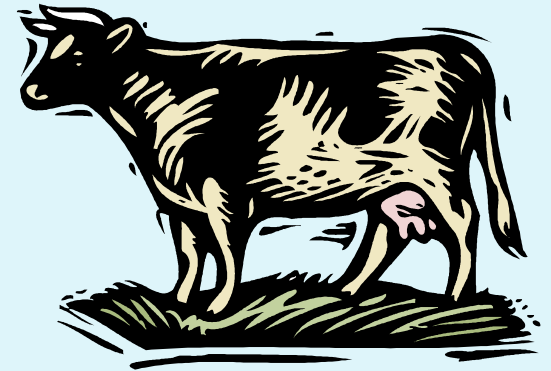


MDA SAMPLE PREP SPLIT STUDY

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AAFCO 2016

MT DEPARTMENT OF AGRICULTURE LAB

- Small Lab
- Feed, Fertilizer, Pesticide testing LAB
- Currently accredited in 5 methods
- Receive approximately 2,500 samples per year



2015-2016 FEED SEASON

- ~ 345 feed samples
 - 93 pet foods
 - 252 animal feeds
- Total deficient samples: 110/345 (31.9 %)
 - 14/93 (15.1%) deficient pet foods
 - 96/252 (38.1%) deficient animal feeds
- Some samples were deficient for more than one component

BREAKDOWN OF FEED TYPES

Feed Class

Beef

Poultry

Goat/Sheep

Swine

Dairy

Horse

Multiple Species

Exotic

Form of Feed

Pellet

Meal

Granular

Texture

Cube

Milk

Crumble

Liquid

Compressed

Poured

Premix

Mineral

Package Type

Packaged

Bagged

Bulk

Totes

BREAKDOWN OF PET FOOD TYPES

Form of Pet Food

Dry

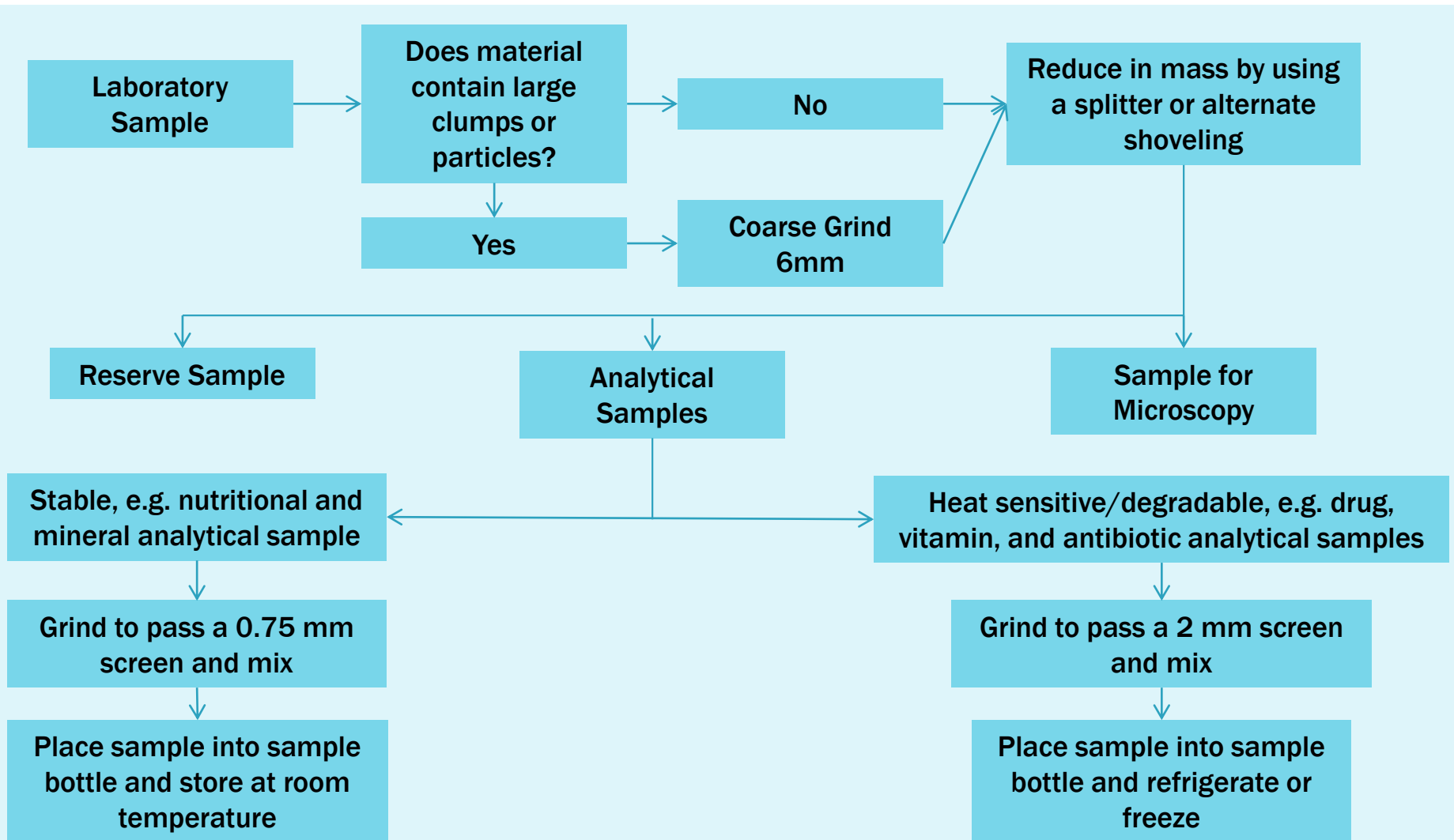
Wet

Package Type

Bagged

Other

AAFCO FLOW CHART FOR DRY COMPLETE FEEDS AND DRY PET FOODS





STANDARD APPROACH

- Mass reduction to a subsample/split with a mechanical splitter
 - 2015 - riffle
 - 2016 - rotary
- Split ground with Retsch ZM 200
 - Nutritional: 0.75 mm grind
- Split ground with Retsch SR 300
 - Vitamin A or drugs: 2 mm grind
- Split ground with Retsch GM300 with dry ice
 - High fat >15%
- Reserve sample unground



EXCEPTIONS

- Samples with large particle size such as large pellets and biscuits that do not fit into a splitter.
 - Grind in Wiley mill: 5.0mm
 - Split with mechanical splitter
 - 2015 - riffle
 - 2016 - rotary
 - Grind with Retsch ZM 200: 0.75mm
 - Grind with Retsch SR 300: 2.0mm
- Gummy/sticky samples such as molasses blocks or treats that stick inside the splitter.
 - Grind with dry ice in Retsch GM300 knife mill
 - Split by Alternate Shovel technique
- Reserve sample ground

DEFICIENT SAMPLES BASED ON APPROACH

95 - standard approach

15 - exceptions



SAMPLE PREP

- Samples typically range from 2-4 pounds
- **SPLIT SIZES**
 - Nutritional Split - $\sim 1/20$
 - EXTRA Split - $\sim 1/20$
 - Vitamin A Split - $\sim 1/10$
 - Drug Split - $\sim 1/15$
- Reserve sample- $\sim 2/3$ of original

DEFICIENT SAMPLE PROCESS

AB53338	1.20.16	beef medicated	texture	packaged	rifle	18%	→	Protein-1	1	15.23		
							→	Protein-1	2	15.36	15.295	
							→	Protein-2	2	15.83		
							→	DUP-2	2	15.93	15.88	
								reported		15.6	15.5875	3.75



1. The first split is tested during the initial run (run-1)
2. If deficient, a second split is obtained and the first and second split are tested together in run-2
3. Often a duplicate of split-2 is also analyzed during run-2
4. An average of each split is calculated
5. An average of the sample splits is calculated and reported.
6. The relative percent difference is calculated between the split values

DEFICIENT SAMPLE DATA

- Evaluate two approaches to splitting samples:
 - STANDARD: Sample is split and only the nutritional and/or vitamin A/drug splits are ground
 - EXCEPTIONS : Entire sample is ground then split

- Compare %RPD for each approach

- Compare the %RPD for rotary splitter and riffle splitter

	Standard		Exception	
Analyte	Average %RPD	Range	Average %RPD	Range
Ash			2.5 n=1	
Calcium	1.8 n=11	0.731 - 2.84	0.49 n=1	
Fat	2.5 n=10	0.219 - 5.67	0.79 n=3	0 - 1.64
Iron	0.78 n=3	0 - 1.28	4.9 n=1	
Magnesium	1.7 n=7	0.319 - 4.44		
Moisture	1.5 n=3	0.424 - 2.17		
NPN	0.94 n=1			
Phosphorous	1.4 n=14	0.115 - 5.28		
Potassium	0.91 n=1			
Protein	1.4 n=21	0.02 - 7.42	0.65 n=8	.0664 - 2.12
Zinc	4.4 n=5	0.836 - 10.7		
AVERAGE	1.79 n=76		1.10 n=14	

GOOD PERFORMERS

POOR PERFORMERS

Compound	Standard		Exception	
	Average %RPD	Range	Average %RPD	Range
Copper	12 n=8	0.294 - 45.4	2.3 n=2	2.32 - 2.35
Fiber	4.6 n=1		8.2 n=1	
Manganese	6.1 n=4	1.01 - 17.5		
Salt	2.0 n=14	0 - 6.03	5.7 n=2	1.44 - 9.89
Selenium	1.9 n=5	1.5 - 2.76	5.2 n=1	
Sodium	8.4 n=2	4.62 - 12.1	6.1 n=1	
Vitamin A	15 n=16	0 - 31.3	10 n=2	8.42 - 11.6
AVERAGE	8.39 n=50		6.17 n=9	

DRUGS & AMINO ACIDS

Compound	Standard		Exception	
	Average %RPD	Range	Average %RPD	Range
Amprolium	2.4 n=1			
Decoquinatate	12 n=1			
Lysine	1.3 n=6	0 - 3.06	3.4 n=2	2.9 - 3.97
Methionine			2.8 n=2	2.17 - 3.42
Monensin	0.91 n=1			
AVERAGE	2.57 n=9		3.1 n=4	

WHEN SPLITS DON'T MATCH

Lab Number	Date Printed	Species	Sample Type	Form	Split	Claim	A.V. Range	Analyte	Run	Result	Split Avg.
AB60117	2.18.16	goats/sheep	texture	packaged	rotary	17.00%	16.46-17.54	Protein-1	1	15.950	
								Protein-1	2	16.810	16.217
								Protein-2	2	14.946	
								DUP-2	2	15.140	15.043
								Protein-1	3	15.890	
								Protein-2	3	15.120	
								Protein-3 ALL	3	15.310	
								DUP-3 ALL	3	15.390	15.350
								reported		15.400	

- %RPD for Split 1 and Split 2 – 7.51%
- Rotary %RPD Average - 1.07% ± 0.939
- Visual Examination – very non homogenous
- Ground entire sample

MONENSIN

Lab Number	Date Printed	Species	Sample Type	Form	Split	Claim	A.V. Range	Analyte	Run	Result	Split Avg.	%RPD
AB60037	2.11.16	beef medicated	pellet	packaged	rotary	40 g/ton	28-52	Monensin-1	1	54.46		
								DUP-1	1	55.80		
								Monensin-1	2	54.30	54.85	
								Monensin-2	2	53.40		
								DUP-2	2	57.30	55.35	
								reported			55.10	0.907

COMPARING RESULTS OF SPLITTERS

- 2 different splitters used
 - Riffle – used for 2015 portion of feed season
 - Rotary – used for 2016 portion of feed season

Splitter	N	%RPD Average	%RPD STDEV
Riffle	56	4.13	7.65
Rotary	54	3.51	5.28

CONSIDERATIONS FOR SAMPLE PREPARATION

An excerpt from the “Overview of Laboratory Subsampling Error”

“Our committee has the following general recommendations for preparing samples: Whenever possible to minimize error, grind the entire laboratory sample before splitting out analytical samples. At a minimum, half of the laboratory sample should be ground before splitting out the analytical samples. The remaining half can then be saved as the reserve sample and for microscopy and other analyses requiring an unground test portion. Grinding less than $\frac{1}{4}$ of the laboratory sample prior to mass reduction / splitting procedures is unacceptable.”

CONCLUSIONS

■ Standard Approach

- ✓ %RPD higher
- ✓ Efficient process
- ✓ Not as true to TOS

■ Exceptions

- ✓ %RPD lower
- ✓ Not as efficient process
- ✓ More true to TOS

■ Rotary vs riffle

- ✓ %RPD lower
- ✓ Efficient process
- ✓ Permanent change

CONCLUSIONS

- Evaluate sample split/preparation
- Good and bad performers
- Lab continues this process
- Careful consideration to change
 - ✓ Efficiency
 - ✓ Theory of Sampling

CREDITS

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