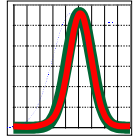


# Sample Homogeneity Testing

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Following the Protocol Outlined in:

***“The International Harmonized Protocol for the Proficiency Testing of Analytical Laboratories”, 2006 (IHP),*** MICHAEL THOMPSON, STEPHEN L. R. ELLISON AND ROGER WOOD



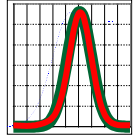
**Heterogeneity between the sample units ( $\sigma^2_{\text{Sampling}}$ ) can inflate the spread in sample results. This can mask the true Lab Bias and interferes with Z scores.**

Sources of Variance for Check Sample Results can be expressed as follows:

$$\sigma^2_{\text{SampleResults}} = \sigma^2_{\text{Analytical}} + \sigma^2_{\text{Labs}} + \sigma^2_{\text{Sampling}}$$

**Ideally, to test for sample homogeneity we would like to minimize the analytical variance ( $\sigma^2_{\text{Analytical}}$ ) and the Lab Bias ( $\sigma^2_{\text{Labs}}$ ) and isolate the variance due to the sample units ( $\sigma^2_{\text{Sampling}}$ ) .**

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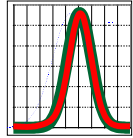
**So, how do we minimize the analytical variance ( $\sigma^2_{\text{Analytical}}$ ) and the Lab Bias ( $\sigma^2_{\text{Labs}}$ ) and isolate the variance due to the sample units ( $\sigma^2_{\text{Sampling}}$ ).**

- Randomly select 10 sample units from a batch for analysis.
  - Represents the sampling bias we wish to measure.
- Select a single expert lab (we used an ISO 17025 accredited lab using methods reported by AAFCO participant labs).
  - Should remove inter Lab bias between the samples.
- Choose a method with very low analytical variance.
  - Minimize analytical variance.

A Quote from the IHP:

*“Homogeneity tests should be regarded as essential, but not foolproof, safeguards.”*

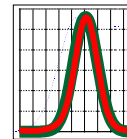
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Let's look at a Dataset for 10 sample units analyzed in duplicate at one lab.

- A Cochran test checks for outliers in duplicates.
- **Subtracting duplicates removes the sampling bias** in each sample unit.
- **Adding the duplicates generates twice this bias.**

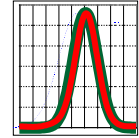
| Sample Code<br>(Sample Unit) | Moisture (%) |       |            |       |
|------------------------------|--------------|-------|------------|-------|
|                              | Dup 1        | Dup 2 | Difference | Sum   |
| 1                            | 5.69         | 5.60  | 0.09       | 11.29 |
| 2                            | 5.84         | 5.72  | 0.12       | 11.56 |
| 3                            | 5.59         | 5.54  | 0.05       | 11.13 |
| 4                            | 5.70         | 5.69  | 0.01       | 11.39 |
| 5                            | 5.69         | 5.56  | 0.13       | 11.25 |
| 6                            | 5.72         | 5.65  | 0.07       | 11.37 |
| 7                            | 5.54         | 5.53  | 0.01       | 11.07 |
| 8                            | 5.58         | 5.42  | 0.16       | 11.00 |
| 9                            | 5.52         | 5.65  | -0.13      | 11.17 |
| 10                           | 5.64         | 5.65  | -0.01      | 11.29 |



## Now we can Calculate the Sampling Variation

|  |               |
|--|---------------|
| Number of Pairs  | 10            |
| Grand Average  | 5.626         |
| <b>Sampling Variation Calculation</b>  |               |
| <b>A</b> - Variance of Differences (sampling error removed)                          | 0.00448       |
| <b>B</b> - Variance of Sums (includes A and 2 x sampling error)                      | 0.02766       |
| <b>Variance Attributed to Sampling (B/2-A)/2</b>                                     | <b>0.0047</b> |
| Repeatability %rsd   | 1.19%         |
| % RSD of Sample Means  | 1.48%         |
| $\sigma_{\text{analytical}} / \sigma_{\text{proficiency}}$ (Should be $\sim < 0.5$ ) | 0.3789        |

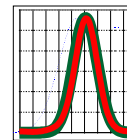
**Next we need to calculate a critical Variance  
which should not be exceeded**



## Calculating a one sided 95% CI for the Sampling Variation

|   |               |
|---|---------------|
| Number of Pairs   | 10            |
| Grand Average   | 5.626         |
| <b>Allowed Variation</b>  |               |
| <b>We must decide what dispersion is ffp!</b><br>Median Robust % RSD from last 19 CSP Samples<br>Chosen to represent the $\sigma_{ffp}$ for the % Moisture Method | 3.14%         |
| SD for Proficiency Testing ( $\sigma_{ffp}$ ) 5.626 * 3.14/100.   | 0.1767        |
| From the IHP: Allowed Variance (30% of target)  | 0.0028        |
| F1 constant (95% Confidence)  | 1.8799        |
| F2 constant (95% Confidence)  | 1.0102        |
| <b>Critical Allowed Variance (<math>\sigma^2_{Allowed}</math>)</b>  | <b>0.0098</b> |

Since **0.0047** is less than **0.0098** there is no evidence of sampling variance.



## Let's Talk About $\sigma_{ffp}$ ...

We need our best estimate of the usual and expected dispersion for the analysis with respect to our sample matrices and concentration types.

In the CSP we calculate  $\sigma_{ffp}$  for each sample from participants data.

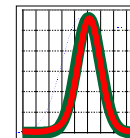
This is not practical for Homogeneity testing. We need to pick a  $\sigma_{ffp}$  appropriate to a particular analyte/method but across all our samples.

I looked back over the last 21 CSP samples and selected the Median Reproducibility %RSD for each Analyte used in the Homogeneity test.

This is a first approximation which we can refine as we move forward with more samples.

I favor using %RSD as it reflects variance dependent on concentration.

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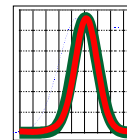
For example, we ran 2 Check Samples for Homogeneity Testing:

- Medicated Chicken Starter (201326)
- Soya Flour (201342)

And requested the following analyses:

| Analyte  | Method  | CSP Method #  |
|----------|---|---------------|
| Moisture | Moisture-Forced Draft (Food Products) - AOAC 930.15 | 011.01        |
| Protein  | Nitrogen by Combustion - AOAC 990.03                | 002.06        |
| Ca       | Calcium by ICP - AOAC 965.17 / 985.01 mod.          | 019.41/019.44 |
| Fe       | Iron by ICP - AOAC 965.17 / 985.01 mod.             | 025.41        |
| Mg       | Magnesium by ICP - AOAC 965.17 / 985.01 mod.        | 027.41/027.44 |
| Mn       | Manganese by ICP - AOAC 965.17 / 985.01 mod.        | 028.41/028.44 |
| P        | Phosphorus by ICP - AOAC 965.17 / 985.01 mod.       | 031.41/031.44 |
| K        | Potassium by ICP - AOAC 965.17 / 985.01 mod.        | 032.41        |
| Na       | Sodium by ICP - AOAC 965.17 / 985.01 mod.           | 035.41        |
| Zn       | Zinc by ICP - AOAC 965.17 / 985.01 mod.             | 037.41/037.44 |





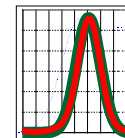
## Medicated Chicken Starter (201326)

| Expert Lab Sample Code          |            | Moisture (%) |       | Protein (%) |       | Ca (%)      |        | P (%)       |        |
|---------------------------------|------------|--------------|-------|-------------|-------|-------------|--------|-------------|--------|
| #                               | Ending in: | 1            | 2     | 1           | 2     | 1           | 2      | 1           | 2      |
| 1                               | 6040235    | 11.11        | 11.59 | 18.63       | 18.88 | 0.9068      | 0.9097 | 0.7447      | 0.7471 |
| 2                               | 6040236    | 11.13        | 11.52 | 18.56       | 18.63 | 0.9417      | 0.9101 | 0.7613      | 0.7667 |
| 3                               | 6040237    | 11.23        | 11.54 | 18.75       | 18.63 | 0.9009      | 0.9175 | 0.7698      | 0.7548 |
| 4                               | 6040238    | 11.13        | 11.44 | 18.63       | 18.63 | 0.9048      | 0.9238 | 0.7677      | 0.7675 |
| 5                               | 6040239    | 11.12        | 11.39 | 18.75       | 18.75 | 0.9088      | 0.9284 | 0.7487      | 0.7665 |
| 6                               | 6040240    | 11.08        | 11.40 | 18.56       | 18.69 | 0.9077      | 0.9340 | 0.7663      | 0.7655 |
| 7                               | 6040241    | 11.09        | 11.32 | 18.81       | 18.94 | 0.9093      | 0.9248 | 0.7576      | 0.7606 |
| 8                               | 6040242    | 11.02        | 11.25 | 18.56       | 18.94 | 0.9206      | 0.9195 | 0.7623      | 0.7668 |
| 9                               | 6040243    | 11.09        | 11.32 | 18.75       | 18.63 | 0.9143      | 0.9243 | 0.7589      | 0.7663 |
| 10                              | 6040244    | 11.27        | 11.28 | 18.81       | 18.50 | 0.9127      | 0.9122 | 0.7715      | 0.7650 |
| <b>Outlier Test</b>             |            | <b>PASS</b>  |       | <b>PASS</b> |       | <b>PASS</b> |        | <b>PASS</b> |        |
| <b>Allowed Variation</b>        |            |              |       |             |       |             |        |             |        |
| Median % RSD from CSP           |            | 3.14%        |       | 1.58%       |       | 4.20%       |        | 4.11%       |        |
| Critical variance               |            | 0.0671       |       | 0.0333      |       | 0.0004      |        | 0.0002      |        |
| <b>Actual Variation</b>         |            |              |       |             |       |             |        |             |        |
| Variance Attributed to Sampling |            | 0.0000       |       | 0.0000      |       | 0.0000      |        | 0.0000      |        |
| <b>Homogeneity Decision</b>     |            | <b>PASS</b>  |       | <b>PASS</b> |       | <b>PASS</b> |        | <b>PASS</b> |        |





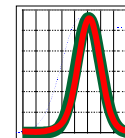
# Soy Flour (201326)



| Expert Lab Sample Code          |            | Moisture (%) |      | Protein (%) |       | Ca (%)      |        | P (%)       |        |
|---------------------------------|------------|--------------|------|-------------|-------|-------------|--------|-------------|--------|
| #                               | Ending in: | 1            | 2    | 1           | 2     | 1           | 2      | 1           | 2      |
| 1                               | 6040245    | 5.69         | 5.60 | 50.44       | 50.38 | 0.2705      | 0.2683 | 0.6982      | 0.7001 |
| 2                               | 6040246    | 5.84         | 5.72 | 50.00       | 50.00 | 0.2662      | 0.2724 | 0.7024      | 0.7019 |
| 3                               | 6040247    | 5.59         | 5.54 | 50.56       | 50.56 | 0.2659      | 0.2687 | 0.6996      | 0.6965 |
| 4                               | 6040248    | 5.70         | 5.69 | 50.06       | 50.44 | 0.2650      | 0.2686 | 0.7022      | 0.7039 |
| 5                               | 6040249    | 5.69         | 5.56 | 50.75       | 51.19 | 0.2669      | 0.2723 | 0.6976      | 0.7032 |
| 6                               | 6040250    | 5.72         | 5.65 | 50.38       | 50.56 | 0.2686      | 0.2706 | 0.7019      | 0.6992 |
| 7                               | 6040251    | 5.54         | 5.53 | 50.75       | 50.81 | 0.2604      | 0.2708 | 0.6934      | 0.6949 |
| 8                               | 6040252    | 5.58         | 5.42 | 50.06       | 50.31 | 0.2668      | 0.2686 | 0.6961      | 0.7014 |
| 9                               | 6040253    | 5.52         | 5.65 | 51.06       | 50.75 | 0.2620      | 0.2703 | 0.6978      | 0.7008 |
| 10                              | 6040254    | 5.64         | 5.65 | 50.81       | 50.75 | 0.2642      | 0.2701 | 0.6980      | 0.7081 |
| <b>Outlier Test</b>             |            | <b>PASS</b>  |      | <b>PASS</b> |       | <b>PASS</b> |        | <b>PASS</b> |        |
| <b>Allowed Variation</b>        |            |              |      |             |       |             |        |             |        |
| Median % RSD from CSP           |            | 3.14%        |      | 1.58%       |       | 4.20%       |        | 4.11%       |        |
| Critical variance               |            | 0.0098       |      | 0.1351      |       | 0.0000      |        | 0.0001      |        |
| <b>Actual Variation</b>         |            |              |      |             |       |             |        |             |        |
| Variance Attributed to Sampling |            | 0.0047       |      | 0.0929      |       | 0.0000      |        | 0.0000      |        |
| <b>Homogeneity Decision</b>     |            | <b>PASS</b>  |      | <b>PASS</b> |       | <b>PASS</b> |        | <b>PASS</b> |        |

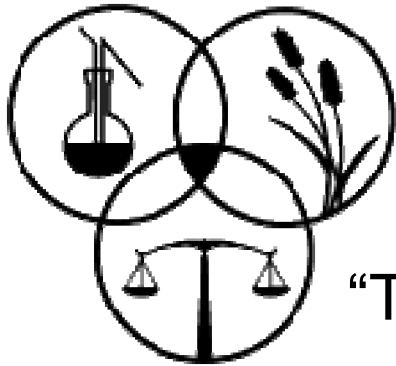
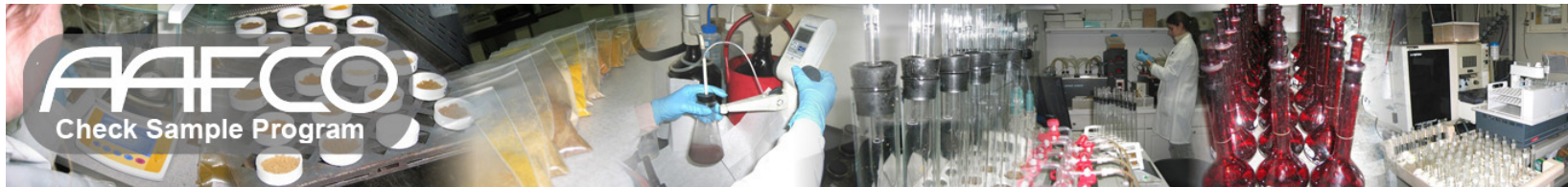


# Soy Flour (201326)



| Expert Lab Sample Code          |            | Fe (%)      |        | Mg (%)      |        | Mn (ppm)    |       | K (%)       |        | Na (%)*        |        | Zn (ppm)    |    |
|---------------------------------|------------|-------------|--------|-------------|--------|-------------|-------|-------------|--------|----------------|--------|-------------|----|
| #                               | Ending in: | 1           | 2      | 1           | 2      | 1           | 2     | 1           | 2      | 1              | 2      | 1           | 2  |
| 1                               | 6040245    | 0.0071      | 0.0072 | 0.2898      | 0.2935 | 33.28       | 32.84 | 2.3078      | 2.2967 | 0.0031         | 0.0032 | 46          | 46 |
| 2                               | 6040246    | 0.0071      | 0.0071 | 0.2870      | 0.2909 | 32.54       | 32.73 | 2.2889      | 2.2632 | 0.0026         | 0.0032 | 46          | 45 |
| 3                               | 6040247    | 0.0070      | 0.0071 | 0.2887      | 0.2910 | 32.97       | 32.47 | 2.3172      | 2.2846 | 0.0055         | <0.002 | 45          | 45 |
| 4                               | 6040248    | 0.0071      | 0.0071 | 0.2876      | 0.2906 | 32.58       | 32.51 | 2.3162      | 2.3006 | 0.0024         | 0.0044 | 45          | 45 |
| 5                               | 6040249    | 0.0071      | 0.0071 | 0.2889      | 0.2901 | 32.76       | 32.59 | 2.3029      | 2.3024 | 0.0033         | 0.0032 | 45          | 48 |
| 6                               | 6040250    | 0.0070      | 0.0072 | 0.2871      | 0.2902 | 32.72       | 32.64 | 2.2905      | 2.2968 | 0.0028         | 0.0035 | 45          | 48 |
| 7                               | 6040251    | 0.0070      | 0.0070 | 0.2851      | 0.2891 | 32.59       | 32.58 | 2.3039      | 2.2784 | 0.0022         | 0.0033 | 46          | 46 |
| 8                               | 6040252    | 0.0071      | 0.0071 | 0.2861      | 0.2917 | 32.36       | 32.58 | 2.2937      | 2.3073 | 0.0035         | <0.002 | 47          | 45 |
| 9                               | 6040253    | 0.0071      | 0.0072 | 0.2835      | 0.2872 | 32.58       | 32.53 | 2.2670      | 2.2966 | 0.0033         | <0.002 | 46          | 45 |
| 10                              | 6040254    | 0.0072      | 0.0073 | 0.2899      | 0.2882 | 32.45       | 32.74 | 2.2924      | 2.3365 | 0.0036         | <0.002 | 46          | 45 |
| <b>Outlier Test</b>             |            | <b>PASS</b> |        | <b>PASS</b> |        | <b>PASS</b> |       | <b>PASS</b> |        | <b>No Test</b> |        | <b>PASS</b> |    |
| <b>Allowed Variation</b>        |            |             |        |             |        |             |       |             |        |                |        |             |    |
| Median % RSD from CSP           |            | 5.73%       |        | 4.56%       |        | 4.52%       |       | 4.77%       |        | 5.06%          |        | 6.32%       |    |
| Critical variance               |            | 0.0000      |        | 0.0000      |        | 0.4016      |       | 0.0023      |        | 0.0000         |        | 2.6772      |    |
| <b>Actual Variation</b>         |            |             |        |             |        |             |       |             |        |                |        |             |    |
| Variance Attributed to Sampling |            | 0.0000      |        | 0.0000      |        | 0.0097      |       | 0.0000      |        | 0.0000         |        | 0.0000      |    |
| <b>Homogeneity Decision</b>     |            | <b>PASS</b> |        | <b>PASS</b> |        | <b>PASS</b> |       | <b>PASS</b> |        | <b>FAIL</b>    |        | <b>PASS</b> |    |

\*A review of the failure for Na reveals concentrations too close to the reporting limit,  $\sigma^2_{\text{Analytical}}$  not consistent.

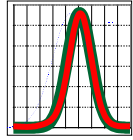


# Sample Homogeneity Testing

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“The Remarkable Case of Combustion Nitrogen”

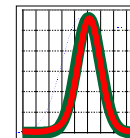
| <b>Protein by Combustion<br/>Nitrogen (# 002.06)<br/>Method Parameters</b> |  | <b>Assigned<br/>Value - Robust<br/>Mean</b> | <b># Labs<br/>Included in<br/>Calculations</b> | <b>Between<br/>Labs<br/>%RSD</b> | <b>Repeatability<br/>%rsd</b> | <b>Reproducibility<br/>%RSD</b> | <b>sR/sr</b> | <b>Modified<br/>Horwitz %RSD</b> |
|--|--|---|--|----------------------------------|-------------------------------|---------------------------------|--------------|----------------------------------|
| 201341   | Chicken Meal                                     | 64.93                                       | 48   | 1.02%                            | 0.75%                         | 1.27%                           | 1.69         | 1.24                             |
| 201342   | Soya Flour                                       | 50.61                                       | 39   | 0.97%                            | 0.41%                         | 1.05%                           | 2.59         | 1.41                             |
| 201230   | Beef Feedlot special, Medicated                  | 43.41                                       | 140  | 1.86%                            | 0.45%                         | 1.91%                           | 4.30         | 1.52                             |
| 201223   | Beef Feedlot special, Medicated                  | 41.18                                       | 141  | 0.82%                            | 0.72%                         | 1.10%                           | 1.51         | 1.56                             |
| 201225   | Dry Cat Food                                     | 32.47                                       | 137  | 1.08%                            | 0.58%                         | 1.22%                           | 2.12         | 1.75                             |
| 201222   | Senior Pig Starter, Medicated                    | 23.37                                       | 140  | 1.07%                            | 0.76%                         | 1.31%                           | 1.73         | 2.07                             |
| 201323   | Swine Pre-starter, Medicated                     | 22.94                                       | 133  | 1.51%                            | 0.66%                         | 1.65%                           | 2.50         | 2.09                             |
| 201229   | Swine Pre-starter, Medicated                     | 22.58                                       | 139  | 1.40%                            | 0.62%                         | 1.53%                           | 2.46         | 2.10                             |
| 201226   | Dairy Herd & Beef Calf Milk Replacer, Medicated  | 19.48                                       | 140  | 2.09%                            | 0.85%                         | 2.26%                           | 2.65         | 2.27                             |
| 201321   | Dry Dog Food                                     | 19.07                                       | 152  | 1.47%                            | 0.61%                         | 1.59%                           | 2.61         | 2.29                             |
| 201322   | Chicken Starter/Grower, Medicated                | 18.85                                       | 122  | 1.70%                            | 0.60%                         | 1.81%                           | 3.00         | 2.30                             |
| 201326   | Chicken Starter/Grower, Medicated                | 18.57                                       | 129  | 1.23%                            | 0.69%                         | 1.41%                           | 2.05         | 2.32                             |
| 201232   | Calf Sarter/Grower, Medicated                    | 18.10                                       | 145  | 1.46%                            | 0.71%                         | 1.63%                           | 2.29         | 2.35                             |
| 201221   | Chicken Sarter/Grower, Medicated                 | 17.81                                       | 142  | 1.59%                            | 0.84%                         | 1.80%                           | 2.14         | 2.37                             |
| 201325   | Pelleted Beef Special, Medicated                 | 17.14                                       | 128  | 1.75%                            | 0.72%                         | 1.89%                           | 2.63         | 2.42                             |
| 201224   | Chicken Starter/Grower, Medicated                | 17.12                                       | 143  | 1.63%                            | 0.80%                         | 1.81%                           | 2.26         | 2.42                             |
| 201228   | Beef Cattle Pellet, Medicated                    | 15.44                                       | 134  | 1.86%                            | 0.95%                         | 2.09%                           | 2.19         | 2.55                             |
| 201227   | Sheep and Goat Grower/Finisher, Medicated        | 15.29                                       | 141  | 2.10%                            | 0.94%                         | 2.30%                           | 2.44         | 2.56                             |
| 201324   | Sheep & Goat Grower/Finisher, Medicated          | 15.04                                       | 140  | 1.81%                            | 0.83%                         | 1.99%                           | 2.39         | 2.58                             |
| <b>201231</b>  | <b>Preconditioning cattle starter, Medicated</b> | <b>12.81</b>                                | <b>144</b>                                     | <b>3.27%</b>                     | <b>1.21%</b>                  | <b>3.49%</b>                    | <b>2.89</b>  | <b>2.72</b>                      |
| <b>201298</b>  | <b>Feed grade monocalcium phosphate</b>          | <b>0.83</b>                                 | <b>17</b>                                      | <b>47.15%</b>                    | <b>5.65%</b>                  | <b>47.48%</b>                   | <b>8.41</b>  | <b>4.12</b>                      |



## Can We Estimate Homogeneity From the CSP Data?

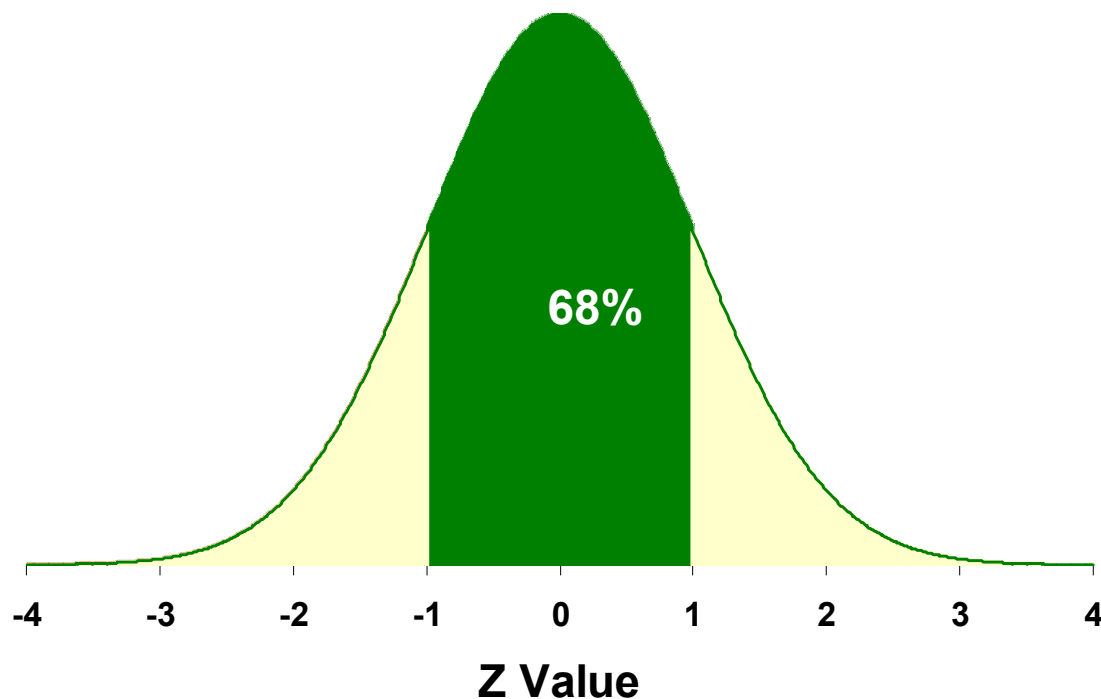
- In “Protein by Combustion N<sub>2</sub>” we have a very precise method with over 140 Analysts consistently reporting Reproducibility < 2% (%RSD) and Repeatability < 1% (%rsd) for each sample.
- This is an extremely narrow dispersion for so many Labs.
- So looking at our sources of variance again:
  - Reproducibility is heavily reflected in  $\sigma_{\text{Labs}}$  and is low!
  - Repeatability is essentially the  $\sigma_{\text{Analytical}}$  and is low!

$$\sigma_{\text{SampleResults}}^2 = \sigma_{\text{Analytical}}^2 + \sigma_{\text{Labs}}^2 + \sigma_{\text{Sampling}}^2$$



## Can We Estimate Homogeneity From Protein Data?

So if I take the center portion of the data ( $Z$  between  $\pm 1$ ,  $\sim 68\%$  representing typically 140 labs), these Labs should begin to approach the data from a Homogeneity study.



Let's call this the "Z" Cut – the "filet mignon" of the data, if you will.



Now let's compare the Soya Flour Homogeneity report with the "Z" Cut samples run by different labs/analysts from the CSP.

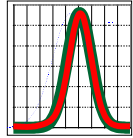
| Soya Flour (201342)                                 | Expert Lab    | CSP 002.06               |
|---|---------------|--------------------------|
| Homogeneity Decision                                | <b>PASS</b>   | <b>PASS</b>              |
| Number of Pairs (samples)                           | <b>10</b>     | <b>88 of 120 (Z Cut)</b> |
| Grand Average                                       | <b>50.53</b>  | <b>50.59</b>             |
| <b>Allowed Variation</b>                            |               |                          |
| Median % RSD from CSP                               | <b>1.58%</b>  | <b>1.58%</b>             |
| SD for Proficiency Testing                          | 0.7984        | 0.7993                   |
| Allowed Variance (30% of target)                    | 0.0574        | 0.0575                   |
| F1 constant   | 1.8799        | 1.2618                   |
| F2 constant   | 1.0102        | 0.2120                   |
| <b>Critical variance</b>                            | <b>0.1351</b> | <b>0.0809</b>            |
| <b>Actual Variation</b>                             |               |                          |
| Variance of Differences                             | 0.02695       | 0.03949                  |
| Variance of Sums                                    | 0.42535       | 0.19100                  |
| <b>Variance Attributed to Sampling</b>              | <b>0.0929</b> | <b>0.0280</b>            |
| Repeatability %rsd ( $\sigma_{\text{analytical}}$ ) | 0.32%         | 0.39%                    |
| % RSD of Sample Means                               | 0.65%         | 0.43%                    |
| Outlier Test  | <b>PASS</b>   | <b>PASS</b>              |

**Medicated Chicken Starter (201326) Homogeneity report with the “Z” Cut samples run by different labs/analysts from the CSP.**

|   |                   |                    |
|---|-------------------|--------------------|
| <b>201326</b>                                       | <b>Expert Lab</b> | <b>CSP 002.06</b>  |
| <b>Homogeneity Decision</b>                         | <b>PASS</b>       | <b>PASS</b>        |
| Number of Pairs                                     | <b>10</b>         | <b>133 (Z Cut)</b> |
| Grand Average                                       | <b>18.7</b>       | <b>18.55</b>       |
| <b>Allowed Variation</b>                            |                   |                    |
| <b>Median % RSD from CSP</b>                        | <b>1.58%</b>      | <b>1.58%</b>       |
| SD for Proficiency Testing                          | 0.2955            | 0.2930             |
| Allowed Variance (30% of target)                    | 0.0079            | 0.0077             |
| F1 constant   | 1.8799            | 1.2107             |
| F2 constant   | 1.0102            | 0.1660             |
| <b>Critical variance</b>                            | <b>0.0333</b>     | <b>0.0131</b>      |
| <b>Actual Variation</b>                             |                   |                    |
| Variance of Differences                             | 0.01836           | 0.0223             |
| Variance of Sums                                    | 0.02795           | 0.0576             |
| <b>Variance Attributed to Sampling</b>              | <b>0.0000</b>     | <b>0.0033</b>      |
| Repeatability %rsd ( $\sigma_{\text{analytical}}$ ) | 0.72%             | 0.80%              |
| % RSD of Sample Means                               | 0.45%             | 0.65%              |
| <b>Outlier Test</b>                                 | <b>PASS</b>       | <b>PASS</b>        |

## Z Cut Pseudo Homogeneity Testing at 3.00 %RSD to Calculate $\sigma_p$

| Sample # and Sample Name |   | Decision | $n_z$ | Mean  | Critical | Allowed |
|--------------------------|---|----------|-------|-------|----------|---------|
| 201321                   | Dry Dog Food                                    | PASS     | 172   | 19.10 | 0.0371   | 0.0155  |
| 201322                   | Chicken Starter/Grower, Medicated               | PASS     | 141   | 18.87 | 0.0381   | 0.0102  |
| 201323                   | Swine Pre-starter, Medicated                    | PASS     | 147   | 22.95 | 0.0549   | 0.0107  |
| 201324                   | Sheep & Goat Grower/Finisher, Medicated         | PASS     | 152   | 15.04 | 0.0238   | 0.0226  |
| 201325                   | Pelleted Beef Special, Medicated                | PASS     | 127   | 17.11 | 0.0324   | 0.0148  |
| 201326                   | Chicken Starter/Grower, Medicated               | PASS     | 133   | 18.55 | 0.0374   | 0.0033  |
| 201341                   | Chicken Meal                                    | PASS     | 100   | 64.93 | 0.4598   | 0.0582  |
| 201342                   | Soya Flour                                      | PASS     | 88    | 50.59 | 0.2699   | 0.0280  |
| 201221                   | Chicken Sarter/Grower, Medicated                | PASS     | 141   | 17.82 | 0.0344   | 0.0045  |
| 201222                   | Senior Pig Starter, Medicated                   | PASS     | 141   | 23.37 | 0.0564   | 0.0069  |
| 201223                   | Beef Feedlot special, Medicated                 | PASS     | 158   | 41.17 | 0.1690   | 0.0210  |
| 201224                   | Chicken Starter/Grower, Medicated               | PASS     | 158   | 17.14 | 0.0305   | 0.0196  |
| 201225                   | Dry Cat Food                                    | PASS     | 145   | 32.49 | 0.1077   | 0.0031  |
| 201226                   | Dairy Herd & Beef Calf Milk Replacer, Medicated | PASS     | 141   | 19.45 | 0.0392   | 0.0206  |
| 201227                   | Sheep and Goat Grower/Finisher, Medicated       | PASS     | 156   | 15.30 | 0.0251   | 0.0206  |
| 201228                   | Beef Cattle Pellet, Medicated                   | PASS     | 144   | 15.45 | 0.0255   | 0.0139  |
| 201229                   | Swine Pre-starter, Medicated                    | PASS     | 158   | 22.60 | 0.0520   | 0.0117  |
| 201230                   | Beef Feedlot special, Medicated                 | PASS     | 156   | 43.42 | 0.1900   | 0.0170  |
| 201231                   | Preconditioning cattle starter, Medicated       | FAIL     | 159   | 12.84 | 0.0225   | 0.0300  |
| 201232                   | Calf Sarter/Grower, Medicated                   | PASS     | 169   | 18.10 | 0.0344   | 0.0133  |
| 201298                   | Feed grade monocalcium phosphate                | FAIL     | 13    | 0.84  | 0.0011   | 0.0319  |



## Recommendation:

- I run a “Z” Cut pseudo Homogeneity test on “Protein by Combustion Nitrogen” data for each sample.
- If it passes the pseudo Homogeneity test at say 3.0 %RSD then we can assume acceptable homogeneity for our purposes.
- If it does not pass we will examine the sample data more closely before reporting a possible homogeneity issue.

This does not substitute for a legitimate Homogeneity study.

But, I think we can make a good case that it is a very reasonable (cost effective!) sample to sample homogeneity flag.

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