.9	21	5
Prallethrin	301.1	7.25	22	10	168.9	9	5	132.9	11	5
Profenofos	373	7.31	86	10	303	25	42	345	17	12
Propachlor	211.8	5.46	17	10	169.7	17	15	105.8	25	5
Propanil	218	5.67	91	10	162	21	8	127	35	6
Propargite	368.1	7.94	150	5	231.2	15	20	175.2	22	20
Propetamphos	281.9	6.5	17	10	137.8	19	5	155.7	11	5
Propiconazole	342	6.5	70	10	159	33	6	123	79	6
Propyzamide (Pronamide)	256.1	6.2	90	2	190.1	18	20	173	29	20
Pyraclostrobin	388	7	65	10	194	17	4	163	33	4
Pyrethrin I	329.2	8	22	10	161	9	5	143	15	5
Pyridaben	365	8.22	51	10	309	17	14	117	83	8
Pyriproxyfen	322	7.67	12	10	95.8	15	5	184.8	23	5
Resmethrin	339.2	8.33	100	10	171.1	20	20	128	60	15
Simazine	202	4.5	96	10	124	25	6	132	25	4
Spirodiclofen	411.1	8.26	75	7	313.1	16	25	71.1	35	12

Analyte	Precursor Ion (m/z)	Retention Time (min)	Declustering Potential (V)	Entrance Potential (V)	First Transition			Second Transition		
					Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Sulprofos	323	7.85	62	10	219	17	6	155	37	4
Tebufenozide	353.2	6.62	55	10	297.1	11	8	133.1	25	6
Tetrachlorvinphos	367	6.4	61	10	127	19	4	206	51	10
Tetraconazole	372	6.14	72	10	159	37	8	70	63	6
Thiamethoxam	292	3.12	66	10	211	17	4	132	27	6
Thiobencarb	257.9	7.1	25	10	125.1	20	5	100.1	10	5
Tribufos (DEF6)	315.1	8.35	110	5	169	22	15	225.1	17	15
Trifloxystrobin	409	7.28	35	10	186	25	8	206	19	4
Negative ESI Mode										
Fipronil	434.8	6.67	-65	-5	249.9	-38	-13	399	-16	-19
Fipronil desulfinyl	387	6.81	-70	-10	351	-18	-17	282	-44	-15
Fipronil sulfide	419	7.03	-76	-10	383	-18	-17	261.9	-40	-13
Fipronil sulfone	450.9	7	-70	-10	415	-23	-20	281.9	-38	-20

Table 31: Veterinary Drug Residue Scan Parameters for 1290 Infinity UHPLC with SCIEX 7500 QTrap Activated mass spectrometer

Analyte	Precursor Ion (m/z)	Retention Time (min)	Entrance Potential (V)	First Transition			Second Transition		
				Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Positive ESI Mode									
2-Amino-flubendazole	256.0	3.51	9	123.0	37	6	95.0	45	14
2-Aminosulfone albendazole	240.0	2.61	10	133.1	40	6	198.0	27	10
2-Quinoxaline carboxylic acid (QCA)	175.0	3.07	4	131.0	23	6	102.0	40	12
Abamectin-Na	895.7	8.17	2	751.4	57	45	449.3	62	25
Acepromazine	327.1	4.25	6	86.0	25	14	222.1	51	12
Albendazole	266.0	4.36	9	234.0	27	12	191.0	45	10
Amoxicillin	366.2	2.13	3	349.0	12	16	114.0	25	18
Ampicillin	350.1	2.72	5	106.0	22	12	160.0	17	12
Azaperone	328.2	3.13	6	165.1	28	10	123.0	44	14

Analyte	Precursor Ion (m/z)	Retention Time (min)	Entrance Potential (V)	First Transition			Second Transition		
				Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Butorphanol	328.2	3.59	5	310.1	32	26	157.1	57	26
Carazolol	299.2	3.67	7	116.1	26	6	194.1	39	10
Carbadox	263.3	2.93	8	229.0	24	14	130.0	28	14
Cefazolin	455.0	3.16	4	323.0	15	16	156.0	23	8
Chlorpromazine	319.0	4.71	6	85.8	25	14	214.0	53	12
Chlortetracycline	479.0	3.51	5	444.0	29	20	154.0	37	6
Cimaterol	220.1	2.16	6	143.1	31	6	89.0	56	10
Ciprofloxacin	332.1	2.90	6	314.1	27	14	288.1	25	14
Clenbuterol	277.0	3.25	10	202.8	23	22	132.0	39	12
Clindamycin	425.1	3.54	5	377.1	26	20	389.2	22	10
Cloxacillin	436.0	5.24	5	277.0	23	14	160.0	16	8
Danofloxacin	358.1	2.96	10	340.0	31	16	314.0	25	16
DCCD	549.0	2.39	7	183.0	38	10	241.0	29	12
Desacetyl cephalixin	382.3	1.85	10	112.0	31	12	124.0	61	14
Desethylene ciprofloxacin	306.1	2.75	4	288.1	25	16	217.0	47	24
Diclofenac	296.0	6.21	7	215.0	27	12	214.0	45	12
Dicloxacillin	470.0	5.58	7	311.0	19	10	160.0	19	16
Difloxacin	400.1	3.29	10	382.1	31	18	356.0	27	18
Dimetridazole	142.0	2.50	10	96.0	22	16	81.0	33	12
Dimetridazole - OH	158.1	2.27	6	139.9	16	16	111.9	26	12
Dipyrene	218.1	2.30	7	97.0	17	15	187.1	14	10
Doramectin	921.6	8.49	5	777.4	59	40	449.0	64	25
Doxycycline	445.1	3.02	3	428.1	26	24	267.0	50	30
Emamectin benzoate	886.5	6.09	4	158.1	40	10	82.1	124	2
Enrofloxacin	360.1	3.05	8	342.1	29	16	316.1	27	16
Erythromycin A	734.3	4.11	8	576.4	28	26	158.1	38	8
Fenbendazole sulphone	332.0	4.33	7	300.0	31	16	159.0	49	8

Analyte	Precursor Ion (m/z)	Retention Time (min)	Entrance Potential (V)	First Transition			Second Transition		
				Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Fenbendazole	300.0	5.01	5	268.0	29	14	159.0	47	8
Florfenicol amine	248.0	1.42	8	230.1	15	12	130.1	31	6
Florfenicol	358.0	3.81	6	241.0	23	12	206.1	34	12
Flubendazole	314.0	4.58	6	282.1	31	14	122.8	47	6
Flunixin	297.1	5.58	3.5	259.1	39	14	236.1	59	14
Flunixin-d ₃	300.0	5.55	7	282.0	33	14	264.1	47	14
Gamithromycin	777.4	3.62	5	619.4	45	26	601.4	49	26
Haloperidol	376.1	4.27	10	165.1	32	8	123.0	43	6
Iprnidazole	170.0	3.91	5	124.1	24	8	109	32	18
Iprnidazole - OH	186.1	3.22	3	106	49	12	168.1	17	14
Ivermectin	897.3	8.14	2	753.6	59	30	329.2	69	16
Ketamine	238.1	2.96	4	125.0	35	11	220.0	20	12
Ketoprofen	255.0	5.36	9	105.0	28	12	194.0	31	16
Levamisole	205.0	2.56	10	178.1	30	8	123.0	38	13
Lincomycin	407.1	2.53	2	126.1	33	14	359.0	24	18
Melengesterol acetate	397.2	6.74	8	337.3	19	16	279.2	27	14
Meloxicam	352.0	5.55	2	115.0	23	8	141.0	25	6
Metronidazole	172.1	2.27	11	128.0	16	14	82.1	31	10
Metronidazole - OH	188.0	2.01	3	123.1	16	6	126.0	22	6
Monensin	693.4	9.30	8	461.3	68	10	479.3	74	13
Morantel	221.0	3.31	6	123.0	47	6	111.0	33	18
Moxidectin	640.4	8.69	7	528.3	13	40	498.4	17	30
Nafcillin	415.1	5.33	5	199.0	17	11	171.0	48	19
Narasin	787.5	9.54	13	431.2	69	7	531.2	65	13
Norfloxacin	320.1	2.84	6	302.0	27	16	233.1	33	12
Orbifloxacin	396.2	3.10	7	352.1	25	20	295.0	32	16
Oxacillin	402.0	4.95	8	243.0	19	12	160.0	19	8

Analyte	Precursor Ion (m/z)	Retention Time (min)	Entrance Potential (V)	First Transition			Second Transition		
				Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Oxyphenylbutazone	325.1	5.41	6	204.1	21	8	120.1	23	6
Oxytetracycline	461.1	2.87	4	426.1	24	20	443.1	18	20
Penicillin G	335.0	4.47	6	160.0	13	10	176.1	16	10
Penicillin G-d ₇	342.2	4.44	6	160.0	14	10	183.0	17	10
Phenylbutazone	309.1	6.47	4	188.1	23	10	120.1	25	8
Pirlimycin	411.1	3.36	2	112.0	32	6	363.1	23	18
Prednisone	359.1	4.19	3	147.1	31	8	171.0	39	8
Promethazine	285.0	4.27	5	86.0	21	10	71.1	61	12
Propionylpromazine	341.1	4.56	2	86.2	25	12	236.0	49	12
Ractopamine	302.1	2.93	7	164.0	22	8	121.1	29	10
Ronidazole	201.0	2.53	2	140.0	15	15	54.1	58	8
Salbutamol	240.2	2.07	10	148.0	23	8	166.1	18	6
Salinomycin	773.3	9.21	4	431.1	71	10	531.4	63	8
Sarafloxacin	386.1	3.25	6	368.0	31	18	342.1	27	16
Selamectin	770.3	8.89	5	626.3	25	26	608.4	29	6
SMZ- ¹³ C ₆	285.0	3.19	7	186.1	23	10	124.1	31	8
Sulfachloropyridazine	285.0	3.59	8	156.0	21	8	92.0	37	14
Sulfadiazine	251.0	2.61	9	156.0	21	8	92.0	33	11
Sulfadimethoxine	311.1	4.22	6	156.0	27	8	108.0	35	18
Sulfadoxine	311.0	3.72	6	155.9	25	8	107.8	33	16
Sulfaethoxypyridazine	295.0	3.78	6	156.0	25	8	140.1	25	8
Sulfamerazine	265.0	2.96	8	156.0	22	8	172.0	22	9
Sulfamethazine	279.0	3.19	7	186.0	23	10	124.1	30	6
Sulfamethizole	270.9	3.19	8	156.0	19	8	92.0	37	11
Sulfamethoxazole	254.0	3.75	8	156.0	21	8	92.0	35	14
Sulfamethoxypridazine	281.0	3.22	6	156.0	22	8	108.0	31	14
Sulfanilamide	173.0	1.55	10	93.0	29	10	65.0	40	12

Analyte	Precursor Ion (m/z)	Retention Time (min)	Entrance Potential (V)	First Transition			Second Transition		
				Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Sulfapyridine	250.1	2.81	8	156.0	22	8	184.0	23	10
Sulfaquinoxaline	301.0	4.22	6	156.0	23	8	108.0	33	16
Sulfathiazole	256.0	3.75	8	92.0	37	14	108.0	21	14
Sulfisoxazole	268.2	3.91	10	155.9	18	10	113.0	20	8
Tetracycline	445.1	3.02	4	410.0	27	20	427.1	19	23
Thiabendazole	202.1	2.64	7	175.0	35	18	131.1	44	14
Tildipirosin	368.0	2.56	5	98.1	23	11	464.3	23	28
Tilmicosin	435.4	3.75	3	99.0	26	34	696.0	24	8
Tolfenamic acid	262.0	6.83	5	244.0	17	14	209.1	38	12
Triflupromazine	353.1	4.95	2	86.0	25	14	280.0	35	14
Tulathromycin A	404.0	2.96	10	577.2	19	29	158.1	28	8
Tylosin	916.6	4.25	6	174.0	47	10	772.4	41	32
Tyvalosin	1042.5	5.27	7	174.1	48	12	814.3	45	20
Virginiamycin M1	526.2	5.04	4	355.1	25	14	337.0	29	16
Xylazine	221.1	3.15	6	90.0	35	14	164.0	35	14
Zilpaterol	262.1	2.07	11	185.1	32	10	157.0	41	8
Negative ESI Mode									
Chloramphenicol	321	3.97	-10	151.9	-22	-9	194.0	-16	-9
Lasalocid A	589.4	8.72	-10	234.9	-40	-13	173.0	-54	-11
Sulfantran	334	4.8	-3	135.9	-38	-7	270.0	-34	-13
Zearalanol	321	4.96	-2	277.0	-28	-15	303.1	-28	-21

Table 32: Pesticide Scan Parameters for 1290 Infinity UHPLC with SCIEX 7500 QTrap Activated mass spectrometer

Analyte	Precursor Ion (m/z)	Retention Time (min)	Entrance Potential (V)	First Transition			Second Transition		
				Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Positive ESI Mode									
Acephate	184.1	2.01	10	125.0	23	15	143.0	11	15
Acetamiprid	223.0	3.75	10	126.0	27	6	90.0	51	6
Alachlor	269.8	6.47	10	237.8	11	5	161.9	19	5
Aldicarb.Na	213.0	4.25	10	89.0	21	10	116.0	17	12
Aldicarb sulfone	223.0	2.72	10	148.0	13	8	86.0	19	4
Atrazine	216.0	5.15	10	174.0	25	10	96.0	33	6
Atrazine desethyl	187.9	3.59	10	146.0	20	5	104.0	28	5
Atrazine-d ₅	221.1	5.12	5	179.1	26	10	137.1	31	10
Azinphos methyl	318.0	5.89	8	261.1	11	15	132.1	21	12
Azoxystrobin	404.0	6.04	10	372.0	21	16	344.0	35	16
Benoxacor	259.7	5.99	10	148.9	17	5	120.0	35	5
Boscalid	343.0	6.12	10	307.0	27	6	272.0	43	12
Buprofezin	306.0	6.71	10	201.0	17	10	106.0	33	6
Carbaryl	202.0	5.06	10	127.0	37	6	145.0	15	6
Carbofuran	222.0	4.87	10	165.0	17	10	123.0	29	8
Carbofuran-3-hydroxy	238.1	3.51	10	181.1	15	20	163.1	20	15
Carboxin	236.0	5.18	10	143.0	24	4	87.0	31	6
Carfentrazone ethyl	412.0	6.71	8	365.9	24	26	345.9	15	15
Chlorpyrifos	350.0	7.80	5	198.0	22	25	96.9	45	15
Chlorpyrifos methyl	321.9	7.18	7	125.0	24	15	289.9	21	15
Clofentezine	303.0	7.07	10	138.0	19	8	102.0	51	4
Clothianidin	250.0	3.45	10	169.0	19	4	132.0	23	6
Coumaphos O	347.0	5.60	10	291.0	22	5	211.0	34	5
Coumaphos S	362.8	7.04	10	227.0	35	4	306.9	23	8
Diazinon	305.0	7.04	10	169.0	29	8	153.0	27	8

Analyte	Precursor Ion (m/z)	Retention Time (min)	Entrance Potential (V)	First Transition			Second Transition		
				Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Dichlorvos	220.9	4.58	10	109.0	23	6	145.0	17	12
Difenoconazole	406.0	6.74	10	251.0	33	14	337.0	23	16
Diflubenzuron	311.0	6.29	10	158.0	21	8	141.0	45	8
Dimethoate	230.0	3.67	10	199.0	13	8	125.0	29	6
Diuron	233.0	5.21	10	72.0	21	12	46.0	37	8
Ethion	384.7	7.83	10	198.8	11	5	171.0	25	5
Ethoprophos	243.1	6.18	10	173.0	22	5	215.0	17	12
Fenoxaprop ethyl	361.9	7.34	10	287.7	19	5	121.0	32	5
Fenpyroximate	422.1	7.94	5	366.2	24	20	215.1	38	15
Fluridone	330.0	5.75	10	309.2	33	5	290.1	35	5
Fluroxypyr-1-methylheptyl-ester	367.0	7.99	10	255.0	10	5	209.0	22	5
Fluvalinate	503.0	8.49	6	208.1	17	15	181.0	40	15
Hexazinone	252.9	4.22	10	170.8	17	5	70.9	33	5
Hexythiazox	353.0	7.83	10	228.0	21	10	168.0	33	8
Imazalil	297.0	4.41	10	159.0	37	8	255.0	21	12
Imidacloprid	256.0	3.56	10	175.0	23	12	209.0	27	10
Indoxacarb	528.0	7.23	10	249.0	23	13	293.0	19	6
Linuron	249.0	5.91	10	160.0	25	6	182.0	23	8
Malathion	331.0	6.41	10	127.0	17	6	99.0	31	4
Metalaxyl	280.0	5.15	10	220.0	19	4	192.0	25	8
Methamidophos	141.9	1.70	10	94.0	19	12	125.1	19	4
Methomyl	163.0	2.90	10	88.0	13	8	106.0	13	8
Methoxyfenozide	369.0	6.32	10	149.0	23	12	313.0	11	4
Metolachlor	283.8	6.47	10	252.2	21	25	176.2	35	20
Metribuzin	214.8	4.66	10	186.7	19	5	83.9	21	5
Myclobutanil	289.1	5.99	10	70.1	18	5	125.1	30	5
Norflurazon	304.0	5.33	10	284.0	33	44	160.0	41	28

Analyte	Precursor Ion (m/z)	Retention Time (min)	Entrance Potential (V)	First Transition			Second Transition		
				Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Omethoate	214.0	2.21	10	183.0	15	10	125.0	29	8
Phenothrin (sumithrin)	351.1	8.58	7	183.1	30	20	305.2	16	20
Piperonyl butoxide	356.0	7.57	10	177.0	21	8	119.0	47	6
Pirimiphos methyl	305.9	6.97	10	107.8	33	5	163.9	21	5
Prallethrin	301.1	7.26	10	168.9	9	5	132.9	11	5
Profenofos	373.0	7.31	10	303.0	25	42	345.0	17	12
Propachlor	211.8	5.47	10	169.7	17	5	105.8	25	5
Propanil	218.0	5.68	10	162.0	21	8	127.0	35	6
Propargite	368.1	7.96	5	231.2	15	20	175.2	22	20
Propetamphos	281.9	6.53	10	137.8	19	5	155.7	11	5
Propiconazole	342.0	6.53	10	159.0	33	6	123.0	79	6
Propyzamide (Pronamide)	256.1	6.21	2	190.1	18	20	173.0	29	20
Pyraclostrobin	388.0	7.01	10	194.0	17	4	163.0	33	4
Pyrethrin I	329.2	8.06	10	161.0	9	5	143.0	15	5
Pyridaben	365.0	8.23	10	309.0	17	14	117.0	83	8
Pyriproxyfen	322.0	7.67	10	95.8	15	5	184.8	23	5
Resmethrin	339.2	8.34	10	171.1	20	20	128.0	60	15
Simazine	202.0	4.50	10	124.0	25	6	132.0	25	4
Spirodiclofen	411.1	8.29	7	313.1	16	25	71.1	35	12
Sulprofos	323.0	7.88	10	219.0	17	6	155.0	37	4
Tebufenozide	353.2	6.63	10	297.1	11	8	133.1	25	6
Tetrachlorvinphos	367.0	6.41	10	127.0	19	4	206.0	51	10
Tetraconazole	372.0	6.15	10	159.0	37	8	70.0	63	6
Thiamethoxam	292.0	3.13	10	211.0	17	4	132.0	27	6
Thiobencarb	257.9	7.10	10	125.1	20	5	100.1	10	5
Tribufos (DEF6)	315.1	8.34	5	169.0	22	15	225.0	17	15
Trifloxystrobin	409.0	7.31	10	186.0	25	8	206.0	19	4

Analyte	Precursor Ion (m/z)	Retention Time (min)	Entrance Potential (V)	First Transition			Second Transition		
				Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)	Product Ion (m/z)	Collision Energy (V)	Cell Exit Potential (V)
Negative ESI Mode									
Fipronil	434.8	6.69	-5	249.9	-38	-13	399.0	-16	-19
Fipronil desulfinyl	387.0	6.86	-10	351	-18	-17	282.0	-44	-15
Fipronil sulfide	419.0	7.07	-10	383	-18	-17	261.9	-40	-13
Fipronil sulfone	450.9	7.04	-10	415	-23	-20	281.9	-38	-20

Instrument Note

All chromatographic and instrument parameters were optimized in accordance with FSIS laboratory system method performance requirements and during annual preventative maintenance and calibration.

- Retention time windows, collision energies, and selected masses for precursor and product ions were set and utilized at time of method validation.
 - Retention time windows may be adjusted to account for aging of UHPLC columns or for improved separation to ensure that all chromatographic peaks are present.
 - Collision energies may be adjusted and optimized for improved mass spectrometry detection.
 - Target masses for precursor and product ions can be optimized to a m/z value that falls within the unit mass resolution of the exact mass, but not to exceed the next integer value (e.g., if the exact mass is 787.5, an allowable target mass range includes 787.0-787.9).
- Parameter modifications to improve instrument performance to ensure all chromatographic peaks are present must meet the acceptance criteria listed in the method's Quality Assurance Plan.
- Significant changes that affect method performance require equivalency testing and FSIS laboratory leadership approval.

Sample Set

The injection sequence below can be modified, as needed, but must include required controls.

- 1) External standards (optional)
- 2) Blank (negative control)
- 3) Matrix matched controls
- 4) Recoveries (positive controls)
- 5) Intra-laboratory check sample (if applicable)
- 6) Up to 35 samples
- 7) Trailing External standard, matrix matched standard, or recovery (positive control) (for system suitability)

**INTRA-LABORATORY
CHECK SAMPLE**

Defined on the [CLG website](#)

System suitability is to be demonstrated prior to sample set injection.

Reporting of Results

Decision Criteria

Screening

- 1) The screening ion, listed in Tables 29-32 for a given analyte must be present in the matrix match standard at the minimum level of applicability (MLA) (Tables 33-36).
- 2) The retention times for the screening ion in the fortified recoveries must match the retention time of the screening ion in the matrix-matched control within 5%.
- 3) Retention time for the screening ions in the samples must match the retention time of the screening ions in a fortified recovery or the matrix matched standard within 5%.
- 4) The screening ion must have a signal-to-noise ratio ≥ 3 . This will be verified by visual inspection. Visual inspection for detection is to also include at least one of the following:
 - a. Assessment of peak shape or drift in relation to standard peaks
 - b. Evaluating the presence or absence of monitored secondary ion (Listed in Table 29-32).
- 5) The fortified recovery at the MLA must be at least 10% of the matrix matched control at the corresponding level.
- 6) A sample is screened positive if the sample response equals or exceeds the fortified recovery at the MLA.
- 7) The level of the screening ion in the blank (negative control) must be less than 10% of the matrix-matched control at the MLA.
- 8) If a sample shows a positive response for an analyte that did not meet screening criteria in the associated QC samples, then rescreen the sample for that analyte.

QUALITY CONTROL

Quality Control Procedures for Screening Identification

- 1) For set acceptance, 90% of the monitored analytes must meet screening criteria in the recovery (positive control) fortified at the minimum level of applicability (Tables 33-36). For sample reporting purposes, the analytes of interest in the fortified recovery (positive control) must meet screening criteria.
- 2) For set acceptance, 90% of the monitored analytes in the blank (negative control) must not meet screening criteria. The blank (negative control) must be negative using the screening criteria for samples containing corresponding presumptive positive analytes.

Intra-Laboratory Check Samples (If applicable)

- 1) Acceptability criteria.
 - a. 90% of the monitored analytes in a fortified Intra-Laboratory Check must meet screening criteria.
 - b. 90% of the monitored analytes in an unfortified Intra-Laboratory Check must be negative using the screening criteria.
 - c. FSIS Field Service Laboratories are to refer to internal FSIS Quality Control Procedures when unacceptable values are obtained:
 - i. Refer to LW-Q1002, Chemistry Non-Conformance Tables, for how to proceed and whether to take corrections or corrective actions.

Minimum Level of Applicability

Table 33: Veterinary Drug Residue Minimum Level of Applicability (MLA) for Muscle Screening

Analyte	Bovine		Porcine		Poultry		Caprine		Ovine		<i>Siluriformes</i>	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Analgesics												
Diclofenac	1/2 X	5	N/A	N/A	1/2 X	5	1/2 X	5	1/2 X	5	2 X	20
Dipyron	1/2 X	25	1/2 X	25	1 X	50	1/2 X	25	2 X	100	1/2 X	25
Flunixin	1/2 X	12.5	1/2 X	12.5	1/2 X	12.5	1/2 X	12.5	1/2 X	12.5	1/2 X	12.5
Ketoprofen	1/2 X	5	1/2 X	5	1/2 X	5	1/2 X	5	1/2 X	5	1/2 X	5
Meloxicam	N/A	N/A	1/2 X	10	1/2 X	10	N/A	N/A	1/2 X	10	1/2 X	10
Oxyphenylbutazone	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Phenylbutazone	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Tolfenamic acid	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25
Avermectins												
Abamectin	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25
Doramectin	1/2 X	7.5	1 X	15	N/A	N/A	1/2 X	7.5	1/2 X	7.5	2 X	30
Emamectin benzoate	1/2 X	7.5	1/2 X	7.5	1/2 X	7.5	1/2 X	7.5	1/2 X	7.5	1/2 X	7.5
Ivermectin	1 X	15	2 X	30	1 X	15	1/2 X	7.5	2 X	30	N/A	N/A
Moxidectin	N/A	N/A	1/2 X	7.5	N/A	N/A	N/A	N/A	N/A	N/A	2 X	30
Selamectin	1 X	15	2 X	30	N/A	N/A	1/2 X	7.5	1 X	15	2 X	30
Beta-Agonists												
Cimaterol	1/2 X	3	1/2 X	3	1/2 X	3	1/2 X	3	2 X	12	1/2 X	3
Clenbuterol	1/2 X	3	1/2 X	3	1/2 X	3	2 X	12	1/2 X	3	1/2 X	3
Ractopamine	1/2 X	3	1/2 X	3	1/2 X	3	1/2 X	3	1/2 X	3	1/2 X	3
Salbutamol	1/2 X	3	1/2 X	3	1/2 X	3	1/2 X	3	1/2 X	3	1/2 X	3
Zilpaterol	1 X	6	1/2 X	3	1/2 X	3	1 X	6	2 X	12	1/2 X	3
Beta-Lactams												
Amoxicillin	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20
Ampicillin	1/2 X	5	1/2 X	5	1/2 X	5	1/2 X	5	1/2 X	5	1/2 X	5
Cefazolin	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Cloxacillin	1/2 X	5	1/2 X	5	1/2 X	5	1/2 X	5	1/2 X	5	1/2 X	5
DCCD	1/2 X	50	1/2 X	50	1 X	100	1/2 X	50	1/2 X	50	N/A	N/A
Desacetyl cephalosporin	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Dicloxacin	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Nafcillin	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Oxacillin	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Penicillin G	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25
N/A = Not Applicable												

Analyte	Bovine		Porcine		Poultry		Caprine		Ovine		Siluriformes	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Benzimidazoles												
2-Amino-flubendazole	1/2 X	10	1/2 X	10	1/2 X	10	1/2 X	10	1/2 X	10	1/2 X	10
2-Aminosulfone albendazole	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25
Albendazole	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25
Fenbendazole	1/2 X	200	1/2 X	200	1/2 X	200	1/2 X	200	1/2 X	200	1/2 X	200
Fenbendazole sulphone	1/2 X	200	1/2 X	200	1/2 X	200	1/2 X	200	1/2 X	200	1/2 X	200
Flubendazole	1/2 X	10	1/2 X	10	1/2 X	10	1/2 X	10	1/2 X	10	1/2 X	10
Thiabendazole	2 X	200	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Fluoroquinolones												
Ciprofloxacin	1/2 X	25	1/2X	25	1/2 X	25	1/2X	25	1/2 X	25	1/2 X	25
Danofloxacin	1/2 X	25	1/2X	25	1/2 X	25	1/2X	25	1/2 X	25	1/2 X	25
Desethylene ciprofloxacin	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25
Difloxacin	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25
Enrofloxacin	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25
Norfloxacin	1 X	50	1/2 X	25	1/2 X	25	1/2 X	25	1 X	50	1/2 X	25
Orbifloxacin	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25
Sarafloxacin	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25
General Drugs												
2-Quinoxaline carboxylic acid (QCA)	1/2 X	15	1/2X	15	1/2 X	15	1/2X	15	1/2 X	15	1/2 X	15
Carbadox	1/2 X	15	1/2X	15	1/2 X	15	1/2X	15	1 X	30	1/2 X	15
Levamisole	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Morantel tartrate	1/2 X	350	1/2X	350	1/2 X	350	1/2X	350	1/2 X	350	1/2 X	350
Hormones												
Melengestrol acetate	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20
Prednisone	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Taleranol (B-Zearalanol)	1/2 X	12	1/2 X	12	1 X	24	N/A	N/A	1/2 X	12	1/2 X	12
Macrolides												
Clindamycin	1/2 X	50	1/2X	50	1/2 X	50	1/2X	50	1/2 X	50	1/2 X	50
Erythromycin A	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Gamithromycin	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Lincomycin	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Pirlimycin	1/2 X	250	1/2 X	250	1/2 X	250	1/2 X	250	1/2 X	250	1/2 X	250
Tildipirosin	1/2 X	500	1/2 X	500	1/2 X	500	1/2 X	500	1/2 X	500	1/2 X	500
Tilmicosin	1/2 X	60	1/2 X	60	1/2 X	60	1/2 X	60	1/2 X	60	1/2 X	60
Tulathromycin A	1/2 X	1000	1/2 X	1000	N/A	N/A	1/2 X	1000	1/2 X	1000	1/2 X	1000
Tylosin	1/2 X	100	1/2 X	100	1/2 X	100	1/2 X	100	1/2 X	100	1/2 X	100
Tylvalosin	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25
Virginiamycin	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
N/A = Not Applicable												

Analyte	Bovine		Porcine		Poultry		Caprine		Ovine		Siluriformes	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Nitroimidazoles												
Dimetridazole	N/A	N/A	1 X	2	N/A	N/A	N/A	N/A	N/A	N/A	2 X	4
Dimetridazole - OH	1/2 X	50	1/2 X	50	1 X	100	1/2 X	50	2 X	200	1/2 X	50
Iprnidazole	2 X	4	1 X	2	N/A	N/A	N/A	N/A	N/A	N/A	2 X	4
Iprnidazole - OH	N/A	N/A	1 X	2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Metronidazole	N/A	N/A	1/2 X	1	N/A	N/A	1 X	2	N/A	N/A	1/2 X	1
Metronidazole - OH	1/2 X	4	1/2 X	4	1 X	8	1/2 X	4	2 X	16	1/2 X	4
Ronidazole	N/A	N/A	1 X	2	N/A	N/A	N/A	N/A	N/A	N/A	1 X	2
Phenolics												
Chloramphenicol	1/2 X	3	1/2 X	3	2 X	12	1 X	6	1/2 X	3	1 X	6
Florfenicol	1/2 X	100	1/2 X	100	1/2 X	100	1/2 X	100	N/A	N/A	1/2 X	100
Florfenicol amine	1/2 X	150	1/2 X	150	1/2 X	150	2 X	150	1/2 X	150	1/2 X	150
Sulfas												
Sulfachloropyridazine	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfadiazine	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfadimethoxine	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfadoxine	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfaethoxypyridazine	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfamerazine	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfamethazine	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfamethizole	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfamethoxazole	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfamethoxypyridazine	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfanilamide	1/2 X	50	1/2 X	50	2 X	200	1/2 X	50	1 X	100	1/2 X	50
Sulfantran	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfapyridine	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfaquinoxaline	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50	1/2 X	50
Sulfathiazole	1/2 X	50	1/2 X	50	1 X	100	1/2 X	50	1/2 X	50	1/2 X	50
Sulfisoxazole	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	50	1/2 X	20
Tetracyclines												
Chlortetracycline	1/2 X	1000	1/2 X	1000	1/2 X	1000	1/2 X	1000	1/2 X	1000	1/2 X	1000
Doxycycline	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25	1/2 X	25
Oxytetracycline	1 X	1000	1 X	1000	1 X	1000	1 X	1000	1 X	1000	1 X	1000
Tetracycline	1/2 X	500	1/2 X	500	1/2 X	500	1/2 X	500	1/2 X	500	1/2 X	500
N/A = Not Applicable												

Analyte	Bovine		Porcine		Poultry		Caprine		Ovine		Siluriformes	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Tranquilizers												
Acepromazine	1/2 X	4	1/2 X	4	1/2 X	4	1/2 X	4	1/2 X	4	1/2 X	4
Azaperone	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1 X	1
Butorphanol	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1
Carazolol	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1
Chlorpromazine	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1
Haloperidol	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1
Ketamine	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20
Promethazine	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1
Propionylpromazine	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1
Triflupromazine	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1
Xylazine	1/2 X	1	N/A	N/A	1/2 X	1	1/2 X	1	1/2 X	1	1/2 X	1
Ionophores												
Lasalocid A	N/A	N/A	2 X	8	N/A	N/A	N/A	N/A	N/A	N/A	2 X	8
Monensin	1/2 X	20	1/2X	20	1/2 X	20	1/2X	20	1/2 X	20	1/2 X	20
Narasin	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20
Salinomycin	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20	1/2 X	20
N/A = Not Applicable												

Table 34: Pesticides Minimum Level of Applicability (MLA) for Muscle Screening

Analyte	Bovine		Porcine		Poultry		Caprine		Ovine		Siluriformes	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Carbamates												
Aldicarb	1 X	10	2 X	20	2 X	20	1 X	10	1 X	10	2 X	20
Aldicarb sulfone	N/A	N/A	1 X	10	N/A	N/A	N/A	N/A	N/A	N/A	1 X	10
Carbaryl	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Carbofuran	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Carbofuran-3-hydroxy	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Methomyl	1 X	30	1 X	30	2 X	60	1 X	30	1 X	30	1 X	30
Thiobencarb	1 X	100	1 X	100	1 X	100	1 X	100	1 X	100	1 X	100
Conazoles / Triazoles												
Difenoconazole	1 X	15	1 X	15	1 X	15	1 X	15	1 X	15	1 X	15
Myclobutanil	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Propiconazole	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Tetraconazole	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
N/A = Not Applicable												

Analyte	Bovine		Porcine		Poultry		Caprine		Ovine		Siluriformes	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Halogenated Pesticides												
Alachlor	1 X	10	1 X	10	1 X	10	2 X	20	1 X	10	2 X	20
Boscalid	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
Carfentrazone ethyl	1 X	50	1 X	50	1 X	50	1 X	50	1 X	50	1 X	50
Diflubenzuron	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Linuron	1 X	50	1 X	50	1 X	50	1 X	50	1 X	50	1 X	50
Norflurazon	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
Pronamide	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Propachlor	2 X	20	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
Propanil	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Neonicotinoids												
Acetamiprid	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Clothianidin	1 X	5	2 X	10	2 X	10	2 X	10	2 X	10	2 X	10
Imidacloprid	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
Thiamethoxam	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
Organophosphates												
Acephate	N/A	N/A	1 X	40	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Azinphos methyl	N/A	N/A	1 X	10	N/A	N/A	N/A	N/A	N/A	N/A	1 X	10
Chlorpyrifos	1 X	7.5	1 X	7.5	1 X	7.5	1 X	7.5	1 X	7.5	1 X	7.5
Chlorpyrifos methyl	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Coumaphos O	1 X	400	1 X	400	1 X	400	1 X	400	1 X	400	1 X	400
Coumaphos S	1 X	500	1 X	500	1 X	500	1 X	500	1 X	500	1 X	500
Diazinon	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
Dichlorvos (DDVP)	1 X	5	1 X	5	2 X	10	1 X	5	2 X	10	1 X	5
Dimethoate	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
Ethion	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
Malathion	1 X	50	1 X	50	1 X	50	1 X	50	1 X	50	1 X	50
Methamidophos	1 X	10	1 X	10	1 X	10	N/A	N/A	2 X	20	1 X	10
Omethoate	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
Pirimiphos methyl	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Profenofos	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
Propetamphos	N/A	N/A	1 X	7.5	2 X	15	2 X	15	N/A	N/A	2 X	15
Sulprofos	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Tetrachlorvinphos	1 X	100	1 X	100	1 X	100	1 X	100	1 X	100	1 X	100
N/A = Not Applicable												

Analyte	Bovine		Porcine		Poultry		Caprine		Ovine		Siluriformes	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
General Pesticides												
Azoxystrobin	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Benoxacor	2 X	10	1 X	5	1 X	5	1 X	5	1 X	5	2 X	10
Buprofezin	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Carboxin	1 X	7.5	1 X	7.5	1 X	7.5	1 X	7.5	1 X	7.5	1 X	7.5
Clofentezine	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Diuron	1 X	500	1 X	500	1 X	500	1 X	500	1 X	500	1 X	500
Fenoxaprop ethyl	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Fenpyroximate	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Fipronil	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Fipronil desulfinyl	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Fipronil sulfide	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Fipronil sulfone	1 X	5	1 X	5	2 X	10	1 X	5	1 X	5	1 X	5
Fluridone	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Fluroxypyr-1-methylheptyl-ester	1 X	5	1 X	5	2 X	10	1 X	5	1 X	5	1 X	5
Hexazinone	1 X	50	1 X	50	1 X	50	1 X	50	1 X	50	1 X	50
Hexythiazox	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Imazalil	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Indoxacarb	1 X	15	1 X	15	1 X	15	1 X	15	1 X	15	1 X	15
Metalaxyl	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Methoxyfenozide	2 X	10	1 X	5	2 X	10	2 X	10	1 X	5	1 X	5
Metolachlor	1 X	10	1 X	10	2 X	20	1 X	10	1 X	10	1 X	10
Metribuzin	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Piperonyl butoxide	1 X	50	1 X	50	1 X	50	1 X	50	1 X	50	1 X	50
Propargite	1 X	5	1 X	5	2 X	10	2 X	10	1 X	5	1 X	5
Pyraclostrobin	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Pyridaben	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Pyriproxyfen	1 X	50	1 X	50	1 X	50	1 X	50	1 X	50	1 X	50
Spirodiclofen	N/A	N/A	1X	20	N/A	N/A	N/A	N/A	N/A	N/A	1X	20
Tebufenozide	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25	1 X	25
Tribufos (DEF6)	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5	1 X	5
Trifloxystrobin	1 X	20	1 X	20	1 X	20	1 X	20	1 X	20	1 X	20
N/A = Not Applicable												

Analyte	Bovine		Porcine		Poultry		Caprine		Ovine		Siluriformes	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Pyrethroids												
Fluvalinate	1 X	7.5	1 X	7.5	2 X	15	1 X	15	1 X	7.5	1 X	7.5
Phenothrin	1 X	20	1 X	20	2 X	40	N/A	N/A	N/A	N/A	2 X	40
Prallethrin	1 X	500	1 X	500	1 X	500	1 X	500	1 X	500	1 X	500
Pyrethrin I	1 X	60	1 X	60	1 X	60	1 X	60	1 X	28	1 X	60
Resmethrin (cis&trans)	1 X	500	1 X	500	1 X	500	1 X	500	1 X	500	1 X	500
Triazines												
Atrazine	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
Atrazine-Desethyl	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10	1 X	10
Simazine	1 X	15	1 X	15	1 X	15	1 X	15	1 X	15	1 X	15
N/A = Not Applicable												

Table 35: Veterinary Drug Residue Minimum Level of Applicability (MLA) for Liquid Egg Product Screening

Analyte	Whole Egg		Egg Yolk		Egg White	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Analgesics						
Diclofenac	1/2 X	5	1/2 X	5	1/2 X	5
Dipyron	1/2 X	25	1/2 X	25	1 X	50
Flunixin	1/2 X	12.5	1/2 X	12.5	1/2 X	12.5
Ketoprofen	1/2 X	5	1 X	10	1/2 X	5
Meloxicam	1/2 X	10	1/2 X	10	1/2 X	10
Oxyphenylbutazone	1/2 X	50	1/2 X	50	1/2 X	50
Phenylbutazone	1/2 X	50	1/2 X	50	1/2 X	50
Tolfenamic acid	1/2 X	25	1/2 X	25	1/2 X	25
Avermectins						
Abamectin	2 X	100	1/2 X	25	1/2 X	25
Doramectin	1 X	15	1 X	15	1/2 X	7.5
Emamectin benzoate	1/2 X	7.5	1/2 X	7.5	1/2 X	7.5
Ivermectin	N/A	N/A	N/A	N/A	1 X	15
Moxidectin	1/2 X	7.5	N/A	N/A	N/A	N/A
Selamectin	N/A	N/A	2 X	30	1 X	15
Beta-Agonists						
Cimaterol	1/2 X	3	1/2 X	3	1/2 X	3
Clenbuterol	1/2 X	3	1/2 X	3	1/2 X	3
Ractopamine	1/2 X	3	1/2 X	3	1/2 X	3
Salbutamol	1/2 X	3	1/2 X	3	1/2 X	3
Zilpaterol	1/2 X	3	1/2 X	3	1/2 X	3
N/A = Not Applicable						

Analyte	Whole Egg		Egg Yolk		Egg White	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Beta-Lactams						
Amoxicillin	1/2 X	20	1/2 X	20	1/2 X	20
Ampicillin	1/2 X	5	1/2 X	5	1/2 X	5
Cefazolin	1/2 X	50	1/2 X	50	1/2 X	50
Cloxacillin	1/2 X	5	1 X	10	1/2 X	5
DCCD	1/2 X	50	1/2 X	50	2 X	200
Desacetyl cephalixin	1/2 X	50	1/2 X	50	1/2 X	50
Dicloxacin	1/2 X	50	1/2 X	50	1/2 X	50
Nafcillin	1/2 X	50	1/2 X	50	1/2 X	50
Oxacillin	1/2 X	50	1/2 X	50	1/2 X	50
Penicillin G	1/2 X	25	1/2 X	25	1/2 X	25
Benzimidazoles						
2-Amino-flubendazole	1/2 X	10	1/2 X	10	1/2 X	10
2-Aminosulfone albendazole	1/2 X	25	1/2 X	25	1/2 X	25
Albendazole	1/2 X	25	1/2 X	25	1/2 X	25
Fenbendazole	1/2 X	200	1/2 X	200	1/2 X	200
Fenbendazole sulphone	1/2 X	200	1/2 X	200	1/2 X	200
Flubendazole	1/2 X	10	1/2 X	10	1/2 X	10
Thiabendazole	1/2 X	50	1/2 X	50	1/2 X	50
Fluoroquinolones						
Ciprofloxacin	1/2 X	25	1/2 X	25	1/2 X	25
Danofloxacin	1/2 X	25	1/2 X	25	1/2 X	25
Desethylene ciprofloxacin	1/2 X	25	1/2 X	25	1/2 X	25
Difloxacin	1/2 X	25	1/2 X	25	1/2 X	25
Enrofloxacin	1/2 X	25	1/2 X	25	1/2 X	25
Norfloxacin	1/2 X	25	1 X	50	1/2 X	25
Orbifloxacin	1/2 X	25	1/2 X	25	1/2 X	25
Sarafloxacin	1/2 X	25	1 X	50	1 X	50
General Drugs						
2-Quinoxaline carboxylic acid (QCA)	1/2 X	15	1 X	30	1/2 X	15
Carbadox	1/2 X	15	1 X	30	1/2 X	15
Levamisole	1/2 X	50	1/2 X	50	1/2 X	50
Morantel tartrate	1/2 X	350	1/2 X	350	1/2 X	350
Hormones						
Melengestrol acetate	1/2 X	20	1/2 X	20	1/2 X	20
Prednisone	1/2 X	50	1/2 X	50	1/2 X	50
Taleranol (B-Zearalanol)	1/2 X	12	2 X	48	1/2 X	12
N/A = Not Applicable						

Analyte	Whole Egg		Egg Yolk		Egg White	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Macrolides						
Clindamycin	1/2 X	50	1/2 X	50	1/2 X	50
Erythromycin A	1/2 X	50	1/2 X	50	1/2 X	50
Gamithromycin	1/2 X	50	1/2 X	50	1/2 X	50
Lincomycin	1/2 X	50	1/2 X	50	1/2 X	50
Pirlimycin	1/2 X	250	1/2 X	250	1/2 X	250
Tildipirosin	1/2 X	500	1/2 X	500	1/2 X	500
Tilmicosin	1/2 X	60	1/2 X	60	1/2 X	60
Tulathromycin A	1/2 X	1000	1/2 X	1000	1/2 X	1000
Tylosin	1/2 X	100	1 X	200	2 X	400
Tylvalosin	1/2 X	25	1/2 X	25	1/2 X	25
Virginiamycin	1/2 X	50	1/2 X	50	1/2 X	50
Nitroimidazoles						
Dimetridazole	1 X	2	N/A	N/A	N/A	N/A
Dimetridazole - OH	1/2 X	50	1/2 X	50	1/2 X	50
Ipronidazole	1/2X	1	N/A	N/A	2 X	4
Ipronidazole - OH	1 X	2	N/A	N/A	N/A	N/A
Metronidazole	1/2X	1	2 X	4	1 X	2
Metronidazole - OH	1/2 X	4	1/2 X	4	1/2 X	4
Ronidazole	1/2 X	1	N/A	N/A	N/A	N/A
Phenicol						
Chloramphenicol	1/2 X	3	1 X	6	2 X	12
Florfenicol	1/2 X	100	1/2 X	100	1/2 X	100
Florfenicol amine	1/2 X	150	1/2 X	150	1/2 X	150
Sulfas						
Sulfachloropyridazine	1/2 X	50	1/2 X	50	1/2 X	50
Sulfadiazine	1/2 X	50	1/2 X	50	1/2 X	50
Sulfadimethoxine	1/2 X	50	1/2 X	50	1/2 X	50
Sulfadoxine	1/2 X	50	1/2 X	50	1/2 X	50
Sulfaethoxypyridazine	1/2 X	50	1/2 X	50	1/2 X	50
Sulfamerazine	1/2 X	50	1/2 X	50	1/2 X	50
Sulfamethazine	1/2 X	50	1/2 X	50	1/2 X	50
Sulfamethizole	1/2 X	50	1/2 X	50	1/2 X	50
Sulfamethoxazole	1/2 X	50	1/2 X	50	1/2 X	50
Sulfamethoxypyridazine	1/2 X	50	1/2 X	50	1/2 X	50
Sulfanilamide	1/2 X	50	1/2 X	50	1/2 X	50
Sulfanitran	1/2 X	50	1/2 X	50	1/2 X	50
Sulfapyridine	1/2 X	50	1/2 X	50	1/2 X	50
Sulfaquinoxaline	1/2 X	50	1/2 X	50	1/2 X	50
Sulfathiazole	1/2 X	50	1/2 X	50	1/2 X	50
Sulfisoxazole	1/2 X	20	1/2 X	20	1/2 X	20
N/A = Not Applicable						

Tetracyclines						
Chlortetracycline	N/A	N/A	N/A	N/A	N/A	N/A
Doxycycline	N/A	N/A	N/A	N/A	N/A	N/A
Oxytetracycline	2 X	2000	1 X	1000	1/2 X	500
Tetracycline	N/A	N/A	N/A	N/A	N/A	N/A
Tranquilizers						
Acepromazine	1/2 X	4	1/2 X	4	1/2 X	4
Azaperone	1/2 X	1	1/2 X	1	1/2 X	1
Butorphanol	1/2 X	1	1/2 X	1	1/2 X	1
Carazolol	1/2 X	1	1/2 X	1	1/2 X	1
Chlorpromazine	1/2 X	1	1/2 X	1	1/2 X	1
Haloperidol	1/2 X	1	1/2 X	1	1/2 X	1
Ketamine	1/2 X	20	1/2 X	20	1/2 X	20
Promethazine	1/2 X	1	1/2 X	1	1/2 X	1
Propionylpromazine	1/2 X	1	1/2 X	1	1/2 X	1
Triflupromazine	1/2 X	1	1 X	2	1/2 X	2
Xylazine	1/2 X	1	1/2 X	1	1/2 X	1
Ionophores						
Lasalocid A	2 X	8	N/A	N/A	N/A	N/A
Monensin	1/2 X	20	1/2 X	20	1/2 X	20
Narasin	1/2 X	20	1/2 X	20	1/2 X	20
Salinomycin	1/2 X	20	1/2 X	20	1/2 X	20
N/A = Not Applicable						

Table 36: Pesticide Minimum Level of Applicability (MLA) for Liquid Egg Product Screening

Analyte	Whole Egg		Egg Yolk		Egg White	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Carbamates						
Aldicarb	1 X	10	1 X	10	1 X	10
Aldicarb sulfone	1 X	10	N/A	N/A	N/A	N/A
Carbaryl	1 X	25	1 X	25	1 X	25
Carbofuran	1 X	25	1 X	25	1 X	25
Carbofuran-3-hydroxy	1 X	25	1 X	25	1 X	25
Methomyl	1 X	30	1 X	30	1 X	30
Thiobencarb	1 X	100	1 X	100	1 X	100
Conazoles / Triazoles						
Difenoconazole	1 X	15	1 X	15	1 X	15
Myclobutanil	1 X	5	1 X	5	1 X	5
Propiconazole	1 X	5	1 X	5	1 X	5
Tetraconazole	1 X	10	1 X	10	1 X	10
N/A = Not Applicable						

Analyte	Whole Egg		Egg Yolk		Egg White	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Halogenated Pesticides						
Alachlor	2 X	20	2 X	20	N/A	N/A
Boscalid	1 X	10	1 X	10	1 X	10
Carfentrazone ethyl	1 X	50	1 X	50	1 X	50
Diflubenzuron	1 X	25	1 X	25	1 X	25
Linuron	1 X	50	1 X	50	1 X	50
Norflurazon	1 X	10	1 X	10	1 X	10
Pronamide	1 X	5	1 X	5	1 X	5
Propachlor	1 X	10	1 X	10	1 X	10
Propanil	1 X	25	1 X	25	1 X	25
Neonicotinoids						
Acetamiprid	1 X	5	1 X	5	2 X	10
Clothianidin	1 X	5	1 X	5	2 X	10
Imidacloprid	1 X	10	1 X	10	1 X	10
Thiamethoxam	1 X	10	1 X	10	1 X	10
Organophosphates						
Acephate	2 X	80	N/A	N/A	N/A	N/A
Azinphos methyl	1 X	10	N/A	N/A	N/A	N/A
Chlorpyrifos	1 X	7.5	1 X	7.5	1 X	7.5
Chlorpyrifos methyl	1 X	5	1 X	5	1 X	5
Coumaphos O	1 X	400	1 X	400	1 X	400
Coumaphos S	1 X	500	1 X	500	1 X	500
Diazinon	1 X	10	1 X	10	1 X	10
Dichlorvos (DDVP)	1 X	5	1 X	5	1 X	5
Dimethoate	1 X	10	1 X	10	1 X	10
Ethion	1 X	10	1 X	10	1 X	10
Malathion	1 X	50	1 X	50	1 X	50
Methamidophos	1 X	10	1 X	10	1 X	10
Omethoate	1 X	10	1 X	10	1 X	10
Pirimiphos methyl	1 X	5	1 X	5	1 X	5
Profenofos	1 X	10	1 X	10	1 X	10
Propetamphos	1 X	7.5	N/A	N/A	2 X	15
Sulprofos	1 X	25	1 X	25	1 X	25
Tetrachlorvinphos	1 X	100	1 X	100	1 X	100
N/A = Not Applicable						

Analyte	Whole Egg		Egg Yolk		Egg White	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
General Pesticides						
Azoxystrobin	1 X	5	1 X	5	1 X	5
Benoxacor	2 X	10	N/A	N/A	1 X	5
Buprofezin	1 X	25	1 X	25	1 X	25
Carboxin	1 X	7.5	1 X	7.5	1 X	7.5
Clofentezine	1 X	25	1 X	25	1 X	25
Diuron	1 X	500	1 X	500	1 X	500
Fenoxaprop ethyl	1 X	25	1 X	25	1 X	25
Fenpyroximate	1 X	5	1 X	5	1 X	5
Fipronil	1 X	5	1 X	5	1 X	5
Fipronil desulfinyl	1 X	5	1 X	5	1 X	5
Fipronil sulfide	1 X	5	1 X	5	1 X	5
Fipronil sulfone	1 X	5	1 X	5	1 X	5
Fluridone	1 X	25	1 X	25	1 X	25
Fluroxypyr-1-methylheptyl-ester	1 X	5	1 X	5	2 X	10
Hexazinone	1 X	50	1 X	50	1 X	50
Hexythiazox	1 X	25	1 X	25	1 X	25
Imazalil	1 X	5	1 X	5	1 X	5
Indoxacarb	1 X	15	1 X	15	1 X	15
Metalaxyl	1 X	25	1 X	25	1 X	25
Methoxyfenozide	1 X	5	2 X	10	2 X	10
Metolachlor	1 X	10	1 X	10	1 X	10
Metribuzin	1 X	5	1 X	5	1 X	5
Piperonyl butoxide	1 X	50	2 X	100	1 X	50
Propargite	2 X	10	2 X	10	NA	NA
Pyraclostrobin	1 X	25	1 X	25	1 X	25
Pyridaben	1 X	25	1 X	25	1 X	25
Pyriproxyfen	1 X	50	1 X	50	1 X	50
Spirodiclofen	1 X	20	N/A	N/A	N/A	N/A
Tebufenozide	1 X	25	1 X	25	1 X	25
Tribufos (DEF6)	1 X	5	1 X	5	1 X	5
Trifloxystrobin	1 X	20	1 X	20	1 X	20
N/A = Not Applicable						

Analyte	Whole Egg		Egg Yolk		Egg White	
	Level	Conc. (ng/g)	Level	Conc. (ng/g)	Level	Conc. (ng/g)
Pyrethroids						
Fluvalinate	1 X	7.5	1 X	7.5	1 X	7.5
Phenothrin	1 X	20	1 X	20	N/A	N/A
Prallethrin	1 X	500	1 X	500	1 X	500
Pyrethrin I	1 X	60	1 X	60	1 X	60
Resmethrin (cis&trans)	1 X	500	1 X	500	1 X	500
Triazines						
Atrazine	1 X	10	1 X	10	1 X	10
Atrazine-Desethyl	1 X	10	1 X	10	1 X	10
Simazine	1 X	15	1 X	15	1 X	15
N/A = Not Applicable						

Safety Hazards

Table 37: Safety Hazards and Recommended Safe Procedures

Procedure Step	Hazard	Recommended Safe Procedures
Acetonitrile, Methanol	Flammable	Keep in well-closed containers away from ignition sources. Avoid contact or prolonged exposure to vapors. Work in fume hood. Keep away from flame or heat.
Formic acid, Sodium hydroxide	Corrosive, Caustic	Wear personal protective equipment, avoid skin contact.
Veterinary Drug and Pesticide Standards	Some individuals may have allergic reactions to veterinary drug compounds, which may cause skin and respiratory irritation. Possible reproductive toxicity.	Wear personal protective equipment, avoid skin contact. Handle with extreme caution. Work in a well-ventilated area.

References

- 1) [40 CFR 180](#) for tolerance values set by EPA.
- 2) [21 CFR 556](#) for tolerance values set by FDA.
- 3) The [National Residue Program](#) sets the number of samples analyzed each year for veterinary drugs.

Contact Information and Inquiries

Inquiries about methods can be submitted through the USDA website via the “Ask USDA” portal at <https://ask.usda.gov> or please contact:

Chemistry Section
Laboratory Quality Assurance, Response, and
Coordination Staff
USDA/FSIS/OPHS
950 College Station Road
Athens, GA 30605
OPHS.LQAD@usda.gov

This method has been validated, reviewed, approved, and deemed suitable and fit for purpose for use in the USDA FSIS Field Service Laboratories.



William K. Shaw, Jr., PhD
Executive Associate for Laboratory Services