



STANDARDS
MALAYSIA

APLAC T064

TRACE METALS IN WATER

PROFICIENCY TESTING PROGRAMME

in

TRACE METALS IN WATER

on behalf of

ASIA PACIFIC LABORATORY ACCREDITATION COOPERATION (APLAC)

Submitted by

**Department of Standards Malaysia (STANDARDS MALAYSIA) in collaboration with
Department of Chemistry Malaysia**

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1. Introduction

Proficiency testing is vital in meeting the requirements of MS ISO/IEC 17025 with regards to ensuring quality of laboratory results. Proficiency testing involves a group of laboratories or analysts performing the analyses on the same samples and comparing results. The exercise is inspired to enhance the competency levels of participating laboratory and the analysts at large. As a member to APLAC, Department of Standards Malaysia is obligated to contribute for the members well being and mutual benefits. Hence this programme is targeted to fulfil one of APLAC's main objective, that is to build up and maintain mutual confidence in the technical competence of APLAC members.

This scheme is jointly organized by Department of Standards Malaysia and Department of Chemistry, Malaysia.

2. Objective of the Programme

The objective of this proficiency testing programme is to evaluate the competence of accredited laboratories among APLAC members in performing test on trace metals in water.

3. Criteria of Participants

This programme is opened for participation to all APLAC members as well as other interested bodies.

4. The number of expected participants in the programme

You are invited to nominate up to a maximum of **three** laboratories from your economy to participate in this programme.

5. Responsibilities

Department of Chemistry, Malaysia (DOC) is responsible for:

- Preparing and dispatching samples,
- Performing homogeneity and stability tests,
- Conducting statistical analysis of data and issuing interim and final reports.

Department of Standards Malaysia (STANDARDS MALAYSIA) is responsible for:

- Inviting participants and sending reports (interim and final) to participating Accreditation Bodies (ABs).
- Collecting test results from all participating laboratories via ABs,
- Act as liaison between ABs and respond on any enquiries, request, issues and concern on the programme.

Contact information of DOC and STANDARDS MALAYSIA are as follows:

Department of Chemistry Malaysia,
Jalan Sultan,
46661 Petaling Jaya,
Selangor, MALAYSIA.
(Attn. Ms. Nor Azian Abu Samah)
Fax No : +603 7985 3014
Tel No : +603 7985 3000
Email : azian@kimia.gov.my / proftest@kimia.gov.my

or

Department of Standards Malaysia (STANDARDS MALAYSIA)
Century Square, Level 1 & 2,
Blok 2300, Jalan Usahawan,
63000 Cyberjaya,
Selangor, MALAYSIA.
(Attn. Mr. Ahmad Razif Abd Rahim)
Fax No : +603-83189339
Tel No : +603-83191400
Email : razif@standardsmalaysia.gov.my

6. Sample

Sample Preparation

The scheme's sample consists of Sample A and Sample B. The same procedures are used to prepare both samples, with concentrations ranging from 0.1 to 2.0 ppm.

- i) Sample in 3% nitric acid (HNO_3) is prepared in a 100L capacity container, previously filled overnight with nitric acid. Various volumes of metal standards will be added. The sample is then mixed for several hours using motorised stirrer to ensure homogenous solution is obtained. 500ml of the final solution will be dispensed into each polyethylene bottles for dispatch to participating laboratories.
- ii) Ten bottles will be randomly selected from Sample A and Sample B for homogeneity testing. The analysis will be done in duplicates using ICP-MS.

Testing Methodology

- i) Proposed instrumentation techniques are by using ICP-MS, ICP-OES, AAS, etc.
- ii) Each participating laboratory will receive 2 bottles of sample labelled as Sample A and Sample B. The laboratories are required to determine the concentrations of various trace metals in the samples using their routine test methods.

7. Statistical Analysis

Evaluation of result will be made via values of z-score. The assigned value is defined as the consensus values from participants using robust statistics.

A simple robust z-score (denoted by Z) for a laboratory's sample X result would be:

$$Z = \frac{X - \text{median}}{\text{Normalised IQR.}}$$

The calculated z-scores will be tabulated in the report (alongside the corresponding results) in the table of results. The results are then evaluated based on their z-scores. An outlier is defined as any result with an absolute z-score of greater than three, i.e. $Z > 3$ or $Z < -3$. An outlier indicates that the result of a laboratory is beyond the consensus value (median).

8. Proposed Time Schedule

Invitation of participants

Invitation of participants will be arranged by STANDARDS MALAYSIA using **APLAC T064 Proficiency Testing Program Nomination Form**. Target implementation of activity is from June to August 2008. All forms shall be returned to STANDARDS MALAYSIA by end5 July 2008.

Preparation of sample

Preparation of samples will be conducted by DOC. Target implementation of activities are in June to July 2008. The sample shall consist of trace metals Al, Cr, Cd (total), Cu, Fe, Mn, Pb, Ni, and Zn at various concentrations.

Dispatch of sample

Dispatch of samples will be arranged by DOC. The samples will be dispatched to ABs tentatively in end of July 2008. Samples are appropriately packed and are accompanied by:

- instructions to ABs and participating laboratories
- receipt forms; and result sheets

ABs are responsible to acknowledge receipt of samples to STANDARDS MALAYSIA and distribute the samples to the participating laboratories. The participating laboratories shall acknowledge receipt of sample to the ABs and confirm that the samples are in good conditions upon arrival. Laboratories are provided approximately 4 weeks to complete the test.

Submission of result

All participating laboratories are to submit their results to their respective ABs and STANDARDS MALAYSIA. There will be two sets of result to be submitted that is, one set for Sample A and one set for Sample B. All results must be accompanied by estimation of measurement uncertainty. Tentative closing date for Participating Laboratories submitting results to their respective ABs is **31 August 2008**. Tentative closing date for ABs to submit the results to STANDARDS MALAYSIA is **7 September 2008**.

Statistical analysis of result

The result will be evaluated using statistical analysis and the outlier will be determined by the z-score. The results will also be represented in charts and histograms. The analysis will be done tentatively in September 2008 after all laboratories have submitted their results.

Interim report

Interim report will be distributed to all participants tentatively by end of September 2008. The report will consist of all participating laboratories result and their z-score. The laboratory codes shall be used as identification to ensure confidentiality is preserved.

Distribution of final report

Upon released of interim report, the final report shall be drafted and submitted to APLAC Proficiency Committee for review tentatively by end of October 2008. Upon granted with approval from the committee, the final report will be delivered to the ABs for distribution to the participating laboratories.

Event	Period	Action Party
Invitation of participants	June – July 2008	STANDARDS MALAYSIA
Preparation of sample	July 2008	DOC MALAYSIA
Dispatch of samples	July 2008	STANDARDS MALAYSIA/ DOC MALAYSIA
Submission of results	August 2008 – September 2008	STANDARDS MALAYSIA/ DOC MALAYSIA
Statistical analysis of results	September 2008	DOC MALAYSIA
Interim report	September 2008	DOC MALAYSIA
Distribution of final report	October 2008	STANDARDS MALAYSIA

Updated on: 21 May 2008

APPENDIX I

INSTRUCTIONS TO ACCREDITATION BODIES

1. Introduction

The APLAC T064 Proficiency Testing Program “Trace Metals in Water” is organized by Department of Standards Malaysia in collaboration with Department of Chemistry, Malaysia. The objective of this programme is to evaluate the competence of accredited laboratories in identification of trace metals in water.

2. Samples

Each laboratory is supplied with 2 samples each ~ 500ml and labelled as:

APLAC T064
Sample A or Sample B

Both samples are packed inside a box. Instructions and result sheets are included. There is no need to open the packaging. Upon receipt of the samples, kindly please complete the enclosed “RECEIPT FORM” and fax to the provided fax number below this form or email to razif@standardsmalaysia.gov.my.

3. Tests to be performed

Please refer to your copy of the “INSTRUCTIONS TO PARTICIPANTS” and “RESULTS SHEET” enclosed.

4. Sample Distribution

The mailing boxes are addressed to each participating laboratory and contain the samples of trace metals in water, instructions and results sheet (marked with their confidential lab code No.). These are to be sent to the laboratories within one week of receipt date. There is no need to open the packaging.

5. Documents to be submitted

- a) **No later than 31 August 2008** participating laboratories are requested to send the following to their accreditation body:
 - Complete results sheet
 - Any supporting documentation to assist in the interpretation of the results.
- b) **Also no later than 31 August 2008** participating laboratories are required to fax a copy of the results sheet to the address stated below.
- c) The accreditation body should collect, and email or faxed the documentation from participating laboratories **no later than 7 September 2008** to STANDARDS MALAYSIA.

The accreditation body is required to ensure that the above information has been supplied by their laboratories and should provide translation into English where necessary.

6. General Information

Additional information may be obtained from:

Department of Standards Malaysia (STANDARDS MALAYSIA)
Century Square, Level 1 & 2,
Blok 2300, Jalan Usahawan,
63000 Cyberjaya,
Selangor, MALAYSIA.
(Attn. Mr. Ahmad Razif Abd Rahim)
Fax No. : +603-83189339
Tel. No. : +603-83191400
E-mail : razif@standardsmalaysia.gov.my

APPENDIX II
RECEIPT FORM

For effective monitoring on the progress of this proficiency testing program, each **participating accreditation body is required** to fill in this RECEIPT form upon receipt of samples and fax or email it to:

Department of Standards Malaysia (STANDARDS MALAYSIA)
Century Square, Level 1 & 2,
Blok 2300, Jalan Usahawan,
63000 Cyberjaya,
Selangor, MALAYSIA.
(Attn. Mr. Ahmad Razif Abd Rahim)
Fax No. : +603-83189339
Tel. No. : +603-83191400
E-mail : razif@standardsmalaysia.gov.my

Thank you in advance for your cooperation.

The APLAC – T064 samples were received on: (date)

I hereby acknowledge the receipt of *sample(s) in (intact / damaged**) conditions and (suitable / not suitable**) for laboratory testing.

Comments :
.....
.....
.....

Participating Accreditation Body :

Contact Person :

Email Address :

Fax No. : Phone No. :

Note:

* Number of samples (per box).

** Choose whichever appropriate.

APPENDIX III

INSTRUCTIONS TO PARTICIPATING LABORATORIES

Introduction

The APLAC T064 Proficiency Testing Program “Trace Metals in Water” is organized by Department of Standards Malaysia in collaboration with Department of Chemistry, Malaysia. The objective of this programme is to evaluate the competence of accredited laboratories in trace metals in water.

Instructions

To ensure that results from this program can be analysed properly, participants are asked to adhere carefully to the following instructions.

1. Two samples each ~ 500ml and labelled APLAC T064 Sample A and APLAC T064 Sample B have been supplied to each laboratory by your respective ABs. Please check your samples condition upon receipt. Please contact with your accreditation body if there is damaged samples.
2. Testing may commence as soon as samples are received. Store your samples in the original packaging in refrigerator at about 4°C until testing commences.
3. The samples are prepared synthetically thus acid digestion is not required prior to testing.
4. Participating laboratories are required to test the samples and determine the concentration (in mg/l) of the following trace metals:
 - Aluminium
 - Cadmium
 - Chromium (total)
 - Copper
 - Iron
 - Manganese
 - Lead
 - Nickel
 - Zinc

Test results are to be reported in the “RESULTS SHEET” and result figures reported are to be round up to 2 decimal places. Analysts should be aware of analyte stability and perform the tests in an appropriate order.

5. Participants should use routine test methods, which would normally used to test customer supplied samples and record the method using the provided results sheet. Please report the Measurement Uncertainty for each result.

6. The information on the identities and results of the participating laboratories will be kept confidential to the accreditation bodies and the organizers. Each participating laboratory will be assigned with a confidential code number.
7. Send the completed results sheet and any supporting documentation to your accreditation body **and** fax a copy of the results sheet no later than **31 August 2008** to:

Department of Standards Malaysia (STANDARDS MALAYSIA)

Century Square, Level 1 & 2,

Blok 2300, Jalan Usahawan,

63000 Cyberjaya,

Selangor, MALAYSIA.

(Attn. Mr. Ahmad Razif Abd Rahim)

Fax No. : +603-83189339

Tel No. : +603-83191400

E-mail : razif@standardsmalaysia.gov.my

Not later than: 31 August 2008

**APPENDIX IV
Nomination Form**

If your laboratories wish to participate in the APLAC Proficiency Testing Programme T064 – Trace Metals in Water please complete this Nomination Form and send to the contact below **no later than 5 July 2008.**

Contact : Mr. Ahmad Razif Abd Rahim
 Department of Standards Malaysia
 E-mail : razif@standardsmalaysia.gov.my
 Fax : +603-8318 9339

Name of contact person:
Organisation:
Postal Address:
Phone:
Fax:
Email:
Accredited? Yes / No

Name of contact person:
Organisation:
Postal Address:
Phone:
Fax:
Email:
Accredited? Yes / No

Name of contact person:
Organisation:
Postal Address:
Phone:
Fax:
Email:
Accredited? Yes / No

From :
 (NATIONAL LABORATORY ACCREDITATION BODY)

Name : _____
 Designation : _____
 Organisation : _____
 Physical Address : _____
 (not a P.O. Box)
 Phone No. : _____ Fax No. : _____
 Email Address : _____

**APPENDIX V
RESULTS SHEET**

Laboratory Code :

Result in mg/l round up to 2 decimal places

Metals	Sample A	Sample B	Uncertainty	Instrumentation Technique
	Result (mg/l)	Result (mg/l)		
Aluminium				
Cadmium				
Chromium (total)				
Copper				
Iron				
Manganese				
Lead				
Nickel				
Zinc				

Signature : Date:

Contact person :

Name of participating lab :

Name of accreditation body :

Email address :

Fax No. : Phone No. :