

***Sample Preparation Variations – an  
Assessment of Different Practices based  
on the Chemical Analysis of Stable Analytes***

***Quick Review and Update***

**Objective was to ascertain the variation in results produced from sample preparation from different laboratory practices and procedures**

**A twenty pound sample of a pellet and a texture feed was spilt into 20- 1 pound subsamples and each of the 14 volunteer labs were sent a 1 pound portion.**

## **Volunteer Laboratory Processing Directions – Outline**

- 1. Prepare the Ground Portion under your SOP Protocols**
- 2. Weigh Out Three (3) Analytical Test Portions of each:  
Protein approximately 250 mg; minerals approximately 500 mg; Monensin approximately 20 grams**
- 3. Send All Test Portions and any remaining Ground and Un-Ground portions to Maryland Department of Agriculture for analysis.**

BAG #2

LAB ID 02  
Pelleted Ground Sample

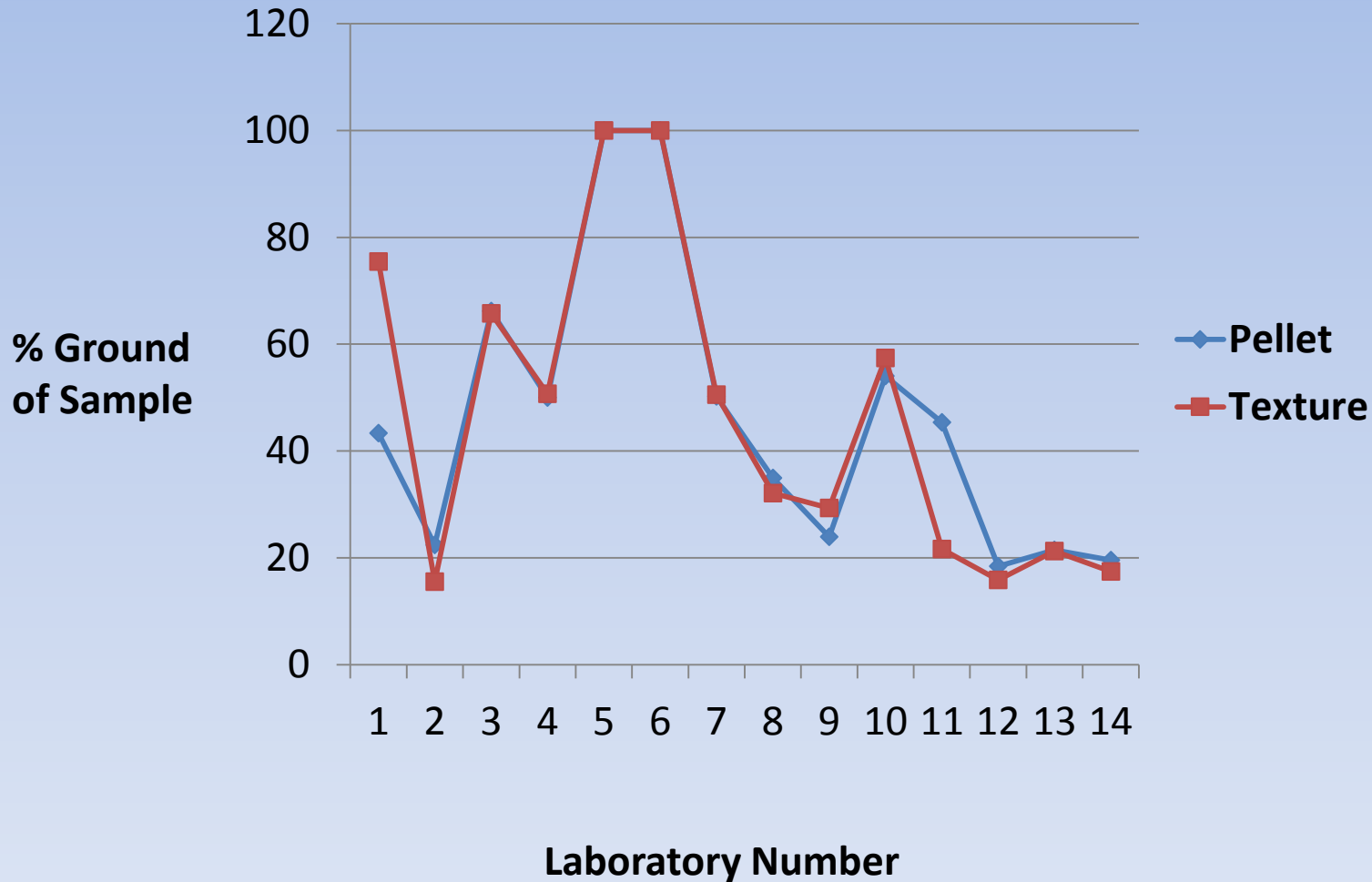
LAB ID 02 Pelleted  
MONENSIN SUB1 20.1713g

LAB ID 02 Pelleted  
MONENSIN SUB2 20.2474g

LAB ID 02 Pelleted  
MONENSIN SUB3 20.071g

Laboratory 02  
Pellet

# Sample Processing – Overlay Plot of Pellet and Texture Feeds , % Ground Feed Per Laboratory



## **Two Questions:**

**Is analyte integrity maintain from the original sample, especially in the 30% and less portions?**

**How would you know?**

**These are questions that can be asked throughout the sample prep process regardless of the % ground.**

## **Sieving Project**

- a ten pound sample of texture feed was spilt using a 10 place rotary splitter. Each pound was sieved through several soil sieves to separate the crush corn, pellets, oats and fines. Manual separation was also necessary to complete the separation.**





# Per Cent Ingredient

|         |                                       | Pellets           |  | Crushed<br>Corn   |  | Oats              |  | Fines |
|---------|---------------------------------------|-------------------|--|-------------------|--|-------------------|--|-------|
| 1       |                                       | 71.49             |  | 12.17             |  | 14.05             |  | 2.29  |
| 2       |                                       | 67.68             |  | 14.21             |  | 15.40             |  | 2.71  |
| 3       |                                       | 72.51             |  | 11.88             |  | 12.99             |  | 2.62  |
| 4       |                                       | 70.43             |  | 13.09             |  | 13.49             |  | 3.00  |
| 5       |                                       | 70.25             |  | 12.71             |  | 14.19             |  | 2.86  |
| 6       |                                       | 72.22             |  | 12.32             |  | 12.51             |  | 2.96  |
| 7       |                                       | 69.93             |  | 13.12             |  | 14.10             |  | 2.84  |
| 8       |                                       | 71.53             |  | 10.89             |  | 14.59             |  | 2.98  |
| 9       |                                       | 71.41             |  | 11.52             |  | 14.14             |  | 2.93  |
| 10      |                                       | 71.43             |  | 12.36             |  | 13.32             |  | 2.90  |
| Average |                                       | 70.89             |  | 12.43             |  | 13.88             |  | 2.81  |
| Min     |                                       | 69.93<br>"(67.68) |  | 10.89             |  | 12.51             |  | 2.29  |
| Max     |                                       | 72.22             |  | 13.12<br>"(14.21) |  | 14.59<br>"(15.40) |  | 3     |
|         | parentheses<br>denote row 2<br>values |                   |  |                   |  |                   |  |       |

**Each pound contains slightly different amounts of ingredients**

**Different analytical results due to sample preparation stems from the summation of differences. These differences are both additive and deductive. ( This statement also in the conclusions at the end of the presentation)**

| Sieve Sizes Used in Grinding |    |    |    |     |     |    |     |     |     |     |     |     |     |     |
|------------------------------|----|----|----|-----|-----|----|-----|-----|-----|-----|-----|-----|-----|-----|
| Lab Number                   | 01 | 02 | 03 | 04  | 05  | 06 | 07  | 08  | 09  | 10  | 11  | 12  | 13  | 14  |
| 2.0 mm                       | X  |    |    |     |     |    |     |     |     |     |     |     |     |     |
| 1.5 mm                       |    |    |    |     |     |    |     |     | XYZ |     |     |     |     |     |
| 1.0 mm                       |    |    | X  |     |     |    |     |     |     |     |     |     |     |     |
| 0.75 mm                      |    |    | Y  | XYZ | XYZ | X  | XYZ | XYZ |     | XYZ | XYZ | XYZ | XYZ |     |
| 0.50 mm                      | YZ |    | Z  |     |     | YZ |     |     |     |     |     |     |     |     |
| 0.71 mm                      |    |    |    |     |     |    |     |     |     |     |     |     |     | XYZ |

Monensin – X    Mineral – Y    Protein - Z

Textured feed ground through a 0.75 mm sieve



- slender long fragments about 3 to 7 mm long were found in 5 or 6 of the 14 textured feeds

- incomplete grind

- May have occurred in more of the samples.

- repeated test portions may be inconsistent in content and vary, especially on smaller test portions.



Oat Sample



- Sample was received in our Lab in August 2014**
- Sample prepared by rotary splitting followed by grinding about 225 Grams through a 0.75 mm sieve**
- Guarantee for protein on the label is 11.0%**
- Protein determined to be 8.3% and 8.7%**

**- For legal action our internal procedure is to visually compare the ground sample to the reserve sample for any obvious difference(s).**



Oat Sample Ground through a 0.75 mm sieve



- Substantial amount of fragments separated**
- Supervisor decided to re-grind the ground portion by**
  - a) rolling entire sample then quartering it**
  - b) a quarter section was removed then ground through a 0.25 mm sieve**
    - Protein determined to be 7.8%**
  - c) requested a repeat analysis; analyst repeated rolling, quartering, and grinding**
    - Protein determined to be 8.4%**

**-Protein was also determined also by taking 2-3 of the un-ground grains of oats and performing several analyses**

**- Protein determined was 7.0%, 7.5%, 7.2% and 6.5%**

**-Protein results - 0.75 mm      8.3% and 8.7%**  
**- 0.25 mm      7.8% (1st quartering, etc.)**  
**- 0.25 mm      8.4% (2<sup>nd</sup> quartering, etc.)**  
**- un-ground      7.0%, 7.5%, 7.2% and 6.5%**

## **Conclusions**

- Sample Preparation of both the pellet and texture feeds exhibited wider analytical variations than their comparative AAFCO matrices (Andy's conclusions)**
- Sample handling, mass reduction and grinding procedures are inconsistent and added to the variation. Different analytical results due to sample preparation stems from the summation of differences; these differences are both additive and deductive.**
- Still unsure as to why the pellet feed had wider variations as compared to the equivalent AAFCO check sample matrix.**