JABATAN KIMIA MALAYSIA
PROFICIENCY TESTING PROGRAMME

FODAS 8-18
Year 2018

Caffeine Content in Coffee Product

KIMIA PT Report No. 6/2018

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This proficiency testing programme was conducted by Quality and Research Management Centre in collaboration with Food Chemistry Division of Department of Chemistry Malaysia, Petaling Jaya.

We would like to thank the management and staff of the participating laboratories for their support towards this programme.

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SUMMARY

This proficiency testing programme was conducted in May 2018. A total of thirty-six (36) laboratories from Malaysia and ASEAN countries participated in this PT scheme. One PT item labelled as FODAS 8-18 “Item A” was dispatched to the participating laboratories and all of them had submitted their results.

Results for this study are summarised as follows:

PT Item	Testing Parameter	Assigned value (mg/L)	Expanded Uncertainty (mg/L)	Number of scores, |z| ≤ 2.0	Total number of responses	% |z| ≤ 2.0
Item A	Caffeine	0.67	0.03	28	36	78

The expanded uncertainties are based on an estimated confidence probability of not less than 95% and have a coverage factor of k = 2

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1. PURPOSE OF PROFICIENCY TESTING SCHEME

This proficiency testing (PT) scheme is to assess the competency of laboratories in analysis of caffeine content in coffee product.

2. INTRODUCTION

Proficiency testing is one of the essential elements of quality assurance. The overall objective of a proficiency testing is to provide an independent assessment of the data quality and to improve the quality of measurements. It also provides valuable information on the performance of the laboratory to the laboratory management and the analysts.

This report summarises the results of a proficiency testing scheme on the analysis of caffeine content in coffee product.

The scheme, named FODAS 8-18 is one in a series of proficiency testing schemes under the Jabatan Kimia Malaysia Proficiency Testing Provider (JKM PTP) Programme. This scheme was conducted in May 2018.

2.1 Participation

A total of 36 laboratories registered to take part in this scheme. These laboratories include laboratories of the Department of Chemistry Malaysia, other government laboratories and commercial laboratories from all over Malaysia as well as from ASEAN countries.

Participating laboratories were assigned a laboratory code number to ensure confidentiality.

3. PROFICIENCY TEST ITEM INFORMATION

3.1 Preparation of PT Item

Instant coffee mixture and commercial coffee mixture were prepared by thorough mixing and labelled as FODAS 8-18 Item A. 15 g of Item A was packed in plastic packet and were dispatched to participating laboratories.

3.2 Homogeneity Testing and Stability Testing

Homogeneity testing and stability testing of the PT item was sub-contracted to a laboratory that meets the quality requirements of the scheme’s accreditation.

Ten (10) PT items were selected randomly for homogeneity testing before dispatched to participating laboratories. The results obtained were analysed and found to be sufficiently homogeneous.

Two (2) PT items were selected randomly for stability testing before due date. The results obtained were analysed and found to be sufficiently stable.

4. FEATURES OF THE SCHEME

4.1 PT Item Dispatch and Receipt

One PT item was dispatched to participants in May 2018. The receipt notification form, instructions to laboratories and results sheet were uploaded online at http://ptos.kimia.gov.my. Participants filled the response and result online before the due date. Please refer to **Appendix 1** for specimen copy of instructions sheet.

4.2 Method of Analysis

Participating laboratories were required to analyse the percentage of caffeine content in coffee mixture based on the dry weight (% w/w) using methods routinely employed by the laboratories.

4.3 Results

Participating laboratories were advised to analyse the sample within certain period of time and all of them have submitted their results.

5. STATISTICAL ANALYSIS OF RESULTS

Robust statistics were used in the analysis of the results. The following information is provided for the PT item:

(a) Table of results and the calculated z-scores

(b) Summary statistics

(c) Ordered z-score charts

5.1 Table of Results and z-scores

Table 1 shows the results submitted by each laboratory entered according to code numbers. The z-scores were calculated for each laboratory's result. The results are then assessed based on their z-scores.

5.2 Assigned Value

5.2.1 The assigned value is defined as 'value attributed to a particular quantity and accepted sometimes by convention, as having an uncertainty appropriate for a given purpose'.

5.2.2 For a proficiency test, the assigned value is the best available measurement of the true concentration of an analyte in the test sample.

5.2.3 The assigned value is calculated as the robust average of the results reported by the participants. The robust average was calculated using the procedure described in ISO 13528:2015(E), Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons.

5.2.4 The consensus of participants' results is not traceable to any external reference, so although expressed in SI units, metrological traceability has not been established.

5.3 Uncertainty of the Assigned Value

When the assigned value is calculated as the robust average using the procedure described in 'ISO13528:2015(E), Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons – Annex C', the uncertainty is estimated as:

$$u_{rob\ average} = 1.25 * S_{rob\ average} / sqrt(p)$$

where:

$u_{rob\ average}$ = robust average standard uncertainty
$S_{rob\ average}$ = robust average standard deviation
P = number of results

The expanded uncertainty ($U_{rob\ average}$) is the standard uncertainty multiplied by a coverage factor of 2 for approximately 95% confidence level.

A worked example is set out below:

Uncertainty of the Assigned Value for the percentage of caffeine content

No. of Results (*p*)	36
Robust Average	0.68
Assigned Value	0.67
$S_{rob\ average}$	0.065
$u_{rob\ average}$	0.01
k	2
$U_{rob\ average}$	0.03

The Assigned Value for percentage of caffeine content is **0.67 mg/L ± 0.03 mg/L.**

5.4 Standard Deviation for Proficiency Assessment

5.4.1 The standard deviation for proficiency assessment (σ) is a parameter that is used to provide a scaling (scale) for the laboratory deviations from the assigned value and this value is used for calculation of participant z-score as presented in the equation below.

$$\sigma = X * CV$$

Where:

σ = standard deviation for proficiency assessment
X = assigned value
CV = interlaboratory coefficient of variation

5.4.2 Based on previous proficiency testing studies conducted by the PT Section, the interlaboratory coefficient of variation (CV) for percentage of caffeine content was 6%.

5.4.3 This σ is in accordance with the value obtained using predictive contemporary model, Horwitz equation and the results are presented in **Table a** below.

PT Item	Testing Parameter	Assigned value (mg/L)	Horwitz CV (%)	FODAS 8-18 CV (%)
Item A	Caffeine content	0.67	6	6

Table a: Values obtained from Horwitz Equation (CV)

5.4.4 Therefore, a standard deviation for proficiency assessment equivalent to 6% coefficient variation (CV) was used to calculate z-scores for percentage of caffeine content.

5.5 Calculation of z-score

5.5.1 For each participant result, a z-score is calculated according to equation

$$Z = \frac{(x - X)}{\sigma}$$

below:

Where:

Z = z-score
x = participant result
X = study assigned value
σ = standard deviation for proficiency assessment

5.5.2 The interpretation of z-score is as follows:

$\|z\| \leq 2.0$	:	"satisfactory" performance and generates no signal
$2.0 < \|z\| < 3.0$	:	"questionable" performance and generates a warning signal
$\|z\| \geq 3.0$	:	"unsatisfactory" performance and generates an action signal

5.5.3 An outlier result indicates that the result is inconsistent with the remainder of the results.

5.6 Summary Statistics

A summary of the robust statistics at the bottom of the table of results include:

5.6.1 The number of results received for the sample (No. of results).

5.6.2 The mean of the result i.e. the average value (Mean).

5.6.3 The median of the results i.e. the middle value (Median).

5.6.4 The standard deviation of the results.

5.6.5 The assigned value that had been used in the proficiency testing.

5.6.6 The uncertainty of the assigned value.

5.6.7 The standard deviation for proficiency assessment (6 % CV).

5.6.8 The robust average is an average calculated by a robust algorithm.

5.6.9 The robust standard deviation is a standard deviation calculated by a robust algorithm.

5.6.10 The maximum and minimum results.

5.6.11 The range of results.

5.7 Comments on Results

5.7.1 **Table b** the participants that obtained questionable and unsatisfactory results, (i.e. $2<|z|<3$ & $|z|\geq 3$).

PT Item	Parameter Testing	z-score	Laboratories
Item A	Caffeine Content	$\|z\|\geq 3$	001, 011, 013 & 295
		$2<\|z\|<3$	017, 098, 105 & 136

Table b: Laboratories with questionable and unsatisfactory z-score

5.8 Comments on Methods

All participating laboratories had given the analytical methods used in the analysis. Most laboratories quoted analysis by in house methods and instrumentation technique using High Performance Liquid Chromatography as given in **Appendix 2.**

5.9 Ordered z-score Charts

5.9.1 **Figure 1** shows the z-scores of each laboratory, in order of magnitude and marked with respective code numbers to enable the laboratories to compare their performance relative to other laboratories.

5.9.2 **Figure 2** shows the summary z-score chart by participants.

5.9.3 Any laboratories with its “bar” extending beyond +2 and -2 lines are advised to carry out investigation into possible causes of questionable and outlier.

6. REFERENCES

6.1 MS ISO/IEC 17043:2010 Conformity assessment – General requirements for proficiency testing

6.2 Jabatan Kimia Malaysia Proficiency Testing Provider Quality Manual

6.3 The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories (IUPAC Technical Report), in Pure and Applied Chemistry, Vol. 78, No. 1, pp. 145-196, 2006

6.4 ISO 13528:2015(E), Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons

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Table 1a: Result and z-scores

No.	Laboratory Code Number	Caffeine (% w/w)		
		Item A	z-score	Expanded Uncertainty
1	001	0.52	**-3.73**	0.05
2	010	0.68	0.25	0.07
3	011	0.85	**4.48**	-
4	013	0.46	**-5.22**	-
5	017	0.77	**2.49**	-
6	018	0.67	0.00	0.07
7	023	0.67	0.00	0.06
8	027	0.64	-0.75	18%
9	054	0.72	1.24	0.072
10	055	0.67	0.00	0.02
11	062	0.61	-1.49	3.67%
12	069	0.72	1.24	-
13	070	0.68	0.25	0.054
14	079	0.68	0.25	0.041
15	098	0.55	**-2.99**	0.03
16	104	0.67	0.00	0.1
17	105	0.77	**2.49**	-
18	115	0.72	1.24	0.1
19	126	0.70	0.75	0.14
20	136	0.56	**-2.74**	0.03
21	150	0.72	1.24	-
22	192	0.71	1.00	0.0691
23	214	0.63	-1.00	0.06
24	217	0.60	-1.74	0.02
25	238	0.70	0.75	0.04
26	240	0.69	0.50	0.07
27	257	0.60	-1.74	+/-0.05

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Table 1a: Result and z-scores

No.	Laboratory Code Number	Caffeine (% w/w)		
		Item A	z-score	Expanded Uncertainty
28	259	0.65	-0.50	-
29	264	0.70	0.75	0.03
30	268	0.68	0.15	0.02
31	269	0.69	0.50	0.17
32	271	0.68	0.25	0.07
33	272	0.59	-1.99	0.1
34	278	0.68	0.25	-
35	286	0.68	0.25	0.02
36	295	0.52	**-3.73**	0.14

No.of results	**36**
Mean	**0.66**
Median	**0.68**
Standard Deviation	**0.08**
Assigned Value	**0.67**
Uncertainty	**0.03**
SD for proficiency assessment *(set @ 6% CV)*	**0.04**
Robust Average	**0.68**
Robust SD	**0.065**
Maximum	**0.85**
Minimum	**0.46**
Range	**0.39**

z-scores outside the satisfactory range, i.e. IzI > 2, are shown in ***bold***

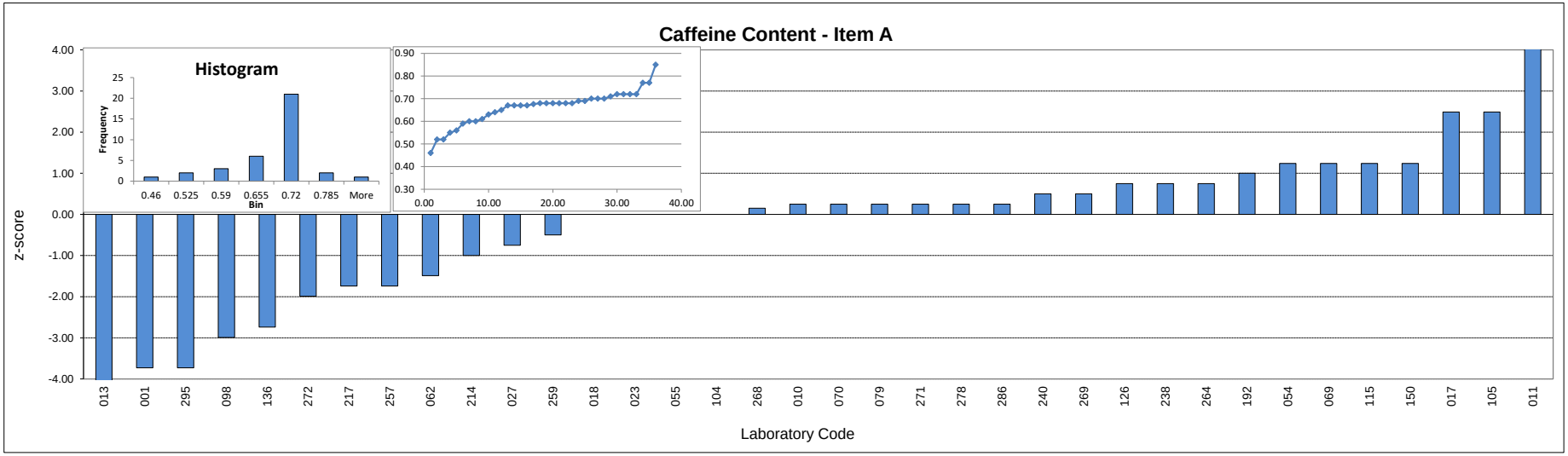

Figure 1: Ordered z-score chart for Caffeine Content - Item A

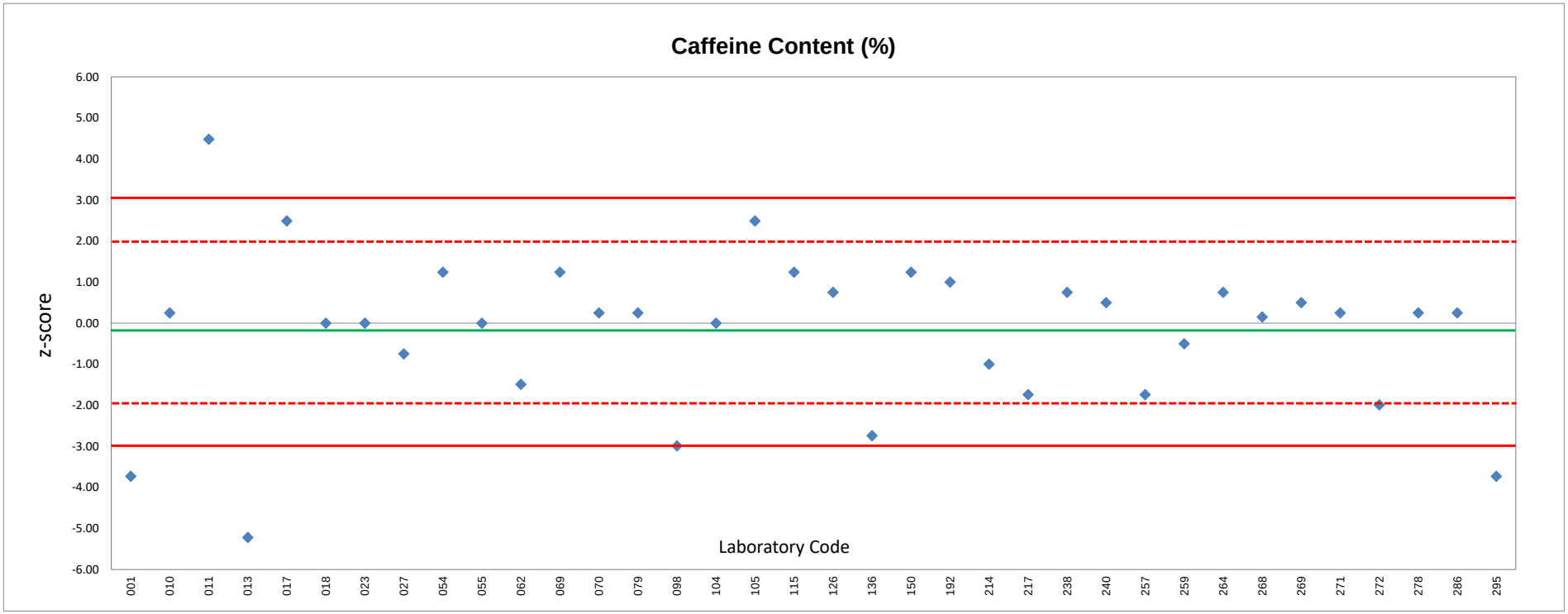

Figure 2: Summary z-score chart by participants for Caffeine Content

Appendix 1

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INSTRUCTIONS TO LABORATORIES

PROMPTLY REPLY THE RECEIPT OF PROFICIENCY TEST ITEM (PT ITEM) FORM AS SOON AS YOU RECEIVE THE PT ITEM

1. PT Item

Each laboratory is supplied with one (1) pack of coffee product (1 x 15 g) and labelled as:

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Caffeine Content
Item A

PT Item ID No.: ☐

2. Tests to be performed

Item A: **Caffeine Content**
Treat the PT item as a routine sample.

3. Preservation/ Storage

You are advised to keep the PT item in a desiccator or cool dry place until analysis.

4. Test methods

You may use any method of analysis you wish.

5. Reporting

(a) For statistical evaluation purposes, report results in **the percentage of caffeine content based on the dry weight, % w/w** to **two (2) decimal places.**

(b) In addition to reporting the results, please report sample weight used per analysis, the method of analysis and type of equipment/ technique used.

(c) **It is the responsibility of the participating laboratories to avoid collusion or falsification of results.** Where any collusion or falsification is proven by Jabatan Kimia Malaysia, the participant's results shall be excluded from the report and the laboratory manager shall be notified.

(d) **Any transposition, miscalculation and alteration of results by participants after the closing date will not be accepted and entertained.**

6. You are reminded that the ability to report results in the specified units and within the given time scale are part of the proficiency test.

Appendix 1

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INSTRUCTIONS TO LABORATORIES

7. Testing should commence **AS SOON AS POSSIBLE** after receiving the PT items and report results in the Proficiency Testing Online System (PTOS) at http:\\ptos.kimia.gov.my no later than **7th June 2018**. Late result submission will not be evaluated.

8. As a guide, the expected range is **0.10 – 2.00 % w/w.**

9. Damaged PT item

In the event of damaged PT item or broken security seal, a new set of PT item will be replaced within three (3) working days after notification to PT provider. This only applies if PT provider finds the PT item is unsuitable for analysis.

10. Safety

a) PT items are for laboratory use only
b) Use pipette fillers; DO NOT fill pipette using the mouth
c) Use of safety glasses, gloves and fume hoods is recommended when handling PT items.
d) Wash any splashes and spills with plenty of water.

11. NOTE: SHAKE PT ITEM THOROUGHLY BEFORE ANALYSIS.

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Appendix 2: Methods and Instrumentation or Technique Used by Participants

Description	Laboratory Code Number
Method of Analysis:	
AOAC 976.08	062, 023
AOAC 979.08	136, 257
GB 5009.139-2014	271
In House Method	001,010, 011, 013, 017, 027, 055, 070, 079,104, 105, 126, 150, 192, 214, 238, 240, 259, 264, 268, 278, 286, 295
In House STP/FP/042/V1 Based On Journal Markus Brannstrom, Karin Edenteg Autumn 2002 By HPLC	098
In-House Method Based on AOAC 925.17	069
ISO 10095:1992	018
ISO 20481:2008	054
MS 1235:1991	272
MS 1360:1994	217, 269, 115
Instrumentation:	
HPLC	054, 278, 011, 295, 238,070, 001 ,264, 105, 062, 150, 079, 017, 214, 192, 104, 217, 269, 286, 069, 055, 023, 027, 136, 013, 115, 018, 257 ,098, 010, 240, 126, 271, 272, 268
UPLC	259
Is your laboratory accredited to ISO/IEC 17025?	
Yes	054, 011, 070, 001, 264, 062, 150, 079, 214, 192, 104, 217, 269, 069, 055, 023, 027, 136, 115, 018, 257, 098, 259, 010, 240, 126, 271, 268
No	278, 295, 238, 105, 017, 286, 013, 272