



PTP
No.0002

Chemical Analysis

**NFI-PTC 07-2018: NaCl (g/100g) in Canned Food
(Baby Corn in Brine)**

May 2018

LABORATORY SERVICES DEPARTMENT



Proficiency Testing Report



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SUMMARY

This summarizes the chemical proficiency testing program of NFI-PTC 07-2018: NaCl (g/100g) in Canned Food (Baby Corn in Brine). There were 28 laboratories participating in this program. The test materials were quality controlled to ensure that consistent levels were achieved throughout the batch by conducting homogeneity test prior to dispatch. Stability testing was conducted at the end of shelf life which was the deadline for all laboratories to run the test – to ensure the integrity of the test materials.

The participant's results were 28 laboratories which were assessed statistically in the form of a robust Z-Score as Z' -Score for performance evaluation for each laboratory due to the standard uncertainty of the assigned value $u(x_{pt})$ was greater than $0.3\sigma_{pt}$. The overall competency evaluation was that there were 22 laboratories (78.6%) had satisfactory performance with $|Z'| \leq 2.00$, while 3 laboratories (10.7%) had questionable performance with $2.00 < |Z'| < 3.00$, and 3 laboratories (10.7%) had unsatisfactory performance with $|Z'| \geq 3.00$.

This proficiency testing program accomplished with smooth operation as expected, and every participating laboratory benefits and acquires valuable information for advancement whatever the performance result is.

1. INTRODUCTION

1.1 Proficiency Testing

Proficiency testing (PT) is a valuable tool for quality assurance of testing and measurement. It is a requirement of accreditation to ISO/IEC 17025 that laboratories take part in proficiency testing schemes. PT involves the use of interlaboratory comparisons for determination of laboratory performance with the purpose of producing laboratory results of greater reliability. Whatever the performance result is, every participating laboratory benefits and acquires valuable information for further advancement in all degree of their current performance.

For the improvement and satisfaction to the requirement of ISO/IEC 17025 of laboratories, hence the Division of Proficiency Testing Services, Department of Laboratory Services, National Food Institute (NFI) provides PT program to follow the international standard ISO/IEC 17043: 2010, Conformity Assessment – General Requirements for Proficiency Testing. This program was named “Chemical Analysis NFI-PTC 07-2018: NaCl (g/100g) in Canned Food (Baby Corn in Brine)” with the focus on %NaCl since it is one of the most common tests for food products in brine.

To ensure performance confidentiality of all participants, a series of Laboratory Number is used in place of participant’s name. A unique Laboratory Number is assigned to each laboratory.

NFI offers Internet access for proficiency testing at ‘<http://pt.nfi.or.th>’ for fast, convenient and easy access for participants to register, submit form / data / test result / request, view other information, and communicate with the provider. All participants are encouraged to utilize all on-line features and more for complete and clear action for their own advantage.

1.2 NaCl

Sodium chloride is a compound formed from the ionic bonding of sodium and chloride that the result is a salt that is very important biologically and commercially. The chemical formula for sodium chloride is NaCl that has a molar mass of 58.44 grams per mole. It appears as a solid, clear crystal with little or no odor. As a salt, sodium chloride dissolves well in water, and the ions in the crystals will separate when in solution.

2. TEST MATERIALS

2.1 Sample Preparation

Test materials were produced by blending baby corn in brine. One hundred and fifty grams of test materials were placed in glass jar, and then sterilized at 121°C for 15 mins. After sterilization, the glass jars were then sealed with cap topped with shrink film seal. Each was labeled with ID numbers and stored at ambient temperature (25-35°C) for the homogeneity test, and further steps.

2.2 Homogeneity

To insure that consistent levels of %NaCl was achieved throughout the batch of test materials, homogeneity test was conducted on 27 April 2018. The random number generator from the website 'random.org' was used to randomize the order of the samples. Ten out of all the glass jars were randomly picked for homogeneity test by carrying out NaCl (g/100g) in duplicate prior to dispatch.

2.3 Stability

To ensure the constancy of the test, materials not being diminished over time and the transport effect, stability testing was conducted at the end of shelf life. Therefore, four sets of the test materials at NFI were kept at ambient temperature (25-35°C) until the final test or after this date. On 23 May 2018, the test materials were examined in duplicate for NaCl (g/100g).

The work on homogeneity and stability tests was conducted by the supporting laboratory – Division of Chemical Laboratory, National Food Institute. The homogeneity and stability data were shown in Table 3 in the Appendix.

2.4 Sample Distribution

Upon completion of homogeneity test, parcel of test materials and documents were dispatched on 14 May 2018 to each participant by an express logistics company with 24-hour guaranteed delivery. Documents enclosed in the box were as follows:

- 1) Letter of Introduction
- 2) Test Instruction Sheet
- 3) Receipt Form

- 4) Results Form
- 5) Technical Form
- 6) On-line Instruction Sheet

Note:

- Filling on-line preferred for 3), 4) and 5).
- Using test methods (materials, procedures, conditions, etc.) consistent with each laboratory routine practice.
- One glass jar of test material delivered for each participant.

Participants were required to report and submit their enumeration of the test material in g/100g by the closing date – “30 May 2018”.

3. REPORTING

Having finished the calculation of the test result, each participating laboratory, then submitted its test result by the closing date for analysis, and for the summary report to be made. With all these data, together with those received through PT provider’s quality control, statistical analysis for homogeneity test, stability test, and others were carried out according to the standard ISO 13528: 2015.

The statistics summarizes are given in Table 1. Table 2 summarizes the result of participants. Technical data of methods used by each laboratory in relation to accreditation, reference, and others are summarized in Table 4 in the Appendix.

Interim report and final report could be uploaded to the website pt.nfi.or.th within two weeks and one month, respectively. After the closing date, while hard copies of interim and final report could be sent to participants with no Internet access by registered mail.

4. STATISTICAL EVALUATION OF RESULTS

Statistical use for this proficiency testing program is robust Z-Score that is based on ISO 13528: 2015.

4.1 Calculation of the assigned value, x_{pt} and standard uncertainty of the assigned value, $u(x_{pt})$

4.1.1 Assigned value, x_{pt} (assigned value for proficiency assessment) was set as the consensus of the results submitted by participants. The procedure used to derive this consensus involved some or all of the following:

4.1.1.1 Removal of data that was not considered valid.

4.1.1.2 Derivation of the robust mean (x^*) of the final data set using a robust statistical procedure that calculate by Algorithm A.

4.1.2 Assessment of the standard uncertainty of the assigned value, $u(x_{pt})$

$$u(x_{pt}) = 1.25 \times \frac{s^*}{\sqrt{p}} \dots\dots\dots(1)$$

Where s^* is robust standard deviation calculated by Algorithm A

and p is number of result.

When the assigned value is calculated from the formulation of the proficiency test item, the standard uncertainty $u(x_{pt})$ of the assigned value is calculated according to equation (1). The standard uncertainty of the assigned value is usually compared with a criterion (2), that $u(x_{pt})$ shall be smaller than $0.3\sigma_{pt}$, then the uncertainty of the assigned value is negligible and need not be included in the interpretation of the results of the proficiency test.

$$u(x_{pt}) < 0.3 \sigma_{pt} \dots\dots\dots(2)$$

4.2 Calculation of standard deviation for proficiency assessment, σ_{pt}

The standard deviation for proficiency assessment used in this proficiency testing program was a pooled standard deviation that it has been calculated by combining results from competent laboratory from previous rounds of the same proficiency testing schemes.

Pooled standard deviation is the standard deviation remaining after removing the effect of special cause variation as calculated by equation (3).

$$RSD_{pooled} = \frac{\sqrt{(n_1-1)RSD_1^2 + (n_2-1)RSD_2^2 \dots + (n_k-1)RSD_k^2}}{n_1 + n_2 \dots + n_k - k} \dots\dots\dots(3)$$

Where k is number of Proficiency Testing Rounds

$RSD_1, RSD_2, \dots, RSD_k$ is relative standard deviations of competent laboratory (Z-Score ≤ 2.00) from each Proficiency Testing Rounds

and n_1, n_2, \dots, n_k is numbers of competent laboratory (Z-Score ≤ 2.00) from each Proficiency Testing Rounds

This *pooled RSD* is applied to σ_{pt} , which are calculated by equation (4)

$$\sigma_{pt} = \frac{(\%pooled\ RSD) \times (x_{pt})}{100} \dots\dots\dots(4)$$

4.3 Calculation of robust Z' -Score

If $u(x_{pt}) > 0.3 \sigma_{pt}$, the Z' -Score for a proficiency test result is calculated as follows
 Z' -Score equation (5)

$$Z'\text{-Score} = \frac{x_i - x_{pt}}{\sqrt{\sigma_{pt}^2 + u^2(x_{pt})}} \dots\dots\dots(5)$$

Where x_i is the participant's reported result,

x_{pt} is the assigned value

σ_{pt} is the standard deviation for proficiency assessment

and $u(x_{pt})$ is the standard uncertainty of assigned value.

4.4 Z' -Score assessment criteria

$$\begin{aligned} |Z'| \leq 2.00 & \text{ is Satisfactory} \\ 2.00 < |Z'| < 3.00 & \text{ is Questionable} \\ |Z'| \geq 3.00 & \text{ is Unsatisfactory} \end{aligned}$$

5. REFERENCES

ISO/IEC 17043: 2010. Conformity assessment – general requirements for proficiency testing.

ISO 13528: 2015. Statistical methods for use in proficiency testing by interlaboratory comparison.

6. RESULTS

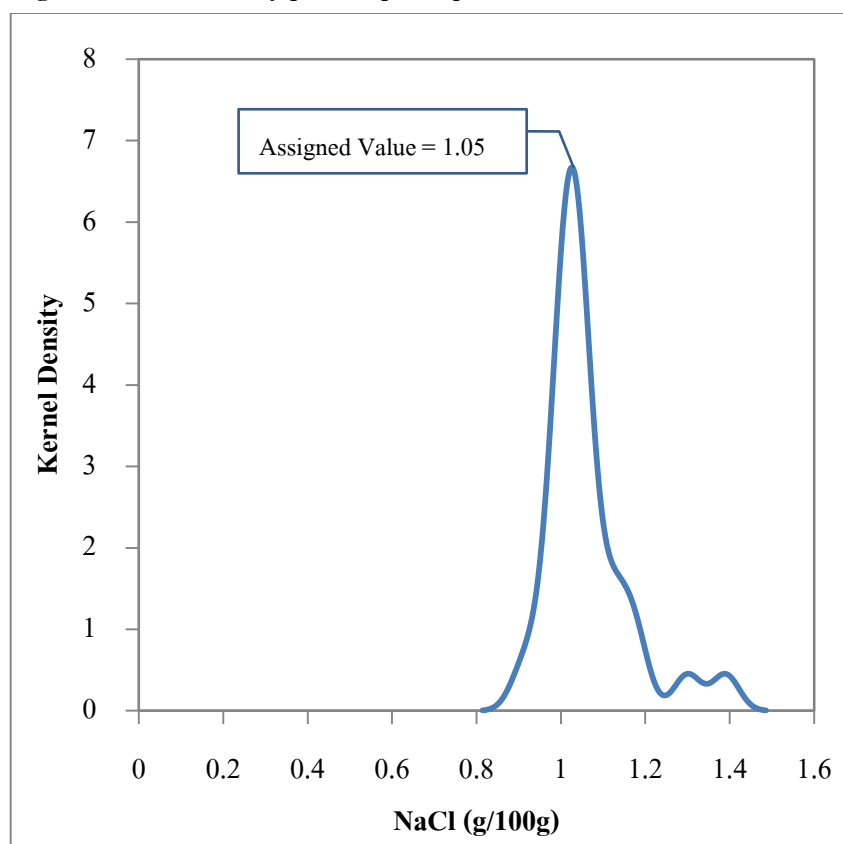
Table 1 Statistics summary

Statistic Data					Standard Deviation for Proficiency Assessment	
Number of Result	Assigned Value, x_{pt} (g/100g)	Robust Standard Deviation, s^*	Standard Uncertainty, $u(x_{pt})$	Coefficient of Variation, (%CV)	Derived from	σ_{pt}
28	1.05	0.068	0.016	6.5	4% Pooled RSD	0.042

Note: σ_{pt} derived from 4% Pooled RSD; $\sigma_{pt} = \frac{4 \times (x_{pt})}{100}$

Where x_{pt} is the assigned value

Figure 1 Kernel density plot for participant results



Note: The Kernel density show distribution of the 28 participant results.

Table 2 Result summary of participants' Z'-Score for NaCl (g/100g) in canned food
(baby corn in brine)

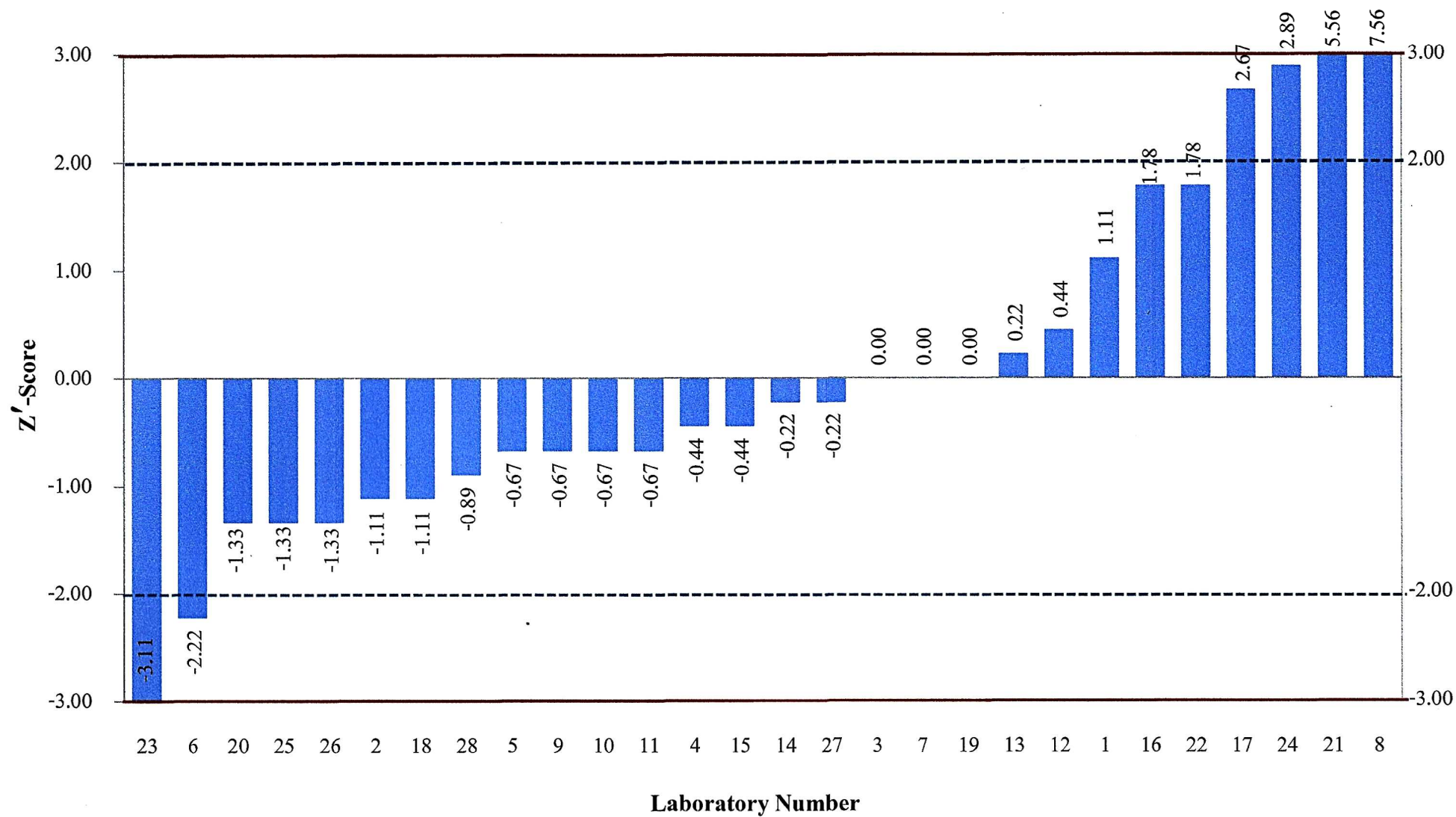
Laboratory Number	Result (g/100g)	Z'-Score
1	1.10	1.11
2	1.00	-1.11
3	1.05	0.00
4	1.03	-0.44
5	1.02	-0.67
6	0.95	-2.22
7	1.05	0.00
8	1.39	7.56
9	1.02	-0.67
10	1.02	-0.67
11	1.02	-0.67
12	1.07	0.44
13	1.06	0.22
14	1.04	-0.22
15	1.03	-0.44
16	1.13	1.78
17	1.17	2.67
18	1.00	-1.11
19	1.05	0.00

Note: Z'-Score values outside the satisfactory range are shown in bold.

Table 2 Result summary of participants' Z'-Score for NaCl (g/100g) in canned food
(baby corn in brine) (continued)

Laboratory Number	Result (g/100g)	Z'-Score
20	0.99	-1.33
21	1.30	5.56
22	1.13	1.78
23	0.91	-3.11
24	1.18	2.89
25	0.99	-1.33
26	0.99	-1.33
27	1.04	-0.22
28	1.01	-0.89

Note: Z'-Score values outside the satisfactory range are shown in bold.

Figure 2 Z' -Score values for NaCl (g/100g) in canned food (baby corn in brine)

7. METROLOGICAL TRACEABILITY

The provider requires participants to report their test results in g/100g. The metrological traceability of a measurement result in chemistry as made available from standard method and CRM.

The laboratory shall have a program for calibrating, verifying the performance of all critical equipment and certified reference standards, traceable to national standards.

8. TECHNICAL COMMENTS

Participants having questionable and unsatisfactory performance should find the cause of their error.

For the laboratories that used manual titration, they should examine their titration closely, especially the following points: titration reagent concentration, endpoint reading, and burette resolution selection.

And the laboratories that used auto titration technique for this program should check % recovery and use CRM for quality control. Electrode of auto titrator may not measure only Cl^- but also other interfering material that has an effect on more NaCl value than manual titration technique. Thus, laboratories that use auto titration technique have to make standard addition.

NFI is pleased to provide further detail, and welcome any discussion regarding the proficiency testing.

9. CONTACT DETAILS

Upon receiving the final report, if there is any question, complaint and appeal regarding the proficiency testing assessment incurs, written notification should be submitted to the project coordinator within thirty days by one of the following channels:

Mail: Miss Kuntida Duangsee (Project coordinator)

Division of Proficiency Testing Laboratory,

Department of Laboratory Services, National Food Institute,

2008 Soi Arun Ammarin 36, Arun Ammarin Rd., Bangyeekhan, Bangphlat, Bangkok

10700 THAILAND.

Fax number: 0 2422 8554

E-mail: pt@nfi.or.th

Website: Contact-Us Form at <http://pt.nfi.or.th>

10. APPENDIX

Table 3 Homogeneity and stability data

NaCl (g/ 100g)*					
Sample No.	Homogeneity (y_1)		Sample No.	Stability (y_2)	
	Replicate 1	Replicate 2		Replicate 1	Replicate 2
1	1.03	1.03	1	1.03	1.03
2	1.03	1.00	2	1.03	1.02
3	1.05	1.03	3	1.01	1.02
4	1.03	1.02	4	1.05	1.02
5	1.03	1.05	-	-	-
6	1.05	1.03	-	-	-
7	1.02	1.03	-	-	-
8	1.03	1.05	-	-	-
9	1.03	1.00	-	-	-
10	1.03	1.03	-	-	-
mean	1.03			1.03	
S_s	0.003			-	
$ \bar{y}_1 - \bar{y}_2 $	-			0.00	
σ_{pt}^{**}	0.041			0.041	
$0.3 \sigma_{pt}$	0.012			0.012	
$S_s \leq 0.3 \sigma_{pt}$	PASS			-	
$ \bar{y}_1 - \bar{y}_2 \leq 0.3 \sigma_{pt}$	-			PASS	

Note: * In-house method T 949 based on AOAC (2012), 937.09

$$^{**} \sigma_{pt} \text{ derived from 4\% Pooled RSD; } \sigma_{pt} = \frac{4 \times (\bar{y}_1)}{100}$$

Where \bar{y}_1 is the average of homogeneity

Table 4 Technical information in relation to the methods

Accredited	Laboratory Number
Yes	5 14 21
No	1 2 3 4 6 7 8 9 10 11 12 13 15 16 17 18 19 20 22 23 24 25 26 27 28
Reference	Laboratory Number
AOAC	1 2 3 6 9 10 12 13 14 17 21 22 24 25 26
Auto Titrator	4 5 7 11 15 16 18 19 20 23 27 28
Shoyu, Shikenho, Soy Sauce Experiment Method	8
Sample Weight (g)	Laboratory Number
0.01-1.00	1 5 6 12 15 16 22 23 24 25
0.25-0.35	2
0.5-5.0	19
1.01-2.00	4 11 13 14 20 27 28
2.01-3.00	8 18 21 26
3.01-4.00	7
4.01-5.00	3
>5.00	10 17
10	9
Titration Reagents	Laboratory Number
Ammonium Thiocyanate	9 10 21 26
Silver Nitrate	1 2 3 4 5 6 7 8 11 12 13 14 15 16 17 18 19 20 22 23 24 25 27 28

Table 4 Technical information in relation to the methods (continued)

Titration Technique	Laboratory Number
Auto Titration	2 4 5 7 11 14 15 16 18 19 20 23 27 28
Manual Titration	1 3 6 8 9 10 12 13 17 21 22 24 25 26
Quality Control : CRM	Laboratory Number
Yes	1 5 6 12 15 17 18 21 24 27
No	2 3 4 7 8 9 10 11 13 14 16 19 20 22 23 25 26 28
Quality Control: Standard Addition	Laboratory Number
Yes	2 4 5 7 11 15 18 19 23 27
No	1 3 6 8 9 10 12 13 14 16 17 20 21 22 24 25 26 28
Quality Control: Duplicate	Laboratory Number
Yes	1 2 5 6 7 8 10 11 13 14 15 17 18 19 20 21 22 23 24 25 26 27 28
No	3 4 9 12 16

COMMITTEE**Technical Consultants**

- | | |
|----------------------------------|--|
| 1. Mrs. Nitaya Pirapatrungsuriya | Executive Vice President, Food Industry Laboratory
Service Center |
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| 3. Mrs. Mayuree Leelawachiropas | Manager, Division of Chemical Laboratory |
| 4. Mr. Prakai Boriboon | Executive Advisor |
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Statistician Consultant

- | | |
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|-------------------------------|--|

Operators

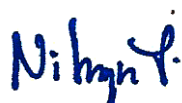
- | | |
|--------------------------|---|
| 1. Miss Tanaporn Borisut | Manager, Division of Proficiency Testing Laboratory
(Statistician) |
| 2. Miss Kuntida Duangsee | Senior Analyst, Division of Proficiency Testing Laboratory |
| 3. Miss Thatsanee Kansan | Analyst, Division of Proficiency Testing Laboratory |

Project Coordinator

- | | |
|--------------------------|--|
| 1. Miss Kuntida Duangsee | Senior Analyst, Division of Proficiency Testing Laboratory |
|--------------------------|--|

Proficiency Testing Provider Accreditation

ISO/IEC 17043: 2010: BLA-DSS Acc. No. PTP-0002

Approved by


(Mrs. Nitaya Pirapatrungsuriya)

Technical Manager

28 June 2018



สถาบันอาหาร
กระทรวงอุตสาหกรรม

ติดต่อสอบถามเพิ่มเติมได้ที่

แผนกทดสอบความชำนาญ ฝ่ายบริการห้องปฏิบัติการ

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