Method Needs and Fitness for Purpose Statement - Draft

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Project: A method for determining pesticides in animal feed, feed ingredients, and pet food.

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1. Needs:

Pesticides are applied ubiquitously around the world to prevent and/or eradicate pests that can damage or kill livestock and agricultural crops. The most commonly used are herbicides, insecticides, fungicides, and rodenticides. Approximately 37% of the world's grain and 66% of U.S. grain is used for livestock feed. These grains, and other ingredients used in feed, are grown on large farms that use pesticides to increase yields and remain competitive. Additionally, crops may become contaminated by pesticides that persist in the environment, even though they are no longer in use on farms. Some of these pesticide have the potential to accumulate in the animals' fatty tissue; when the meat and dairy products derived from these animals are consumed, we are exposed to the same pesticides.

Methodology is required to verify residue levels in animal feed, feed ingredients, and pet food. Methodology for the clean up of possible carry over of compounds should be included in the validation work. The most appropriate method of analysis would be HPLC MS/MS and/or GC MS/MS and must be able to determine all commonly used pesticides at trace levels.

The FDA, who is responsible for the enforcement of pesticide tolerances, has established action levels (ppm) for unavoidable pesticide residues in food and feed commodities. The pesticides covered are Aldrin and Dieldrin, Benzene Hexachloride (BHC), Chlordane, Chlordecone, DDT, DDE and TDE, Dicofol, Ethylene Dibromide (EDB), Heptachlor, Lindane and Mirex. Some of the commodities listed are found in animal feed, feed ingredients and pet food, each with a different action level for the pesticide. Below is a list of those commodities and their action levels:

	Pesticide and Action Level (ppm)									
Commodity	Aldirn & Dieldrin	BHC	Chlordane	Chlordecone	DDT, DDE, TDE	Dicofol	EDB	Heptachlor	Lindane	Mirex
Alfalfa	0.3									
Animal Fat, rendered			0.3							
Animal feed, processed	0.3	0.05	0.1		0.5	0.5		0.03	0.1	
Beans		0.05	0.1					0.05	0.5	
Cereal grains (except buckwheat, millet, teosinte, and wild rice)	0.02	0.05			0.5					
Corn			0.1						0.1	
Corn, fresh sweet					0.1				0.5	
Crabmeat				0.4						
Fats and oils (animal feed)	0.3									
Fish (edible portion)	0.3		0.3	0.3	5			0.3		0.1

Commodity	Aldirn & Dieldrin	BHC	Chlordane	Chlordecone	DDT, DDE, TDE	Dicofol	EDB	Heptachlor	Lindane	Mirex
Forage, fodder, and straw of cereal grains*	0.3									
Grain Products**							150			
Hay	0.3	0.05			0.5			0.03	0.01	
Milk (fat Basis)	0.3	0.3			1.25				0.3	
Oats									0.1	
Peanuts	0.05		0.1		0.2					
Potatoes	0.1		0.1		1					
Rice								0.03	0.1	
Rye									0.1	
Soybean hay	0.03									
Soybean oil (crude)					1					
Tomatoes	0.05	0.05	0.1		0.05					
Tomato pomace					0.5					
Wheat									0.1	

* (except those of buckwheat, millet, teosinte, and wild rice)

** Intermediate (milled) grains products (must be cooked prior to consumption)

2. Performance Characteristics: (Based on laboratory samples)

The method should be capable of detecting pesticides in complete feed, feed ingredients, and pet food.

Selectivity:

The method should be capable of detecting the more common pesticides, indicated in table 1, and be flexible enough to include more pesticides if desired. The method must be sensitive enough to resolve all compounds of interest and be free of interferences.

Limit of Quantitation (LOQ) Levels:

The method should aim to quantify as many pesticides in feeds, feed ingredients, and pet foods as possible at or below the LOQ levels. Our current LOQ levels for common pesticides are listed on table 1 (not matrix specific at this time).

Accuracy (Recovery):

The recommended recoveries will vary depending on the quantitation level. The AOAC's Single Lab Validation document recommends general recovery limits of 75 – 120% at levels below 1 mg/kg, 80 – 115% at levels of 10 mg/kg, 85 – 110% at 100 mg/kg, 90 – 108% at 1000 mg/kg, and 92% – 105% at 1% (see Figure 1). It notes, however, that "These limits may be modified as needed in view of the variability of individual results or which set of regulatory requirements are referenced. See table 1 for assumed recovery limits.

Precision Repeatability and Reproducibility:

The coefficient of variation will depend upon the target quantitation level. The repeatability and reproducibility coefficient of variation will vary depending on the concentration of pesticides in feed matrix.

Table 1.

Pesticide	LOD (ppm)	LOQ (ppm)	Accuracy, %
Chlorpyrifos	0.0012	0.00210	75-120
Malathion	0.003	0.01	75-120
Chlor-methal	0.0030		75-120
Permethrin	0.0024	0.008	75-120
Pirimphos-m	0.0024	0.008	75-120
Piperonyl butoxide	0.0048	0.016	75-120
Resmethrin	0.0024	0.008	75-120
Methoxychor p,p[1]	0.0019	0.0064	75-120
DDT	0.0019	0.0064	75-120
Atrazine	.0023	.0075	75-120
2,4-D	.0036	.012	75-120

Special Considerations:
The method is to be rugged/robust and critical parameters are to be identified and controlled. Method performance criteria are to be defined. A training plan should be developed which will demonstrate that the laboratory analyst can successfully perform the method prior to analyzing samples.
Spiking of a fortification compound into a clean matrix sample can be used for QA/QC, and results plotted on a control chart.
Matrix blanks and reagent blanks can also be utilized for QA/QC.

Traceability:
Reference standards and acceptable sources are to be identified. Standards are to be provided with assigned purity or potency and uncertainty value.