BUNGE

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Shea Laboratories Proficiency Test

Season 2023/2024

Contents

I. INTRODUCTION

This report presents the findings of the inter-laboratory analysis conducted on shea kernels and shea butter samples.

It is part of an external quality assurance program. Bunge Industries Ghana has organized this Inter-laboratory test for the Shea Supply Chain in cooperation with other tollers and suppliers. Bunge Limited (Ghana) prepared and sent identical sets of samples to different participating laboratories.

The key requirements of such comparisons are that the samples are homogenous and stable so results can be compared and relate to the performance of each lab in relation to the other participating laboratories. The participants carried out the requested quality analyses using their Standard Operating systems and submitted their results.

The results were statistically handled and reviewed to compare results as performance against the other participants. The results confidentially reviewed and described in a final report sent to each participant to allow them to compare their performance with other participants.

Objectives

It is important to acknowledge that no test procedure can ever yield identical results, even when performed on identical specimens under identical conditions. This is due to inherent errors in the testing process. To interpret test data accurately, these errors must be considered.

Several factors can contribute to the variability of test results, apart from the expected variations between identical samples. These factors include the competence of the operator conducting the test, the equipment used, the calibration of the equipment, the surrounding environment (temperature, humidity, etc.), and the time elapsed between measurements.

Precision, which refers to the variability between repeated tests, is a fundamental aspect that needs to be considered in test procedures.

Different test methods or test procedures may result in different variations and errors in test results. Understanding and managing these variations is essential for ensuring the reliability and consistency of testing and analysis outcomes. Considering all these factors informed the following objectives:

- To determine inter-laboratory reproducibility or accuracy of participating laboratories.
- To determine performance between participating laboratories.
- To determine the repeatability/precision within the existing laboratory.

II. TERMS AND DEFINITION

Grubb's outlier test

Grubb's test is a statistical method used to identify outliers in a dataset, primarily focusing on a single outlier. It calculates test statistics for each data point, comparing them to a critical value, and flags them as outliers.

Mandel's h and k statistics

Mandel's k and h statistics are data consistency metrics that are helpful in inter-laboratory research. Each laboratory is assigned an h and k value, evaluated against a critical value. Any value surpassing critical values are deemed significant. In this test, the 1% critical value serves as a threshold for significance, although the 5% critical value is also considered for appraisal purposes.

Mandel's k statistic compares the repeatability standard deviation of a laboratory data set with the average repeatability standard deviation of all other laboratories. This test statistic reflects a single lab's repeatability against the average repeatability of all participating laboratories.

Mandel's h statistic is used to examine the consistency of inter-laboratory data, confirming if any laboratory data is an outlier. The h test statistic reflects the deviation of a single laboratory's mean test results from the overall mean results obtained from the participating laboratories. By studying the collated data deviations and accuracy, the performance of a laboratory in terms of its accuracy and reproducibility can be established.

Youden plot

A Youden plot is a graphical representation used to assess performance of an analytical test method. It helps identify systematic errors or biases in a test process.
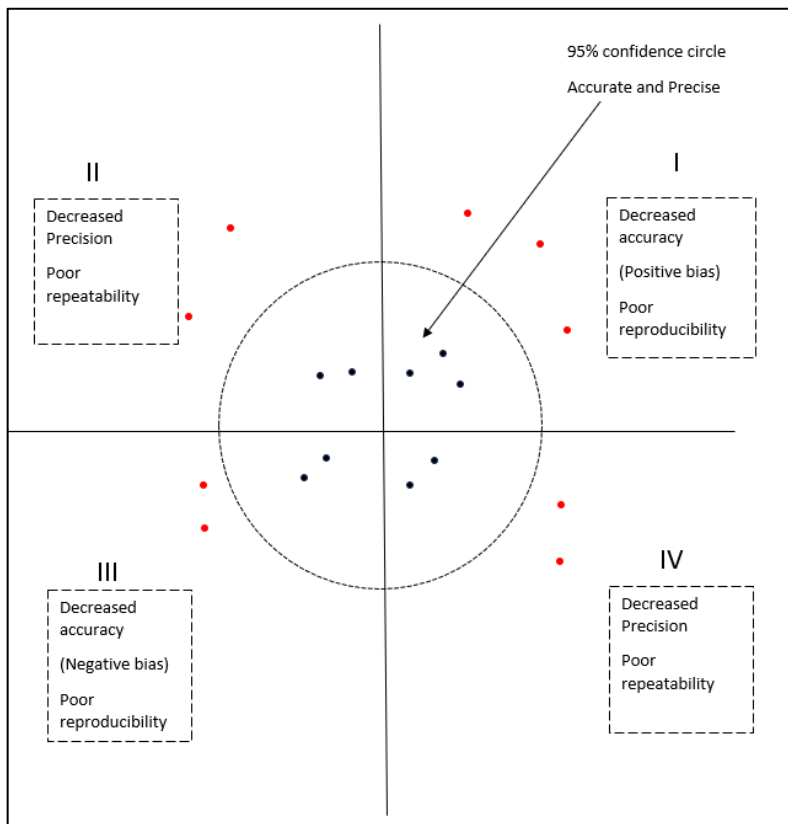
Z score

The z score, also known as standard score, is a statistical metric that expresses how a data point deviates from the data set mean in terms of standard deviations. It describes a value relationship to the mean.

Repeatability

Repeatability describes an instrument's or measurement procedure's capacity to yield consistent results when applied to the same sample or object under the same conditions.

Reproducibility

It is the consistency of a measurement technique or experiment results when carried out by several people, with various tools, or in various settings. It evaluates how reliable study results are when they are replicated under different conditions or by different operators in the lab.

Precision

Precision is the degree to which repeated measurements under the same conditions show the same results.

Accuracy

The closeness of a measured value to a standard or true value.
Statistical measurements of accuracy and precision reveal a test's basic reliability. These terms, which describe sources of variability, are not interchangeable. A test method can be precise (reliably reproducible in what it measures) without being accurate (measuring what it is supposed to measure), or vice versa.

III.   METHOD

Sample Collection

Shea kernels and shea butter samples were collected (representing samples from various sources) representing different geographical locations and mixed homogenously.

Sample Preparation

Samples were prepared according to standardized procedures to ensure uniformity and minimized variability. Six samples from Shea kernel and butter were prepared by Bunge Industries Ghana's quality control department and labelled as sample 'A' to 'F'. Within these samples, a "blind" duplicate was prepared for each category: Sample A and B for the kernels and sample E and F for butter. Adding blind duplicates is to obtain objective information without the knowledge of the participants in measuring a duplicate analysis as well as the precision of the laboratories to measure identical samples. The distribution of those six samples shown in table 1

Sample Label	Description of Sets
A	Shea kernels
B	Shea kernels identical to A
C	Shea kernels
D	Shea Butter
E	Shea Butter
F	Shea Butter identical to E

Testing Parameters

All samples were assigned for specific analyses such as FFA as oleic, moisture and volatile matter, impurities, oil content and color depending on the matrix or origin of the sample. Participating laboratories were requested to analyze the parameters using their internal daily operating procedures. Detailed information was provided by most of the participants (appendix). All analyses were requested to be measured in duplicate. The separate duplicate values were reported by the participants. The details on the sample test parameters are shown below:

For Shea butter (Sample D, E & F)
•   FFA as oleic (%)
•   Moisture and volatile matter (%)
•   Impurities (%)
For Shea nuts/kernels (Sample A, B & C)
•   FFA as oleic (%)
•   Moisture and volatile matter (%)
•   Oil content (%)

A total of 13 laboratories received the samples and participated in this ring test, all of whom submitted results. Laboratories included Bunge stakeholders from different countries. Laboratories were coded in performance evaluation to maintain confidentiality. Table below shows laboratories and corresponding samples to be analyzed.

Table 1. Laboratories and corresponding samples to be analyzed.

Laboratory	Product
K	Shea Kernels and butter
F	Shea Kernels and butter
D	Shea Kernels and butter
B	Shea Kernels and butter
E	Shea Kernels and butter
H	Shea Kernels and butter
A	Shea Kernels and butter
J	Shea Kernels and butter
M	Shea Kernels and butter
I	Shea Kernels and butter
G	butter
C	butter
L	butter

Table 2: Analysis performed by each participating Laboratory

	Shea Kernel				Shea Butter samples	
Laboratory	% FFA	% Moisture	% Oil Content	% FFA	% M & V	% Impurities
K	X	X	X	X	X	n/a
F	X	X	X	X	X	X
D	X	X	X	X	X	X
B	X	X	X	X	X	X
E	X	X	n/a	X	X	n/a
H	X	X	X	X	X	X
A	X	X	X	X	X	n/a

J	X	X	X	X	X	X
M	X	X	X	X	X	X
I	X	X	X	X	X	n/a
G	n/a	n/a	n/a	X	X	X
C	n/a	n/a	n/a	X	X	n/a
L	n/a	n/a	n/a	X	X	X

Statistical method

For the below flowchart, Shea Kernel is labelled (A, B, C with A=B, C) used as an example, same statistical method was applied to Shea Butter (D, E, F with E=F, D)

3 sets of test results
(A, B, C)

Set A
Identical to B

Set A
Identical to B

Set C
Non-Identical to A nor B

h and k validation
(*See APPENDIX II*)

A and B combined to quadruplet

Outlier detection
(Grubbs)

Statistical description

Youden plot

h and k statistics
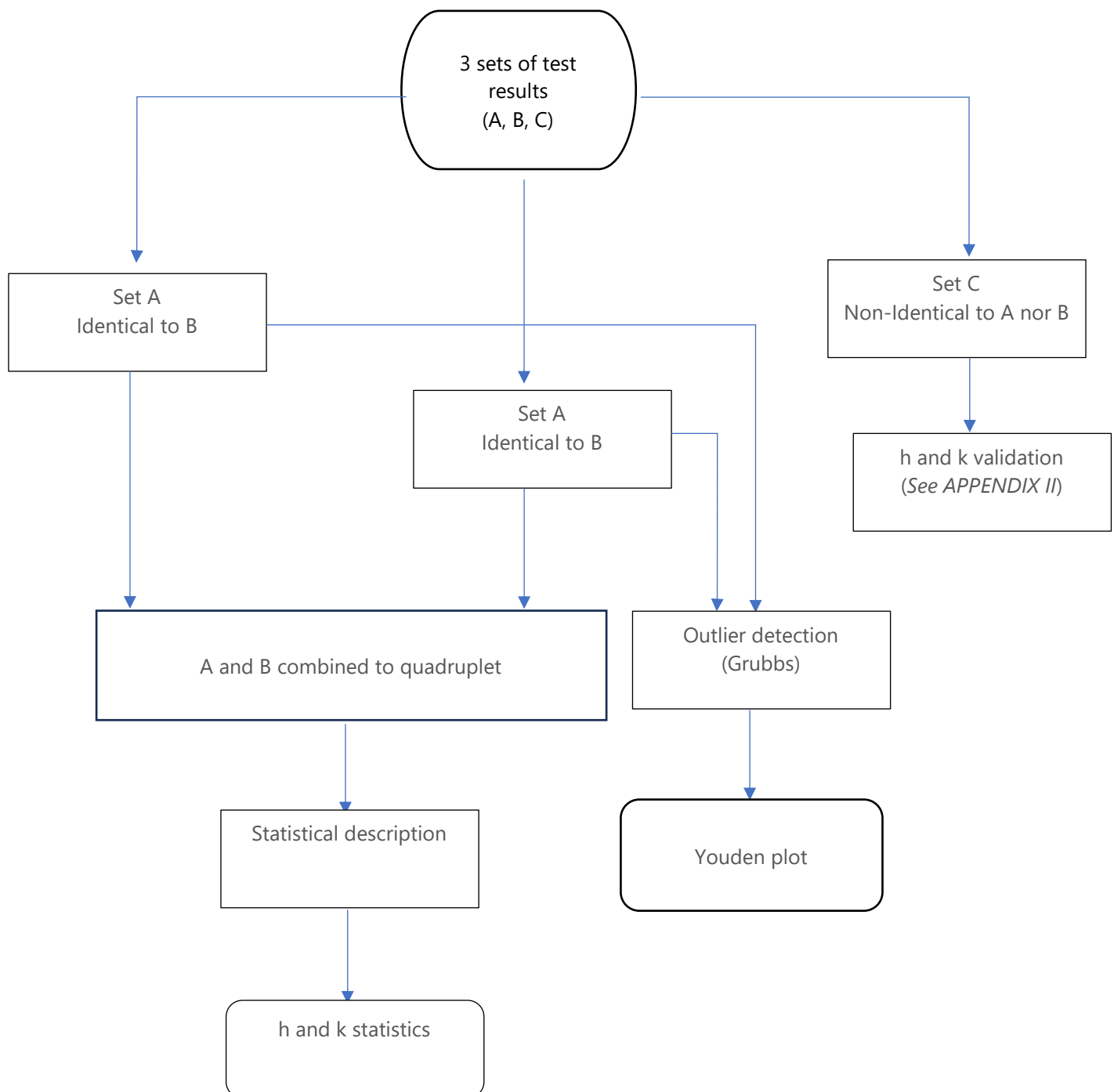
IV.   PERFORMANCE EVALUATION

1. Free Fatty Acids as Oleic (FFA) in Crude Shea Butter

Table 1: Overall mean. overall standard deviation, and number of outliers - FFA in crude shea butter

Mean	Standard deviation	Number of outliers
11.8	0.4	0

Test Results

Table 2: Test results and summary - FFA in crude shea butter

	TEST RESULTS				SUMMARY		
LAB	1	2	3	4	MEAN	Z-SCORE	OUTLIERS #
A	12.05	12.06	12.05	12.04	12.05	0.63	0
B	12.20	12.29	12.34	12.38	12.30	1.22	0
C	12.34	12.08	12.29	12.10	12.20	0.99	0
D	11.53	11.56	11.39	11.46	11.49	-0.70	0
E	11.69	11.60	11.04	11.31	11.41	-0.88	0
F	11.48	11.17	11.41	11.36	11.36	-1.01	0
G	11.82	11.11	11.50	11.59	11.51	-0.66	0
H	11.81	11.53	11.53	11.61	11.62	-0.38	0
I	12.26	12.20	12.43	12.39	12.32	1.27	0
J	11.10	11.41	11.11	11.28	11.23	-1.31	0
K	12.45	12.16	12.37	12.06	12.26	1.12	0
L	11.72	11.78	11.49	11.65	11.66	-0.29	0

Performance Evaluation 1: Consistency – k statistic and h statistic
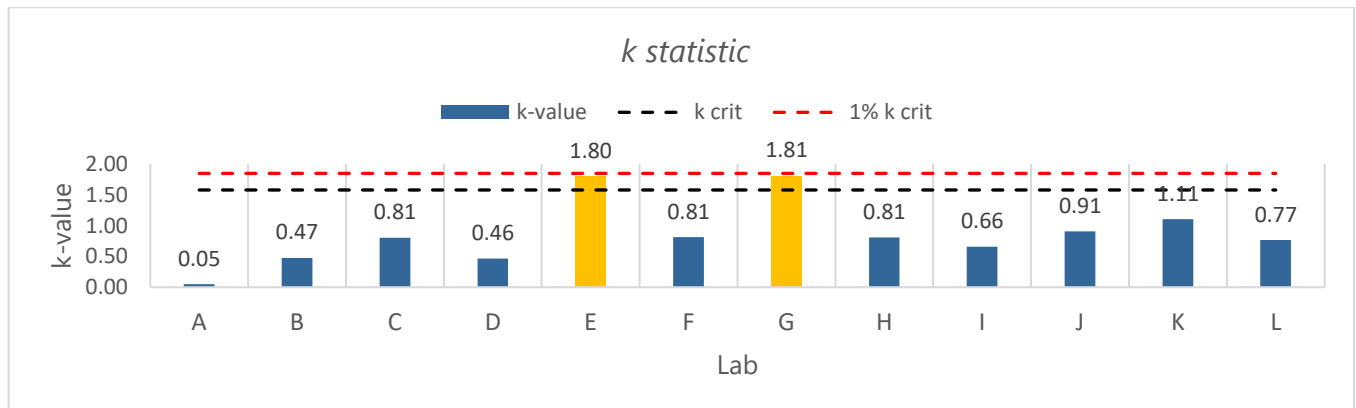

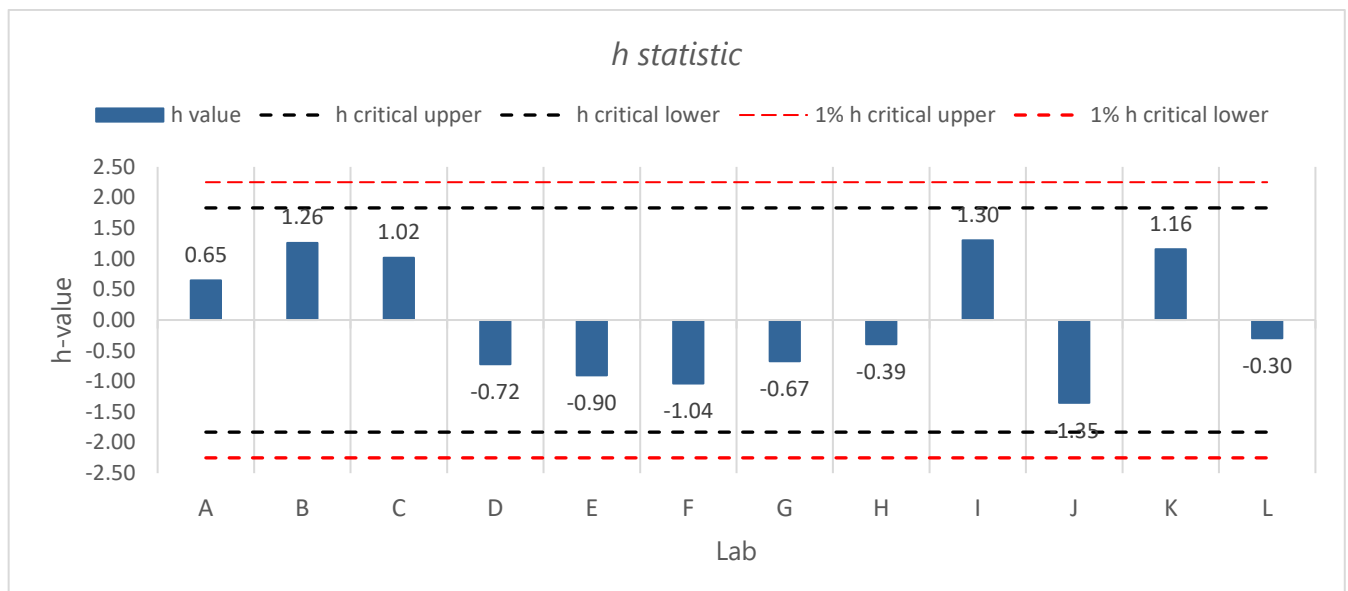*Figure 1: Mandel's K statistic – FFA Crude Shea Butter*

Figure 2: Mandel's H statistic – FFA Crude Shea Butter

Remarks

Among the participating labs, E and G recorded k values above the 1% critical value. This indicates a questionable laboratory repeatability compared to the other labs. Remaining labs showed good consistency within laboratory; lab A particularly shows excellent consistency (*see figure 1*).

From figure 2, all labs demonstrate excellent consistency between their testing methods, resulting in comparable results across different laboratory settings (*see figure 2*).
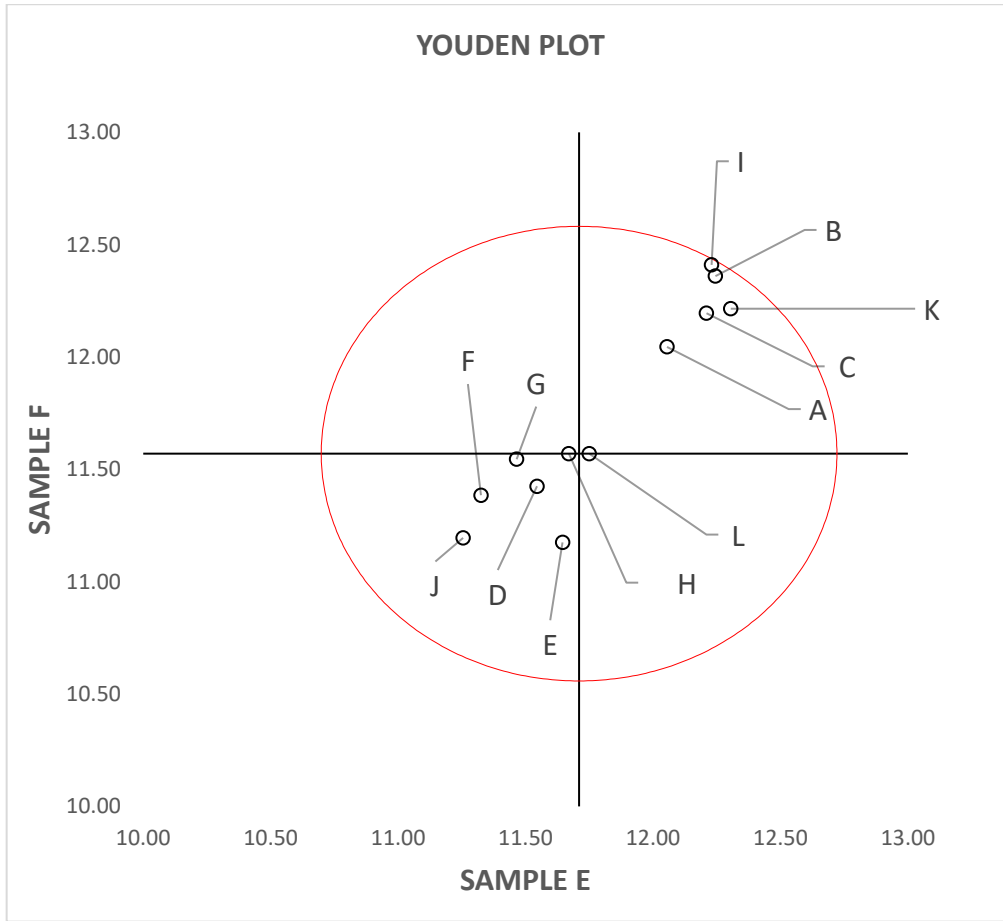
Performance Evaluation 2: Youden plot

Figure 3: Youden plot – FFA in Crude Shea Butter

<u>Remarks</u>
No outlying labs.
No laboratory significantly deviated.
There was neither a significant bias nor clustering.

Conclusion
All labs performed well in FFA determination in crude shea butter. Participating labs produce accurate and precise results. This shows in the overall standard deviation of 0.4.

2. Free fatty acids as oleic in Shea kernels

Test results

Table 3: Overall mean. overall standard deviation, and number of outliers - FFA in shea kernels

Mean	Standard deviation	Number of outliers
11.4	2.31	5

Table 4: Test results and summary - FFA in shea kernels

	TEST RESULT				SUMMARY		
LABS	1	2	3	4	MEAN	Z-SCORE	OUTLIERS #
A	13.22	12.62	12.16	12.18	12.55	0.50	0
B	12.19	12.16	12.44	12.38	12.29	0.40	0
D	14.66	14.9	8.99	9.06	11.90	0.23	1
E	10.46	11.09	10.51	11.21	10.82	-0.24	0
F	5.42	6.12	5.41	5.56	5.63	-2.48	4
H	11.25	11.36	11.7	11.89	11.55	0.07	0
I	11.86	11.55	12.49	12.46	12.09	0.31	0
J	12.82	12.58	11.92	11.43	12.19	0.35	0
K	12.35	12.15	13.02	13.16	12.67	0.56	0
M	13.99	13.99	10.2	10.2	12.10	0.31	0

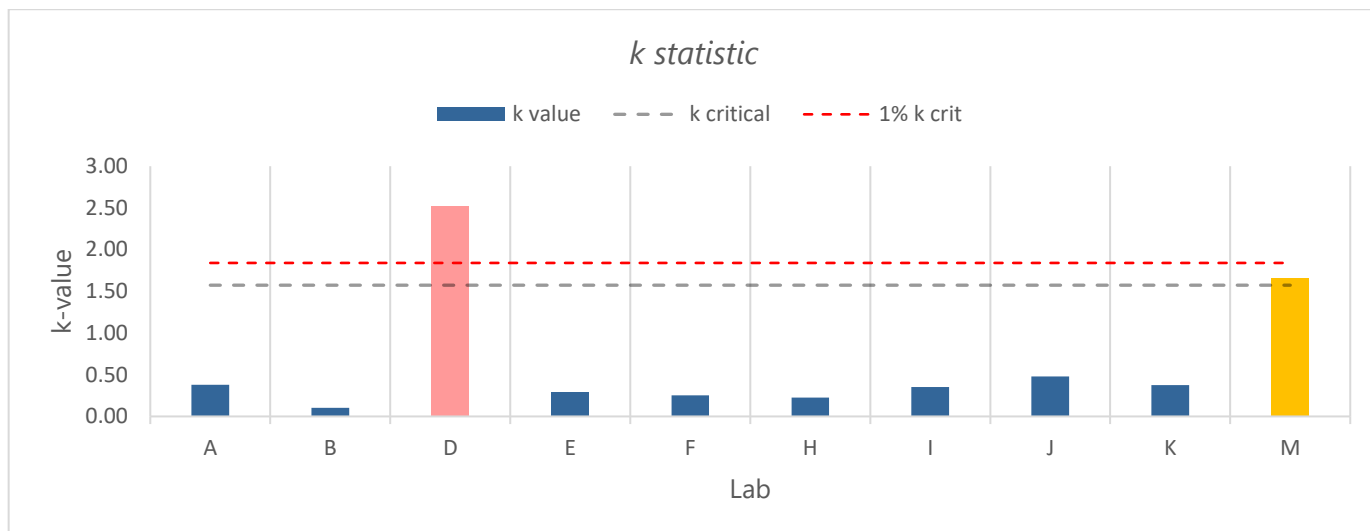**Performance Evaluation 1: Consistency – k statistic and h statistic**
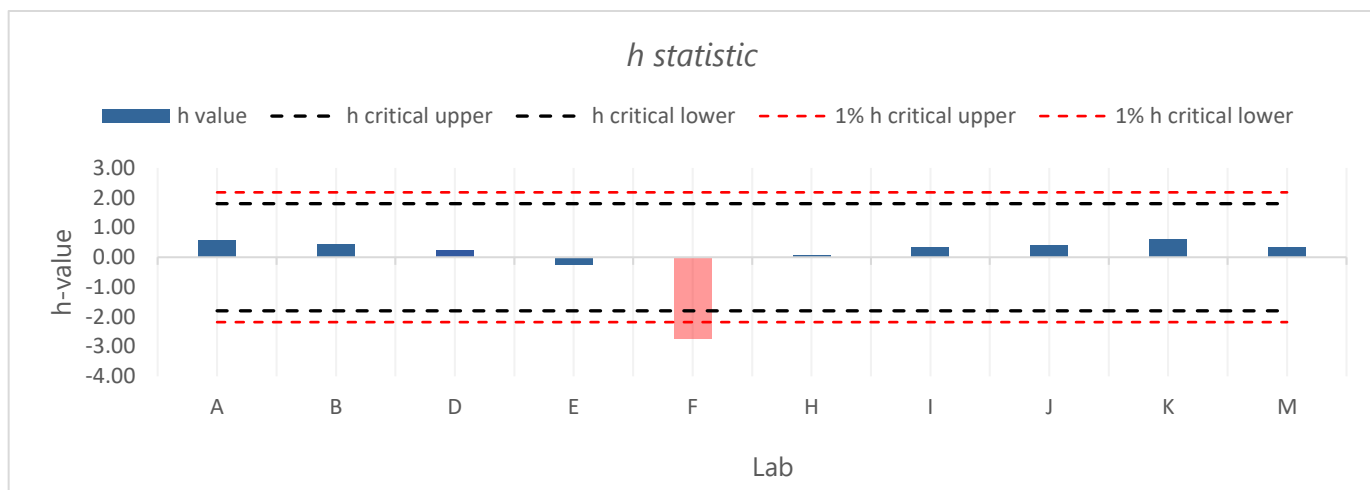

Figure 4: Mandel's k statistic – FFA in shea kernels

Figure 5: Mandel's h statistic – FFA in shea kernels

Remarks

All labs except lab D maintained k-values below the critical thresholds showing good within-laboratory consistency. Lab D exceeded the 1% critical value rendering its performance unsatisfactory. Also, lab M's results, while not as significant, are still below optimal standards. *see figure 4.*

Lab F shows poor between-laboratory consistency. However remaining labs indicate good consistency. *See figure 5.*
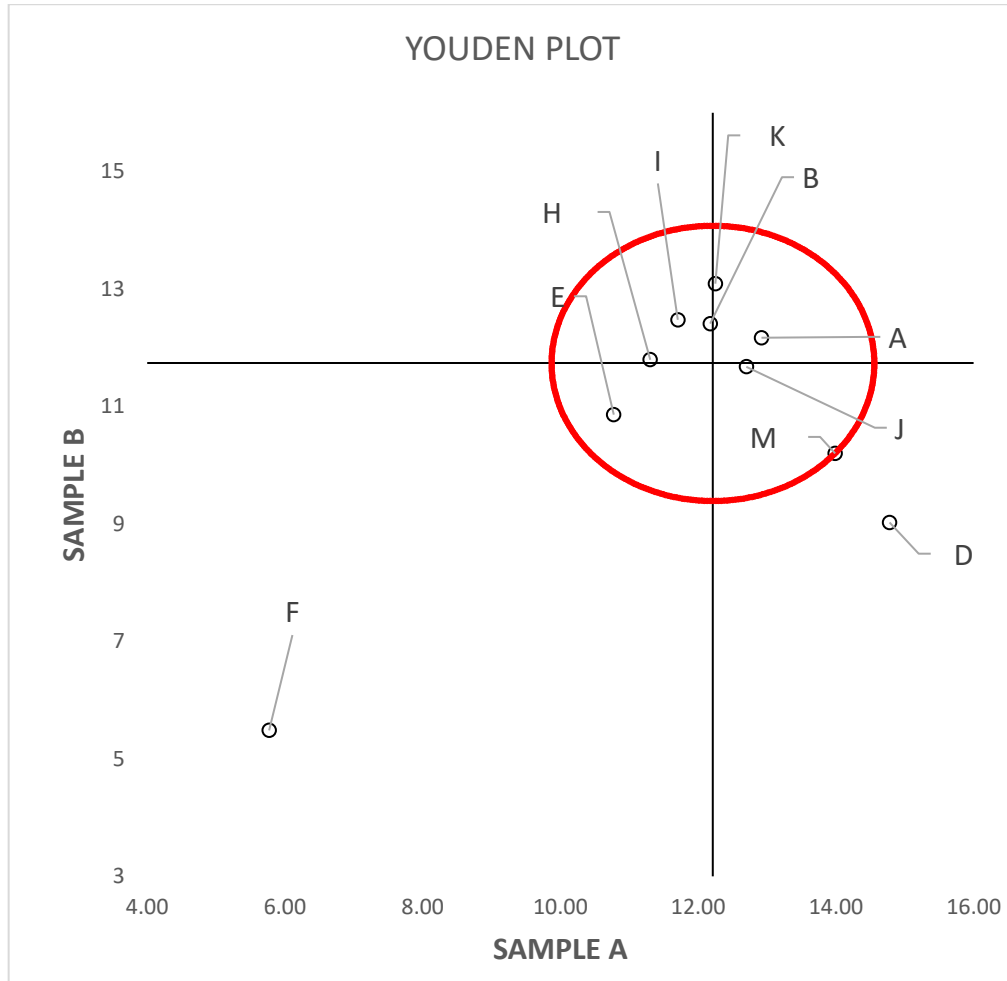
Performance Evaluation 2: Youden plot

Figure 6: Youden plot – FFA in shea kernels

Remarks

Inspection of the Youden plot (*figure 6*) reveals lab D and lab F outside the 95% circle. Lab D on the lower right quadrant with a high score from a sample and low score from an identical sample implies poor repeatability in lab D. Lab M shows a less severe repeatability challenge.
Lab F, on the other hand, shows a consistent negative bias in the lower left quadrant. This implies poor reproducibility.

Conclusion

Overall performance of FFA in shea kernels is skewed by poor performance of lab D and lab F which have glaring repeatability and reproducibility challenges, respectively.

3. Oil content in Shea kernels

Table 5: Overall mean, overall standard deviation, and number of outliers – Oil content in shea kernel

Mean	Standard deviation	Number of outliers
50.1	2.8	3

Test results

Table 6: Test results and summary- Oil content in Shea kernels

	TEST RESULTS				SUMMARY		
LABS	1	2	3	4	MEAN	Z-SCORE	OUTLIERS #
A	51.01	51.91	49.77	49.12	50.45	0.12	0
B	51.58	51.30	51.20	51.03	51.28	0.42	0
D	54.47	54.28	50.07	49.90	52.18	0.74	1
F	51.74	49.54	51.15	52.14	51.14	0.37	0
H	51.35	51.64	49.40	49.05	50.36	0.09	0
I	49.66	48.58	50.81	49.85	49.73	-0.14	0
J	50.96	50.44	48.79	48.66	49.71	-0.14	0
K	48.77	49.41	50.20	50.64	49.76	-0.13	0
M	41.00	39.80	53.40	51.60	46.45	-1.31	2

Performance Evaluation 1: Consistency – k statistic and h statistic

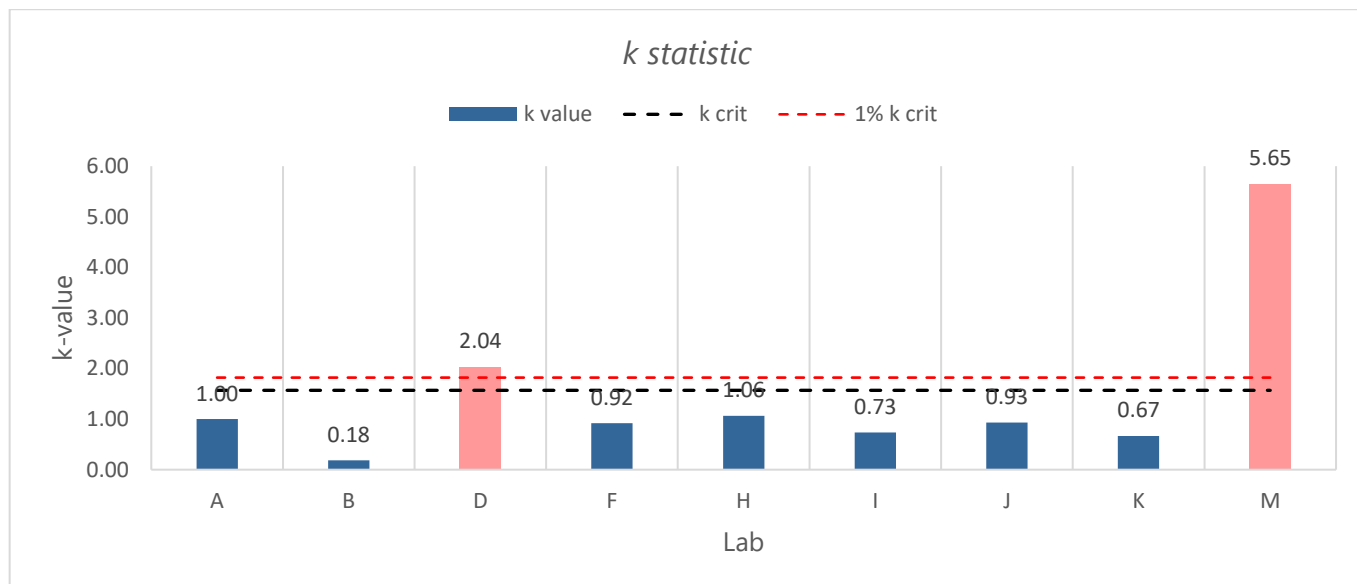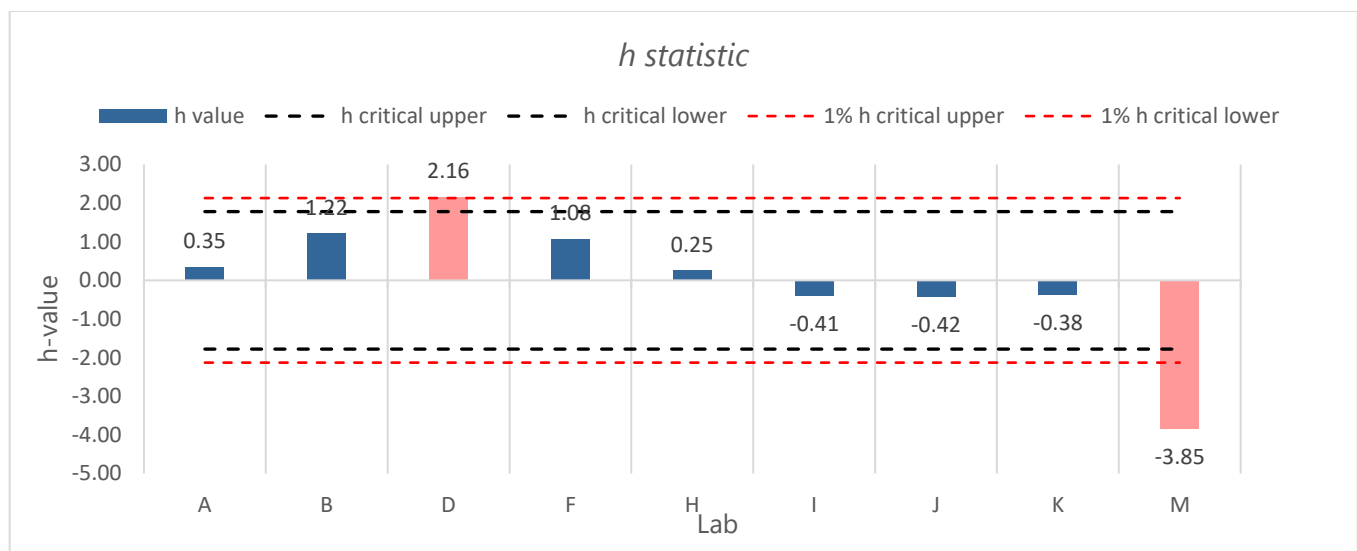Figure 7: Mandel's k statistic – Oil Content in Shea kernels

Figure 8: Mandel's h statistic – Oil Content in Shea kernels

Remarks

Lab D and Lab M show poor within-laboratory and poor between-laboratory consistencies as shown in figure 7 and figure 8 respectively.
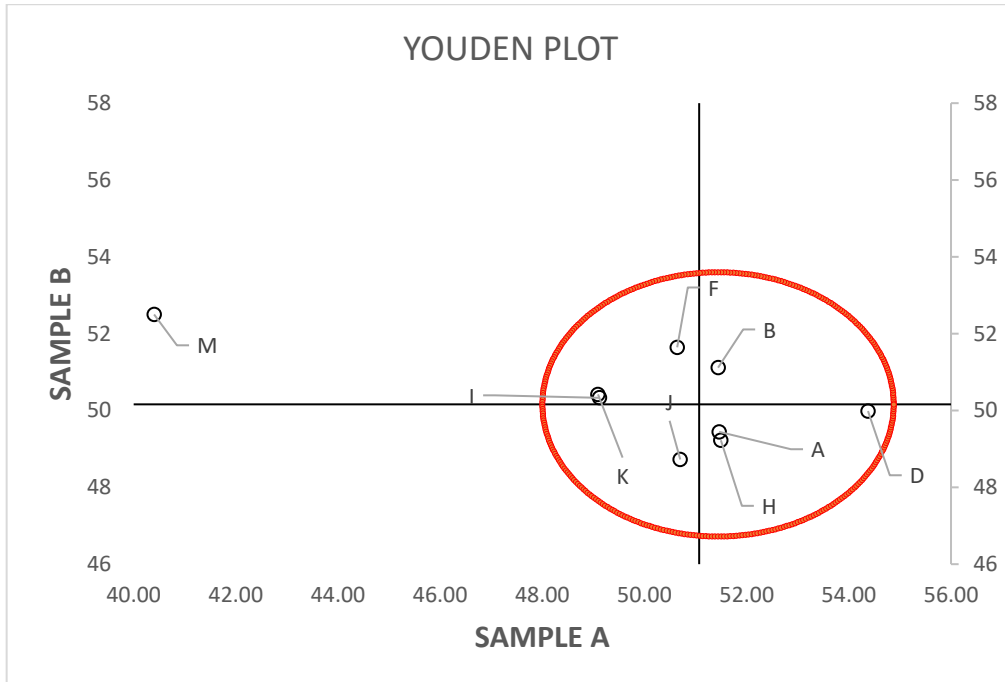
Performance Evaluation 2: Youden plot

Figure 9: Youden plot – Oil Content in Shea kernels

Remarks

Lab M sitting at the lower part of the upper left quadrant suggests poor accuracy and poor precision albeit the inaccuracy is more severe as can be seen in figure 7 and figure 8 above.

Conclusion

Both Lab D and Lab M have significant repeatability and reproducibility challenges. Lab M particularly significantly deviates from the other participating labs.

4. Moisture and Volatile in Crude Shea Butter

Test results

Table 7: Overall mean, overall standard deviation, and number of outliers- Moisture and Volatile in Crude Shea Butter

Mean	Standard deviation	Number of outliers
0.08	0.03	2

Table 8 8: Test result and summary- Moisture and Volatile in Crude Shea Butter

	TEST RESULTS				SUMMARY		
LABS	1	2	3	4	MEAN	Z-SCORE	OUTLIER #
A	0.10	0.11	0.09	0.09	0.09	0.38	0
B	0.07	0.07	0.09	0.09	0.12	1.48	2
C	0.08	0.09	0.06	0.06	0.05	-0.94	0
D	0.09	0.08	0.10	0.10	0.09	0.46	0
E	0.10	0.09	0.08	0.07	0.09	0.30	0
F	0.14	0.08	0.08	0.08	0.09	0.22	0
G	0.05	0.05	0.04	0.04	0.05	-1.11	0
H	0.08	0.06	0.05	0.05	0.06	-0.69	0
I	0.05	0.05	0.04	0.03	0.04	-1.19	0
J	0.13	0.14	0.13	0.12	0.12	1.47	0
K	0.13	0.11	0.09	0.07	0.08	-0.11	0
L	0.07	0.07	0.07	0.08	0.07	-0.28	0

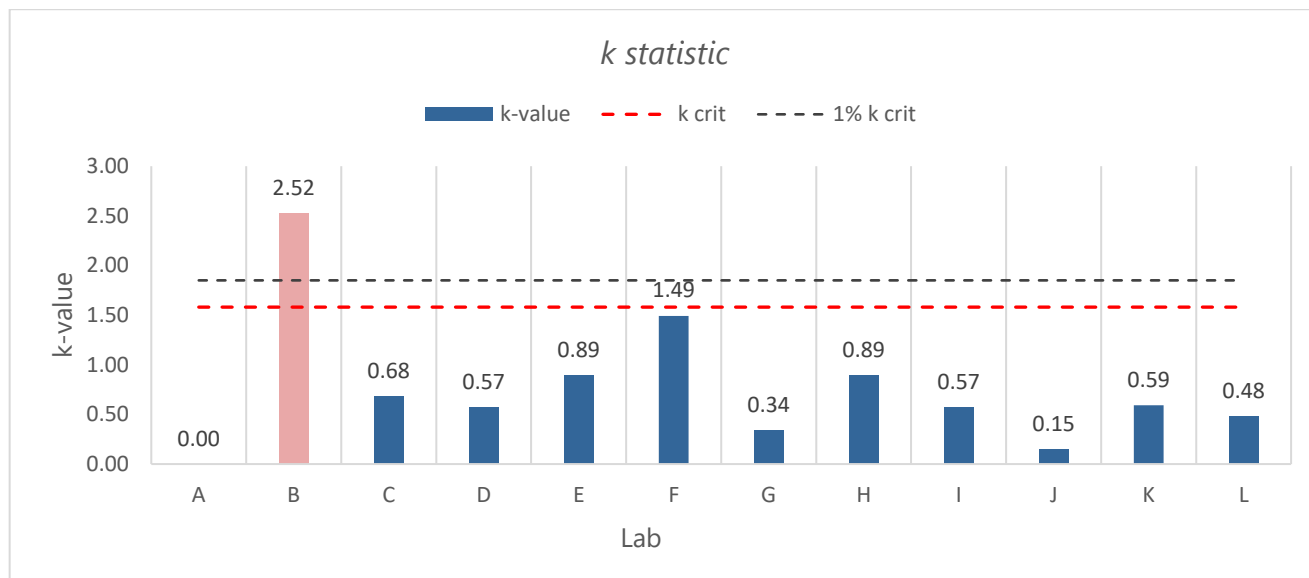**Performance Evaluation 1: Consistency – k statistic and h statistic**

Figure 10: Mandel's k statistic –Moisture and Volatile in Crude Shea Butter
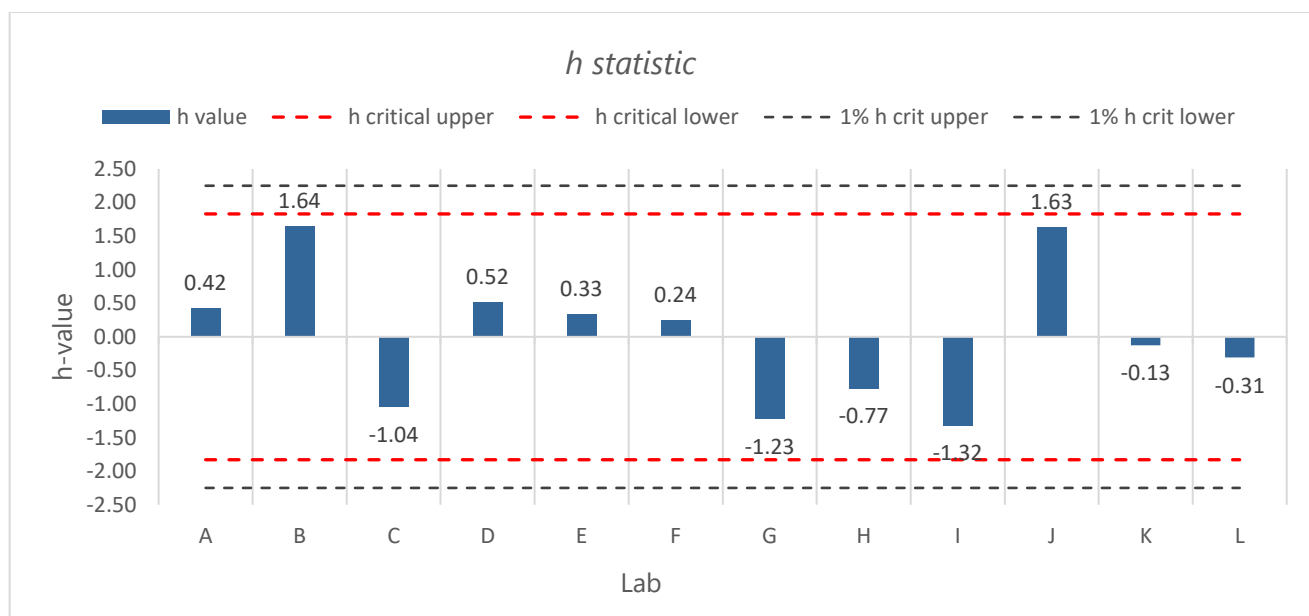

Figure 11: Mandel's h statistic –Moisture and Volatile in Crude Shea Butter

Remarks

All labs show good within-laboratory and between-laboratory consistencies with the exemption of lab B revealing unsatisfactory within-laboratory consistency (*see figure 10*).
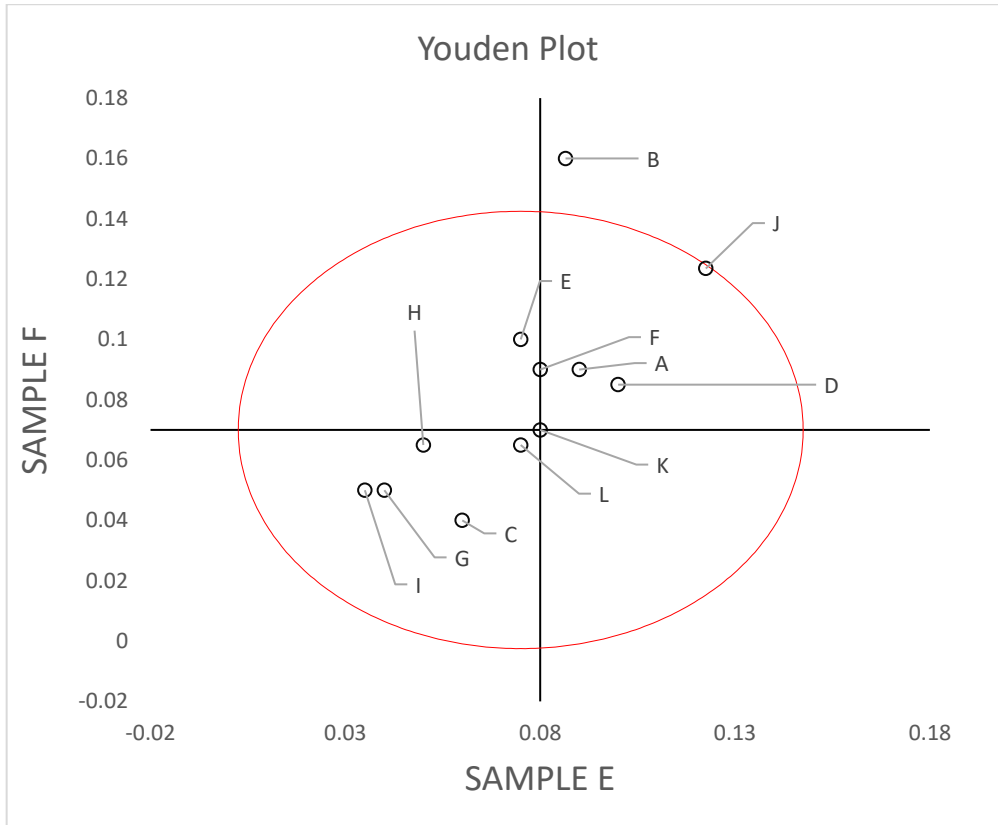
Performance Evaluation 2: Youden plot

Figure 12: Youden plot - Moisture and Volatile in Crude Shea Butter

Remarks

Inspection of figure 12 tells that lab B on the upper right quadrant has a reproducibility challenge. Lab J also has a similar yet less severe challenge.
Lab B's reproducibility challenge is exacerbated by its repeatability issue.

Conclusion

Lab B has a repeatability challenge. Overall, the performance was good.

5. Moisture and Volatiles in Shea kernels

Test results

Table 9: Overall mean, overall standard deviation, and number of outliers- Moisture and volatiles in Shea kernels

Mean	Standard deviation	Number of outliers
6.0	2.0	4

Table 10: Test results and summary- Moisture and Volatiles in Shea kernels

	TEST RESULTS				SUMMARY		
LABS	1	2	3	4	MEAN	Z-SCORE	OUTLIERS #
A	5.22	5.2	5.26	5.22	5.23	-0.37	0
B	5.13	5.18	4.78	4.81	4.98	-0.49	0
D	5.04	4.96	4.46	4.38	4.71	-0.62	0
E	4.95	4.96	5.08	4.68	4.92	-0.52	0
F	12.24	11.48	12.36	11.42	11.88	2.89	4
H	5.42	5.37	5.62	5.55	5.49	-0.24	0
I	5.65	5.95	5.66	5.68	5.74	-0.12	0
J	5.62	5.53	5.6	5.77	5.63	-0.17	0
K	6.27	6.48	5.52	5.47	5.94	-0.02	0
M	5.5	5.43	5.35	5.03	5.33	-0.32	0

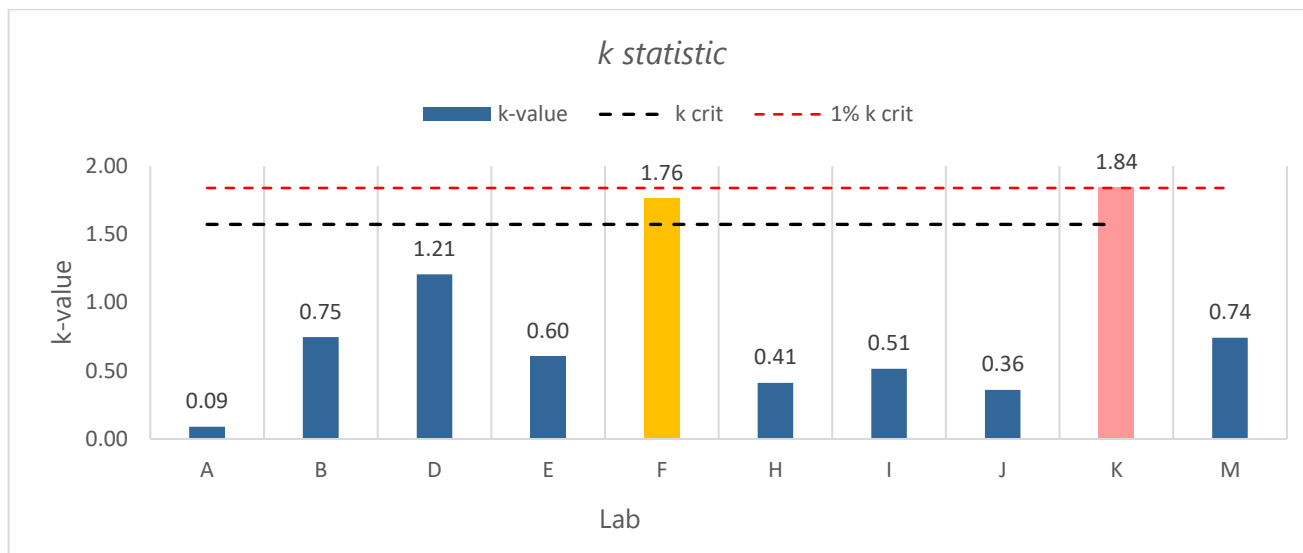**Performance Evaluation 1: Consistency – k statistic and h statistic**

Figure 13: Mandel's k statistic – Moisture and Volatile in Shea kernels

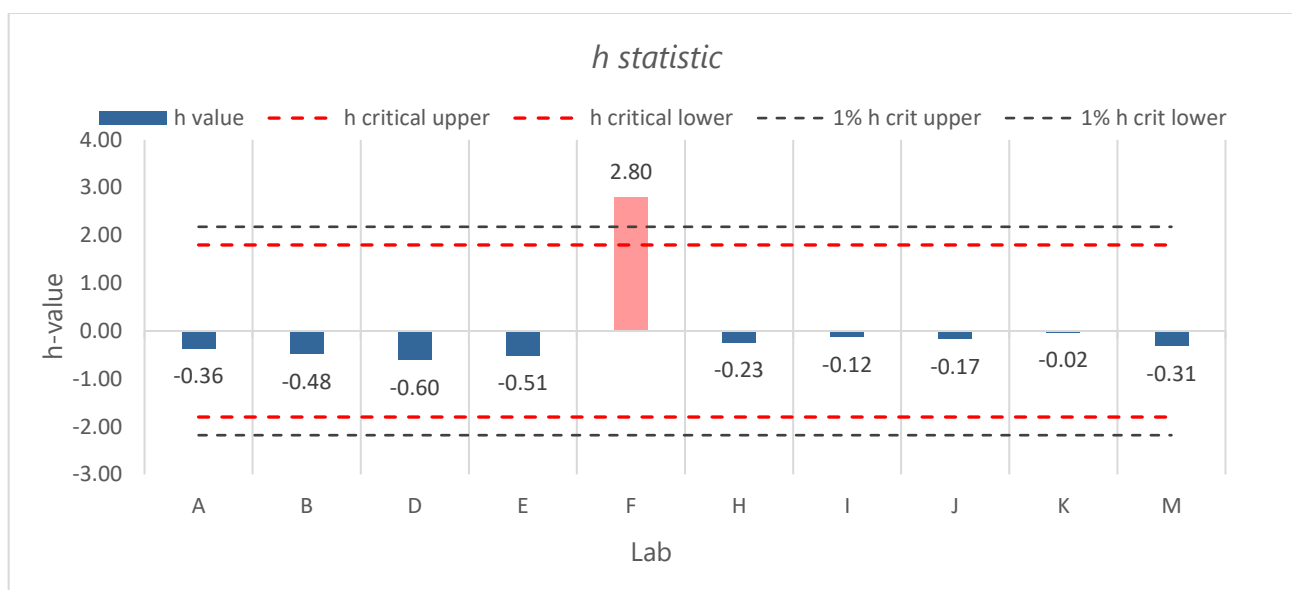*Figure 14: Mandel's h statistic – Moisture and Volatile in Shea kernels*

<u>Remarks</u>

Lab K exceeded the 1% critical value for the k-statistic rendering its within-laboratory performance unsatisfactory. Also, lab F's results, while below the 5% critical threshold, are still below optimal standards. *see figure 13.*

Lab F also showed poor between-laboratory consistency while all labs indicated good consistency. *See figure 14.*
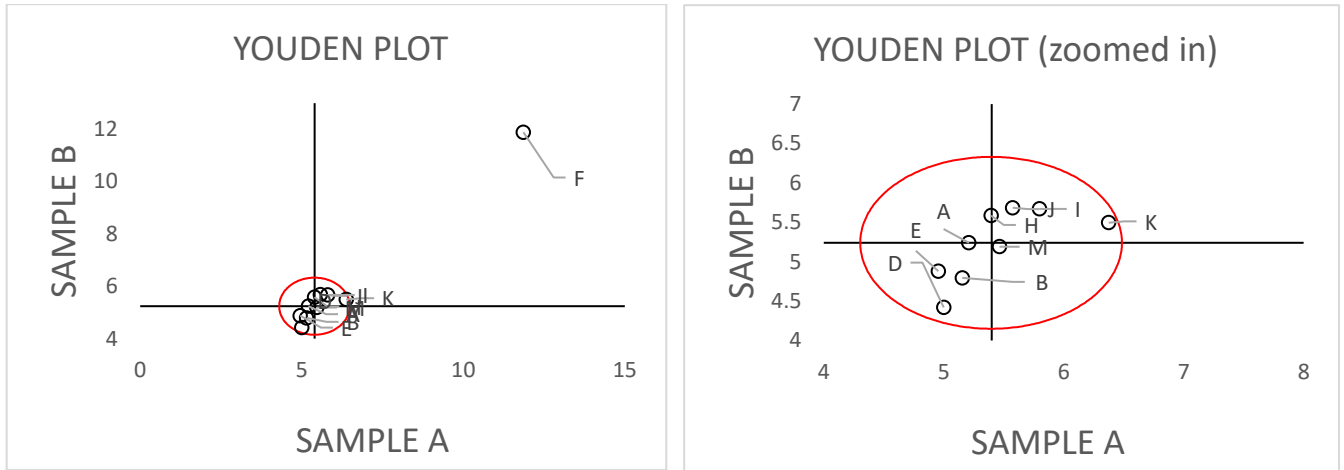
Performance Evaluation 2: Youden plot

Figure 15: Youden Plot – Moisture and Volatile in Shea kernels

Remarks

Lab F shows a strong positive bias on the upper right quadrant of figure 15.

Conclusion

Lab F recorded unsatisfactory performance, particularly showing poor reproducibility exacerbated by questionable precision. Lab K, while showing satisfactory reproducibility, recorded poor precision.

6. Insoluble Impurities in Crude Shea Butter

Test results

Table 11: Overall mean, overall standard deviation, and number of outliers- Insoluble Impurities in Crude Shea Butter

Mean	Standard deviation	Number of outliers
0.05	0.03	4

Table 12: Test results and summary-Insoluble Impurities in Crude Shea Butter

	TEST RESULTS				SUMMARY		
LABS	1	2	3	4	MEAN	Z-SCORE	OUTLIERS #
B	0.08	0.08	0.06	0.06	0.07	0.49	0
D	0.02	0.02	0.03	0.03	0.03	-0.71	0
F	0.06	0.04	0.03	0.04	0.04	-0.23	0
G	0.01	0.02	0.02	0.02	0.02	-0.91	0
H	0.03	0.04	0.02	0.03	0.03	-0.57	0
J	0.04	0.04	0.05	0.04	0.04	-0.20	0
L	0.12	0.13	0.14	0.13	0.13	2.13	4

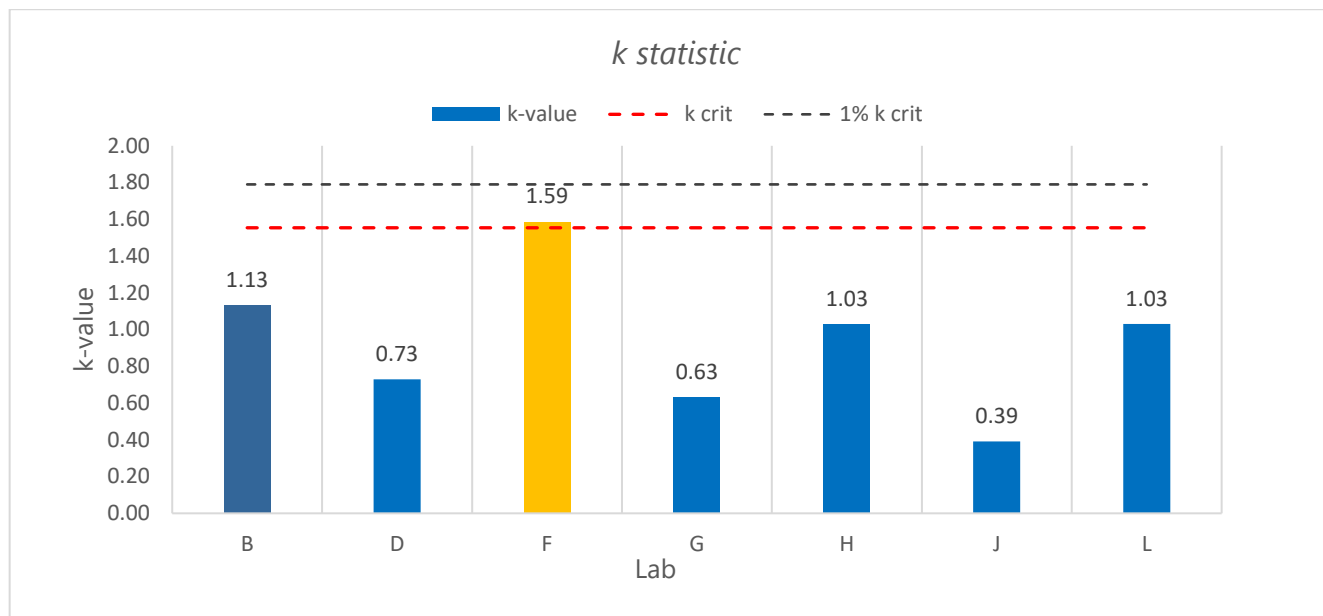**Performance Evaluation 1: Consistency – k statistic and h statistic**

Figure 16: Mandel's k statistic – Insoluble Impurities in Crude Shea Butter
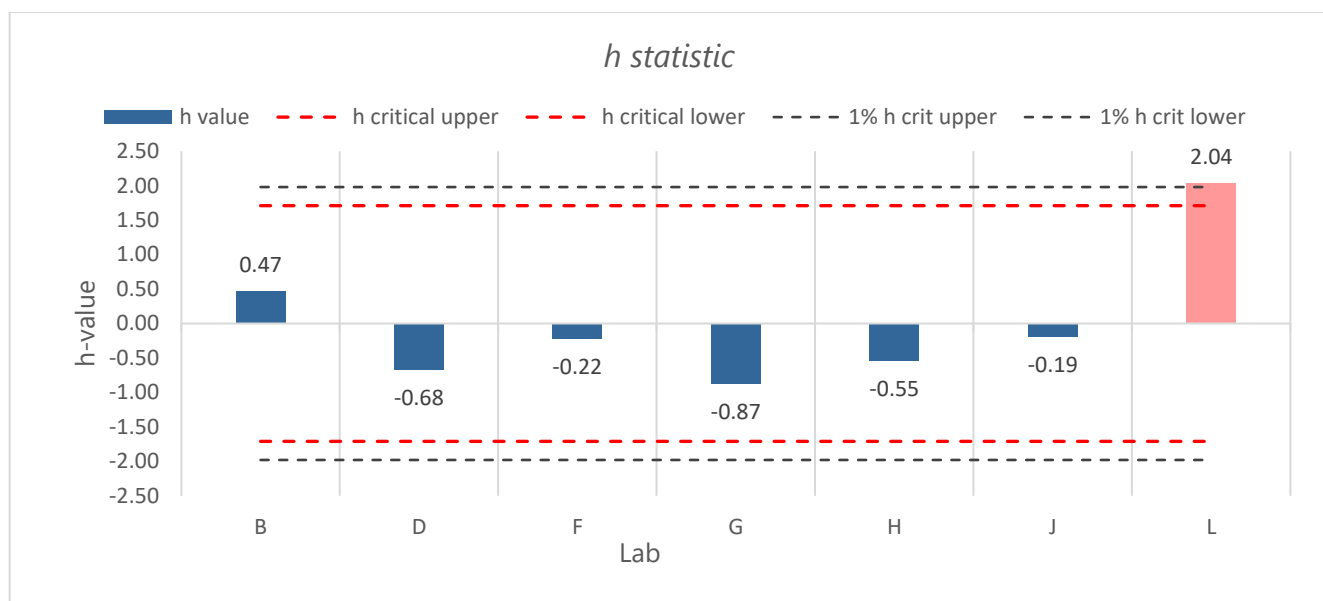

Figure 17: Mandel's h statistic – Insoluble Impurities in Crude shea butter

Remarks

Lab F exhibited poor precision in terms of within lab repeatability. _see figure 16._
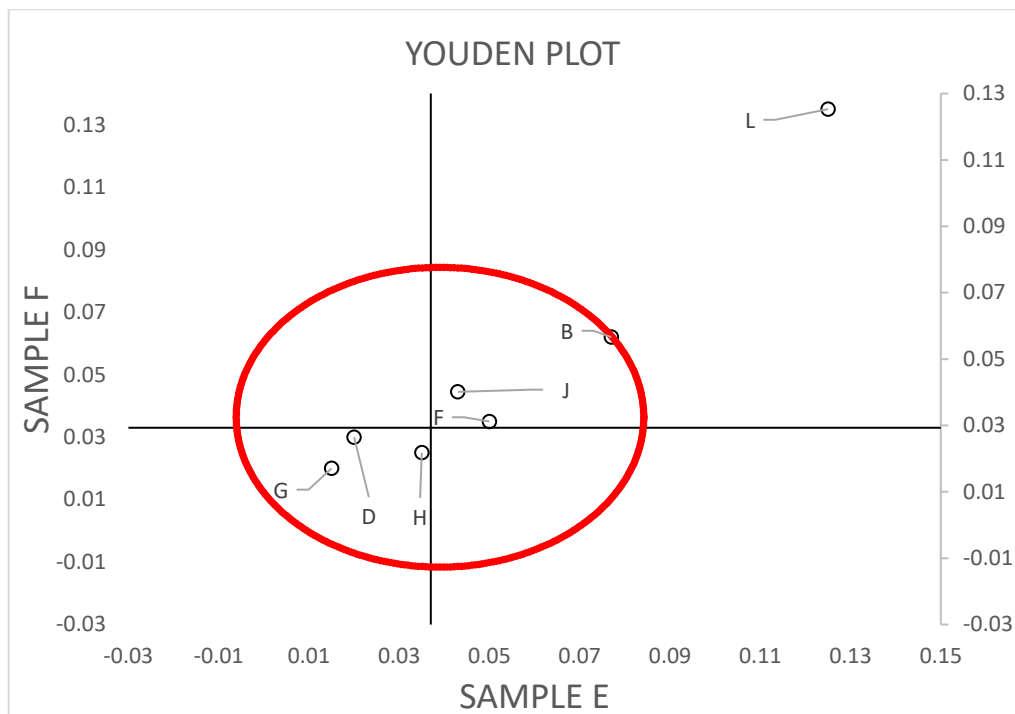
Performance Evaluation 2: Youden plot

Figure 18: Youden plot – Insoluble Impurities in Crude Shea Butter

Remarks

Lab L lies on the far-top right corner of the quadrant showing bias regarding poor reproducibility in the lab results.

Conclusion

Although lab F exhibited satisfactory results in terms of accuracy, it posed a poorer precision in terms of repeatability. On the other hand, lab L recorded unsatisfactory performance in terms of between lab reproducibility.

V. FINAL NOTES

LAB A		
Parameter	**Precision**	**Accuracy**
Free Fatty Acid as oleic in crude shea butter	Satisfactory	Satisfactory
Free Fatty Acid as oleic in shea kernels	Satisfactory	Satisfactory
Oil content	Satisfactory	Satisfactory
Moisture and volatile in crude shea butter	Satisfactory	Satisfactory
Moisture and volatile in shea kernels	Satisfactory	Satisfactory
LAB B		
Free Fatty Acid as oleic in crude shea butter	Satisfactory	Satisfactory

Free Fatty Acid as oleic in shea kernels	Satisfactory	Satisfactory
Oil content	Satisfactory	Satisfactory
Moisture and volatile in crude shea butter	**Poor** **Recommendations:** • Operator retraining on reference method. • Ensure environmental conditions are optimum. For example: high breeze from AC may interrupt weighing balance reading. • Use of effective desiccant/drying agent. • Place scale in a controlled area	**Questionable**; due to poor precision **Recommendations:** • Check precision. If accuracy is poor after precision, • Ensure oven and weighing balances are calibrated. • Ensure internal verification method of calibration is carried out.
Moisture and volatile in shea kernels	Satisfactory	Satisfactory
Insoluble impurities	Satisfactory	Satisfactory
LAB C		
Free Fatty Acid as oleic in crude shea butter	Satisfactory	Satisfactory

Moisture and volatile in crude shea butter	Satisfactory	Satisfactory
LAB D		
Free Fatty Acid as oleic in crude shea butter	Satisfactory	Satisfactory
Free Fatty Acid as oleic in shea kernels	**Poor** **Recommendations** • Operator retraining on reference method. • Ensure solvent is pure • Ensure solvent is neutralized before titration • Ensure solvent is dried after extraction.	Satisfactory
Oil content	**Poor** **Recommendations** • Operator retraining on reference method. • Ensure environmental conditions are optimum. • Serve up the appropriate quantity for extraction. • Ensure in-between grinding is carried out. • Use solvent of appropriate purity. • Use control.	**Poor** (positive bias) **Recommendations** • Ensure precision. • Ensure calibrations of oven and weighing balance are carried out. • Ensure internal verification of calibration is carried out.

Moisture and volatile in crude shea butter	Satisfactory	Satisfactory
Moisture and volatile in shea kernels	Satisfactory	Satisfactory
Insoluble impurities	Satisfactory	Satisfactory
LAB E		
Free Fatty Acid as oleic in crude shea butter	**Questionable** **Recommendations** • Operator retraining on reference method. • Ensure environmental conditions are optimum.	Satisfactory
Free Fatty Acid as oleic in shea kernels	Satisfactory	Satisfactory
Moisture and volatile in crude shea butter	Satisfactory	Satisfactory
Moisture and volatile in shea kernels	Satisfactory	Satisfactory
LAB F		
Free Fatty Acid as oleic in crude shea butter	Satisfactory	Satisfactory

Free Fatty Acid as oleic in shea kernels	Satisfactory	**Poor** (negative bias) **Recommendations** • Ensure calibration of weighing balance. • Ensure internal verification method of calibration is carried out. • Ascertain solvent used is of the right purity.
Oil content	Satisfactory	Satisfactory
Moisture and volatile in crude shea butter	Satisfactory	Satisfactory
Moisture and volatile in shea kernels	**Questionable** **Recommendations** • Retrain operators on reference method. • Ensure environmental conditions are optimum. For example: Ensure oven is closed appropriately.	**Poor** (positive bias) **Recommendations** • Ensure calibration of weighing balance and oven. • Ensure internal verification method of calibration is carried out.
Insoluble impurities	**Questionable** **Recommendations** • Retrain operators on reference method. • Ensure vacuum pump is operating optimally.	Satisfactory

LAB G		
Free Fatty Acid as oleic in crude shea butter	**Questionable** **Recommendations** • Use a control sample.	Satisfactory
Moisture and volatile in crude shea butter	Satisfactory	Satisfactory
Insoluble impurities	Satisfactory	Satisfactory
LAB H		
Free Fatty Acid as oleic in crude shea butter	Satisfactory	Satisfactory
Free Fatty Acid as oleic in shea kernels	Satisfactory	Satisfactory
Oil content	Satisfactory	Satisfactory
Moisture and volatile in crude shea butter	Satisfactory	Satisfactory
Moisture and volatile in shea kernels	Satisfactory	Satisfactory
Insoluble impurities	Satisfactory	Satisfactory

LAB I		
Free Fatty Acid as oleic in crude shea butter	Satisfactory	Satisfactory
Free Fatty Acid as oleic in shea kernels	Satisfactory	Satisfactory
Oil content	Satisfactory	Satisfactory
Moisture and volatile in crude shea butter	Satisfactory	Satisfactory
Moisture and volatile in shea kernels	Satisfactory	Satisfactory
Lab J		
Free Fatty Acid as oleic in crude shea butter	Satisfactory	Satisfactory
Free Fatty Acid as oleic in shea kernels	Satisfactory	Satisfactory
Oil content	Satisfactory	Satisfactory
Moisture and volatile in crude shea butter	Satisfactory	**Questionable** (mild positive bias) **Recommendations**

		• Ensure oven and weighing balances are calibrated. • Ensure internal verification of calibration is carried out.
Moisture and volatile in shea kernels	Satisfactory	Satisfactory
Insoluble impurities	Satisfactory	Satisfactory
Lab K		
Free Fatty Acid as oleic in crude shea butter	Satisfactory	Satisfactory
Free Fatty Acid as oleic in shea kernels	Satisfactory	Satisfactory
Oil content	Satisfactory	Satisfactory
Moisture and volatile in crude shea butter	Satisfactory	Satisfactory
Moisture and volatile in shea kernels	**Poor** **Recommendations** • Retrain operators on reference method. • Ensure environmental conditions are optimum. Ensure oven is closed appropriately during analysis.	Satisfactory

Lab L		
Free Fatty Acid as oleic in crude shea butter	Satisfactory	Satisfactory
Moisture and volatile in crude shea butter	Satisfactory	Satisfactory
Insoluble impurities	Satisfactory	**Poor** (positive bias) **Recommendations** • Ensure weighing balances are calibrated. • Ensure internal verification of calibration is carried out.
Lab M		
Free Fatty Acid as oleic in shea kernels	**Questionable** **Recommendations** • Use a control	Satisfactory
Oil content	**Poor** **Recommendations** • Retrain operators on reference method.	**Poor** (negative bias) **Recommendations** • Ensure weighing balances are calibrated. • Ensure internal verification of calibration is carried out.

	• Ensure the appropriate quantity of samples is served up for analysis. • Ensure in-between grinding is carried out. • Use solvent of the appropriate purity.	
Moisture and volatile in shea kernels	Satisfactory	Satisfactory

VI.   APPENDIX I: Analytical methods

Lab	FFA	M&V	Oil Content	Impurities
K	ISO 660:2009, Solvent used: Hexane+Ethanol(1:1)	ISO 662:1998: Oven Temp: 105^0C ISO 665:2000: dry oven	FOSFA Total Extraction time: 4hrs+2hrs+2hrs In between grinding: Yes	N/A
F	ISO 660, Normality of solution: 0.1N Solvent used: Petroleum ether+Ethanol(1:1)	ISO 662, Oven temperature: 103 +/- 2°C ISO 665 2006 (dry oven) [OBJ]	ISO 659 internal, Total Extraction time: 3hours, In between grinding: Yes	Internal

D	ISO 660. Normality of solution determined: yes, Solution used: NaOH	ISO 662, Oven temperature: 105°C ISO 665 2006 (dry oven)	ISO 659, Total Extraction time: 8hours in between grinding.	ISO 663
B	ISO 660:2006, Normality solution determined: NaOH 0.1N, used solution: Ethanol 70°C	ISO 662: 2006, Oven temperature: 105^0C ISO 665 2006 (dry oven)	NB ISO 659:2006 Extraction time: [OBJ] 6 Hours in between grinding.	ISO 663:2006,
E	ISO 660 Version 2020, Normality solution determined: 0.1N, used solution to solve SHB: NaOH	ISO 662 Version 2016, Oven temperature: 105^0C ISO 665 Version 2020(dry oven), Temp: 105^0C	N/A	N/A

H	ISO 660, Normality of solution determined: 0.192MUsed solution to solve SHB: NaoH, Solvent: Petroleum ether+Ethanol(1:1) [OBJ]	ISO 662:1998, Oven temperature: 105°C ± 2^{0}C ISO 662:1998/ISO 665:2000(dry Oven)	ISO 659:2009 FOSFA International official method, Total Extraction time: 8Hours, (4,2,2) in between grinding	ISO 663, About 20grams of sample weighed and dissolved in 200ml of petroleum ether is filtered with a glass fiber filter
A	FTNIR Analysis,	Oven temperature: 104°C	FTNIR	
J	AOCS Ca 5a – 40 Normality solution determined: 0.0977N, used solution to solve SHB: NaOH	AOCS Ca 2b-38 Oven temperature: 105^{0}C GAFTA130-2.1:2014(dry oven)	GAFTA 130-3.0:2014 Total Extraction time: 6hours in between grinding	AOCS Ca 3a- 46
M	Reference Method not submitted	Reference Method not submitted	Reference Method not submitted	Reference Method not submitted
I	FTNIR Analysis,	FTNIR Analysis,	FTNIR Analysis,	N/A

G	AOCS Ca 5a-40 Used solution to solve SHB: Iso-Propyl Alcohol	AOCS Ca 2c-25 Oven temperature: 130°C	N/A	AOCS Ca 3a-46
C	No reference: 2g of shea into 50ml ethanol, add 2 drops of phenolphthalein and titrate with NaOH. Normality of solution: 0.1N., Solvent used: Ethanol	Oven temperature: 105°C	N/A	N/A
L	ISO 660, [OBJ] Normality solution determined: 0.05M Used solution to solve SHB: Petroleum ether: ethanol	ISO 662:2016, IDT) Oven Temp not stated,	N/A	ISO 663

VII. APPENDIX II: h and k statistics validation

Free fatty acid as oleic in crude shea butter
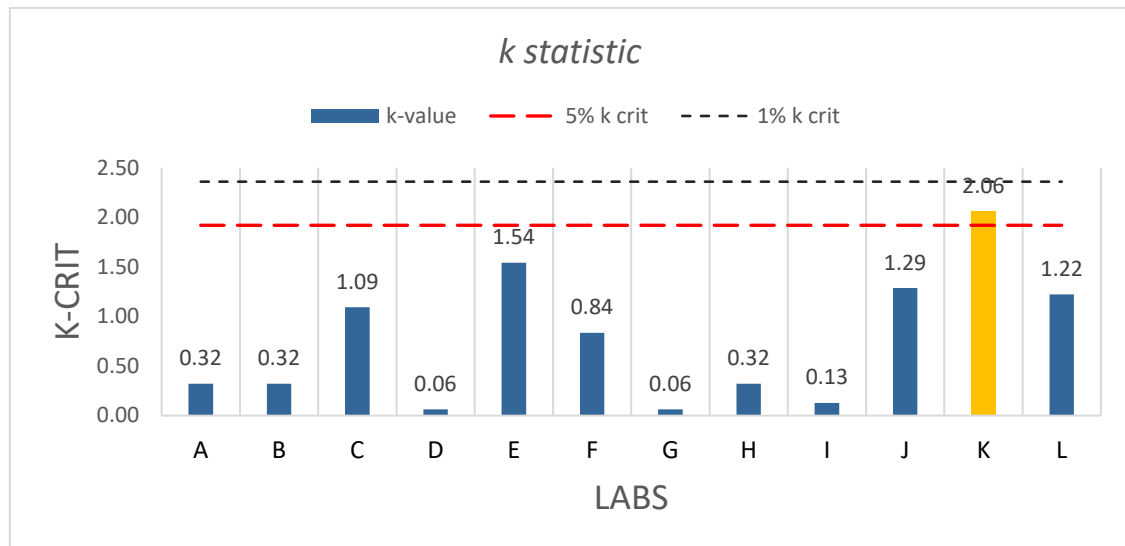
Figure 19: Mandel's k statistic - FFA in shea butter
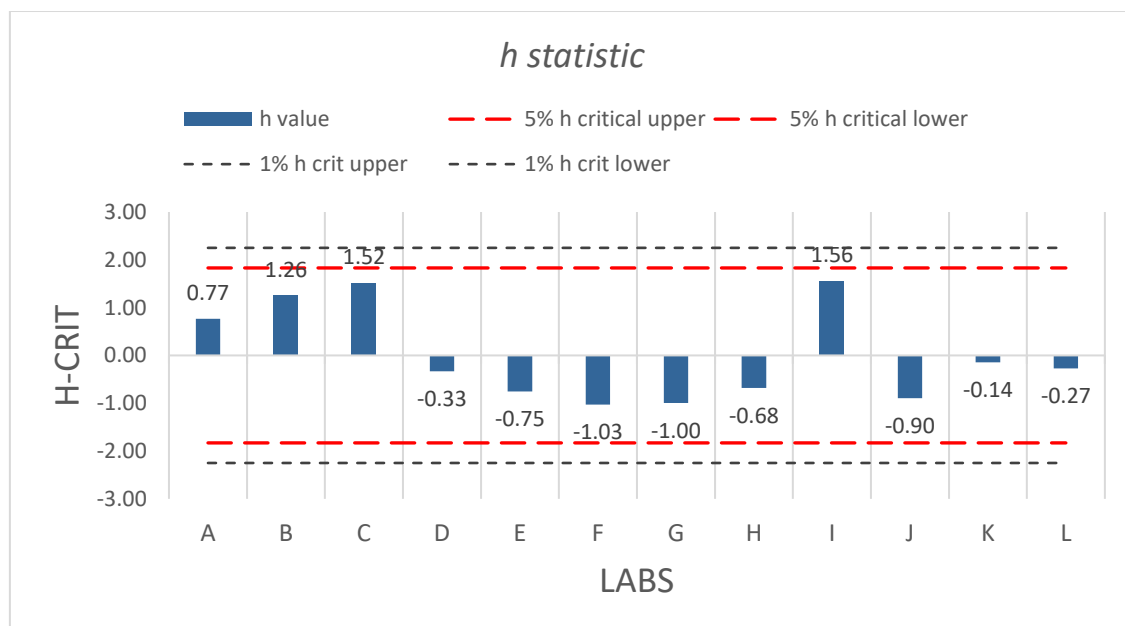
Figure 20: Mandel's h statistic - FFA in shea butter

Free fatty acid as oleic in shea kernel
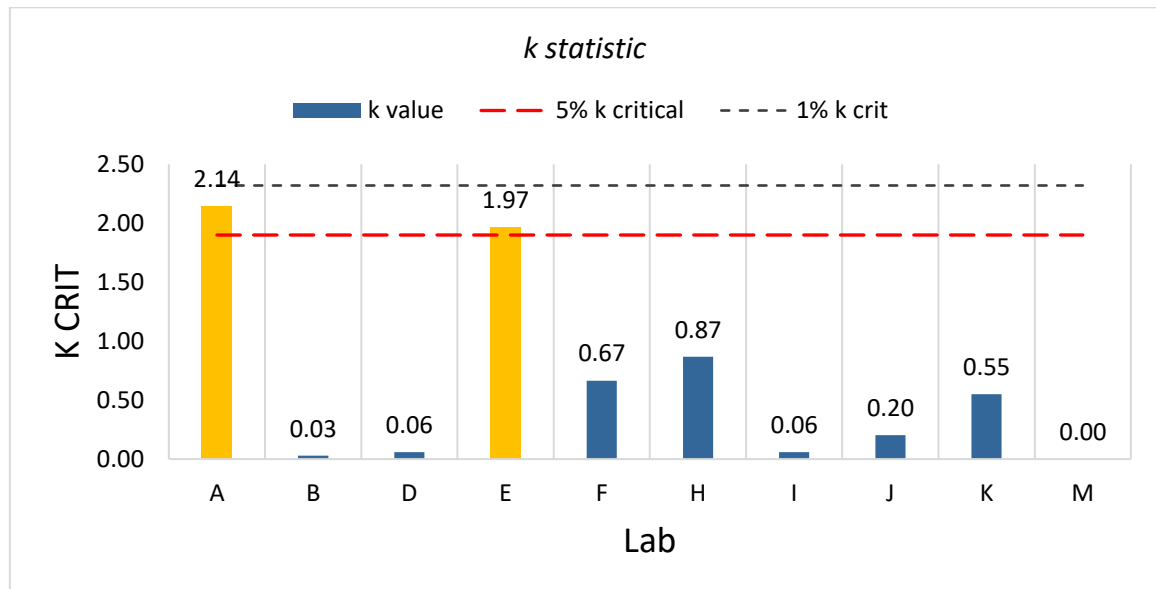
Figure 21: Mandel's k statistic - FFA in shea kernel
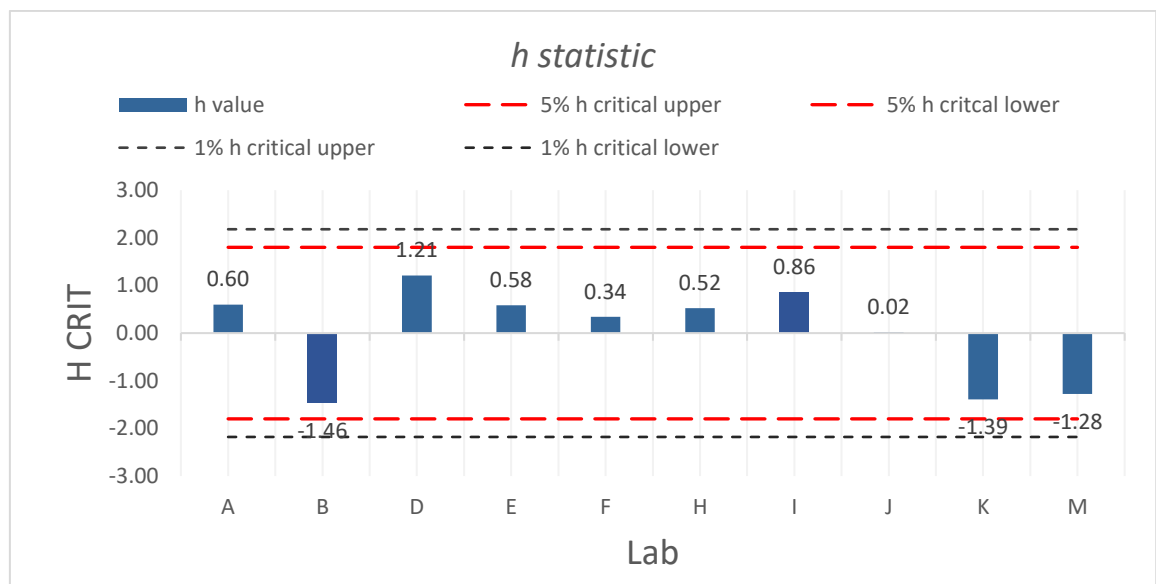
Figure 22: Mandel's k statistic - FFA in shea kernel

Oil content in shea kernel
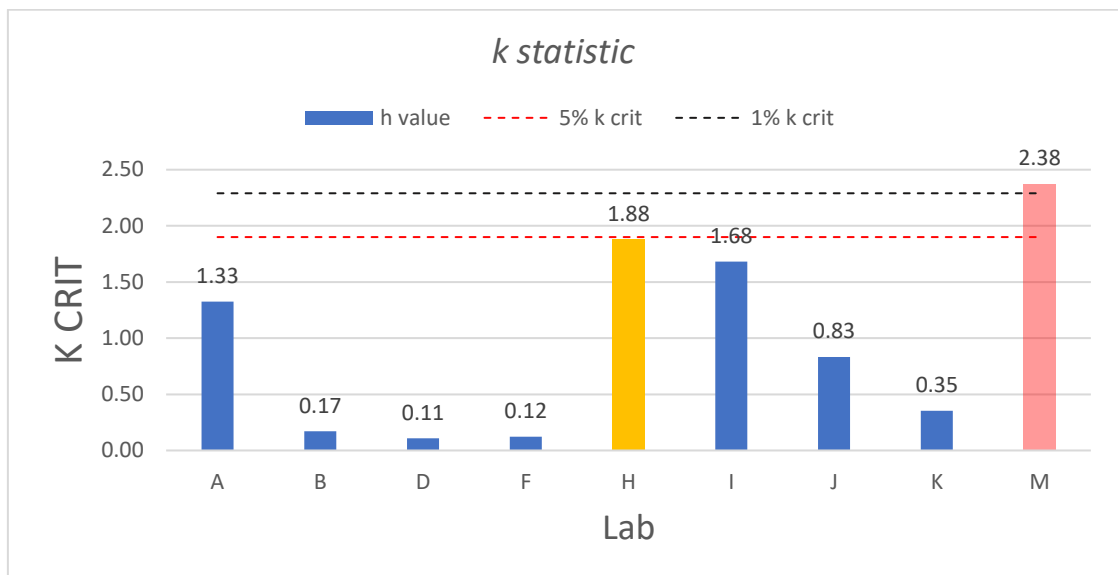
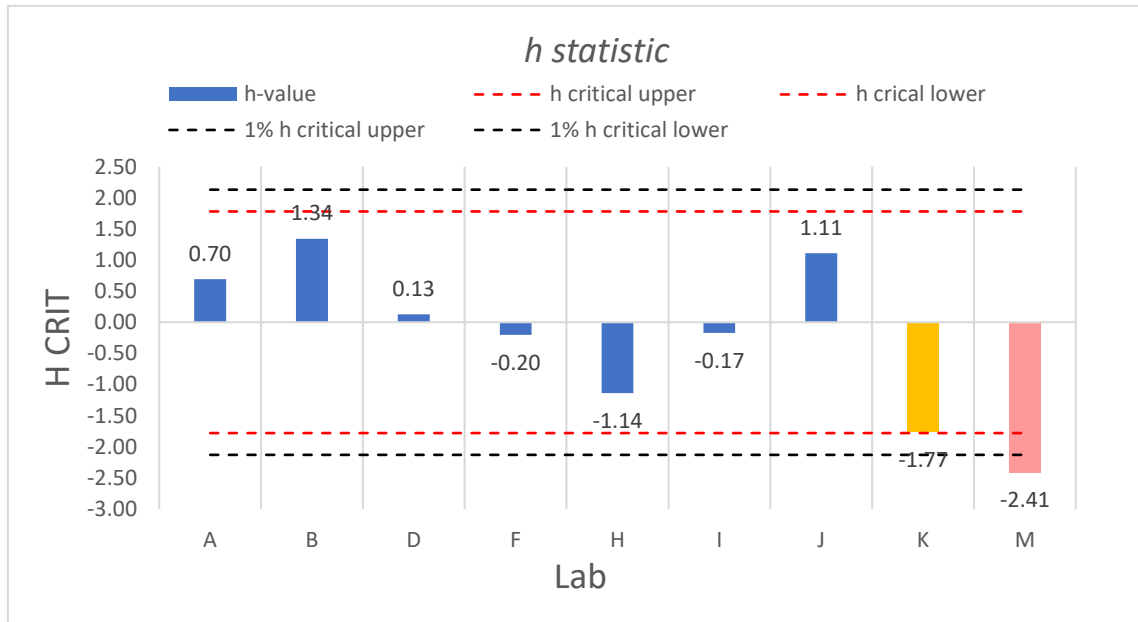Figure 23: Mandel's k statistic - Oil content in shea kernel

Figure 24: Mandel's h statistic - Oil content in shea kernel

Impurities In Crude shea Butter
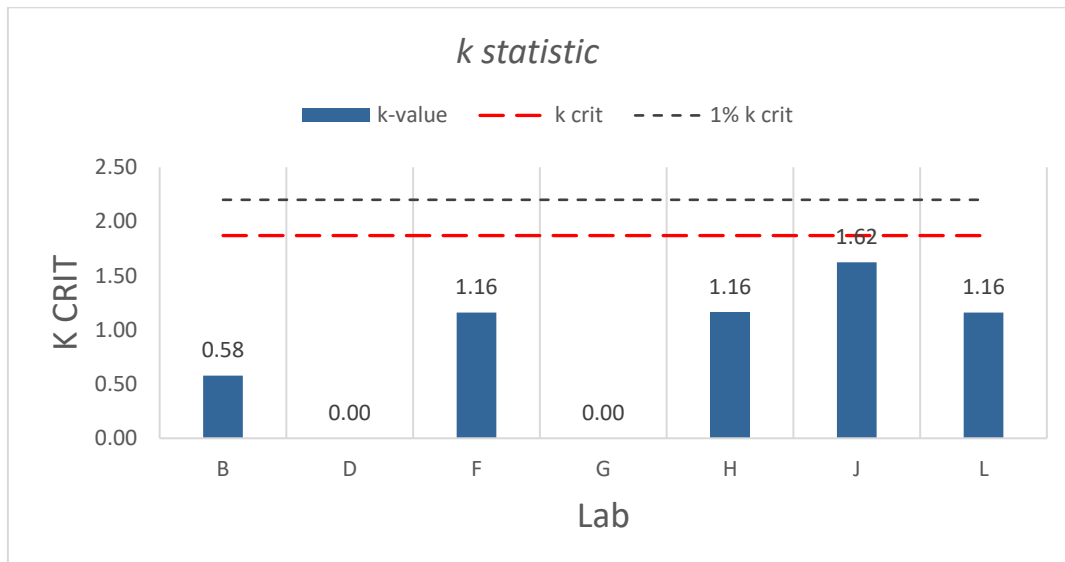
Figure 25: Mandel's k statistic - Impurities in crude shea butter
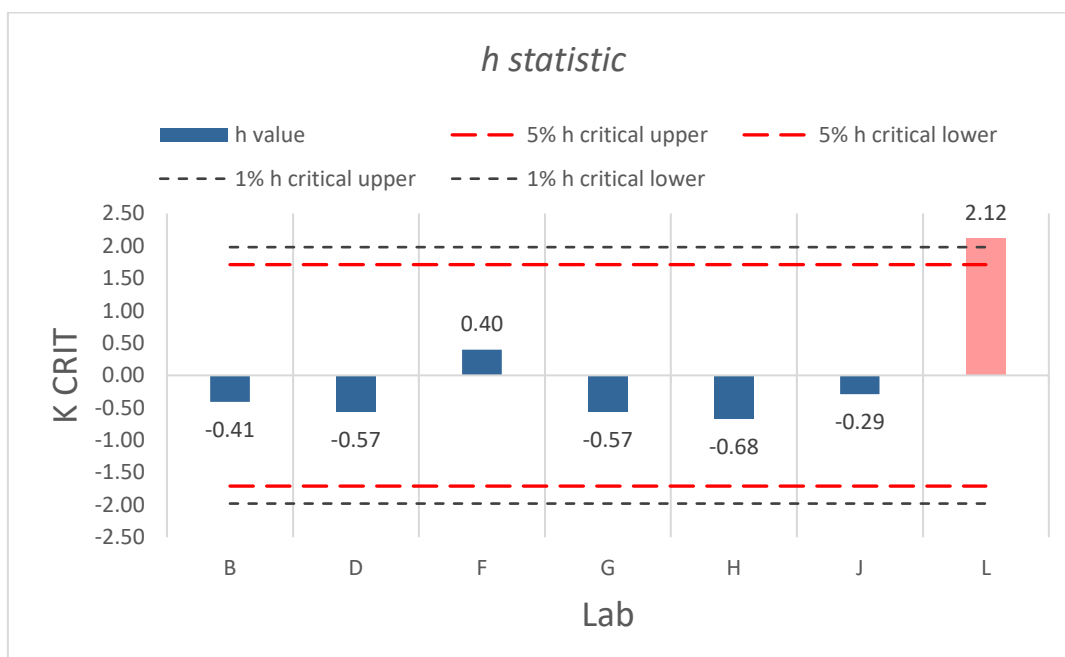
Figure 26: Mandel's h statistic – Impurities in crude in shea butter
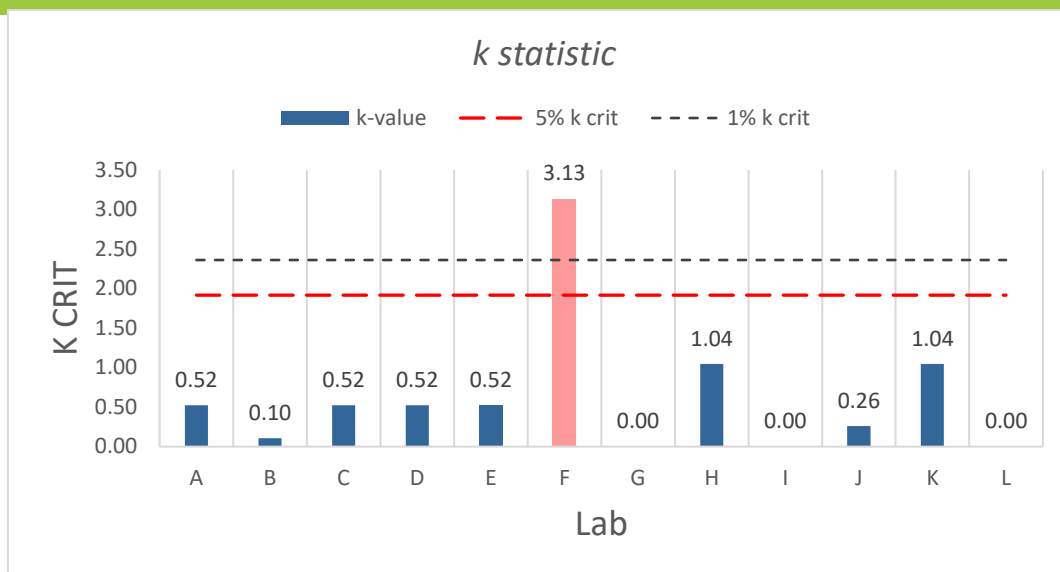
Moisture and volatile in crude shea butter

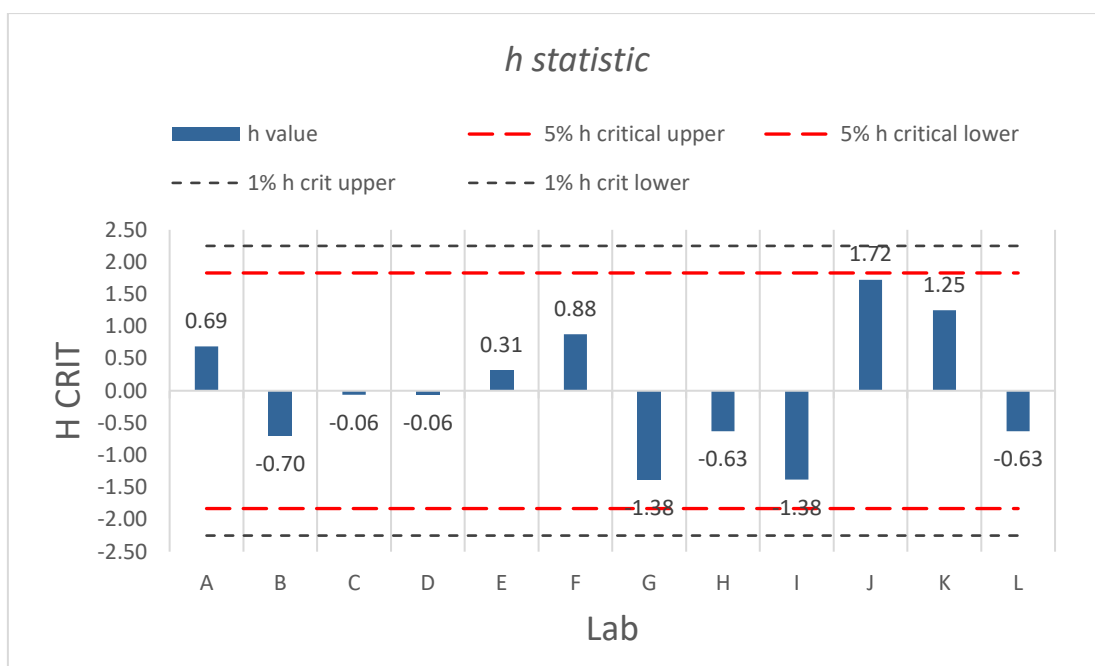Figure 27: Mandel's k statistic – Moisture content in crude shea butter

Figure 28: Mandel's h statistic – Moisture content in crude shea butter
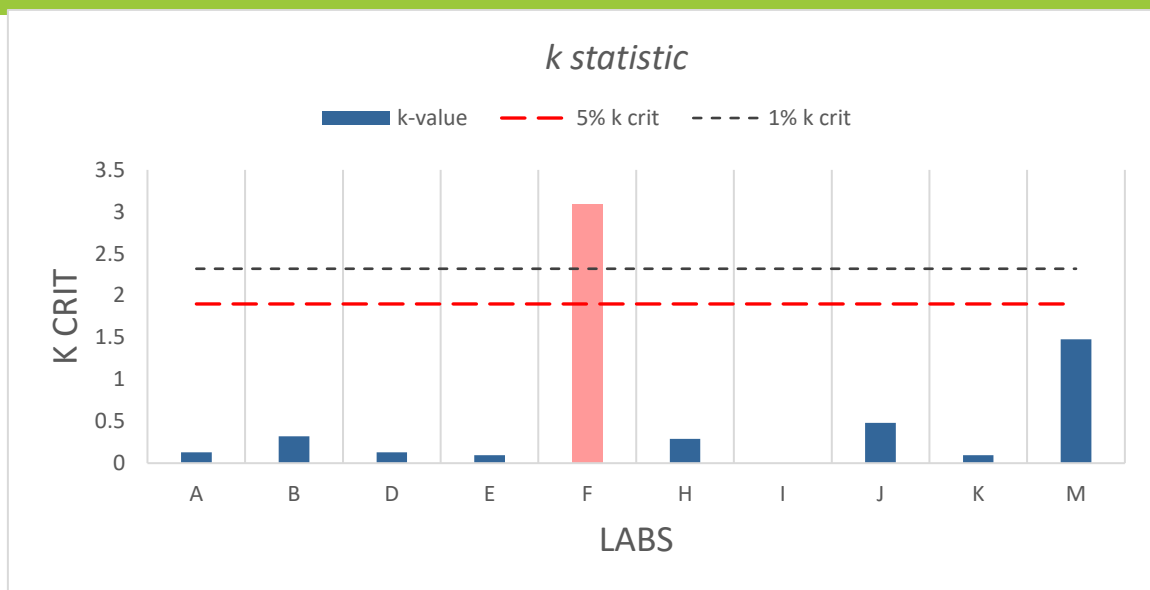
Moisture and volatile in shea nuts
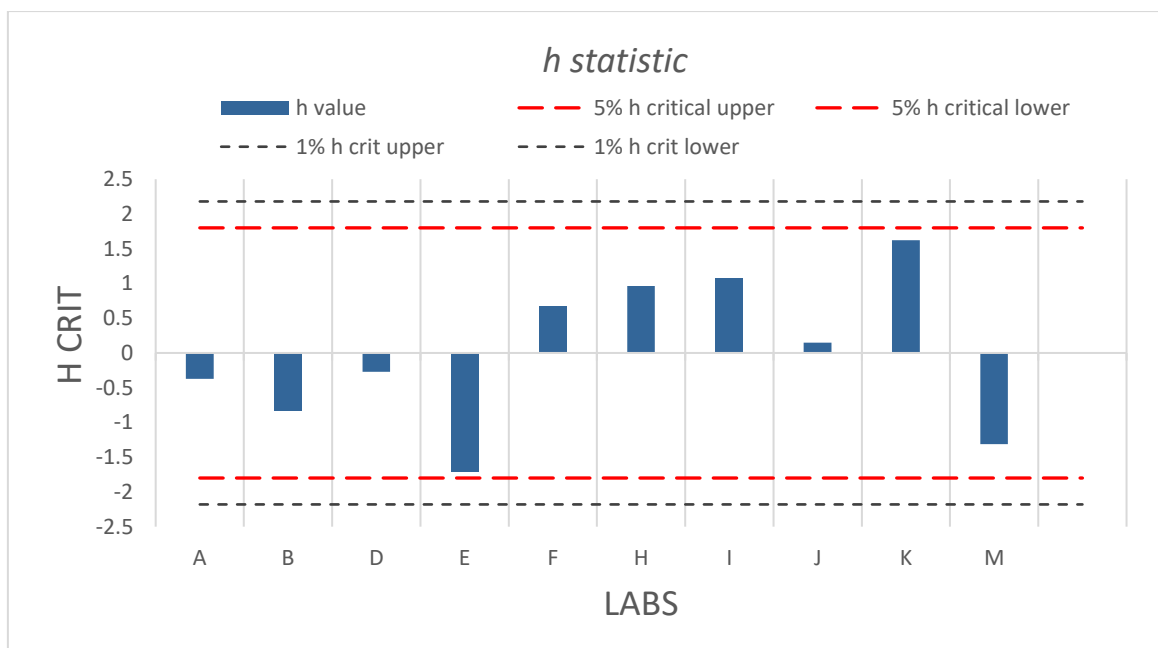
Figure 29: Mandel's k statistic – Moisture content in shea nuts

Figure 10: Mandel's h statistic - Moisture content in shea nuts