Instructions for Participants

1. Analysis of the proficiency test sample

(1) Sample information

- Participating laboratories will be provided with **ONE** bottle containing about **20 g** of wheat flour. The analyte contents in each bottle should be considered equivalent after dry-mass correction within the level of the between-sample homogeneity. The date of dispatching of samples will be informed to participants.
- **Participants are required to confirm the receipt of the samples (including the serial number of the sample, date of receipt, any damages found in the sample package) by e-mail (aplacpt.inorg@kriss.re.kr) using the sample receipt form attached in Annex A. If any damage on the sample is observed on arrival, please contact us immediately.**

- Analytes and their approximate mass fractions are given as follows.

Element	Mass fraction (expected range of values)
Cadmium (Cd)	0.05 mg/kg -1 mg/kg
Lead (Pb)	0.05 mg/kg -1 mg/kg

- Homogeneity: ID-ICP/MS analysis of one subsample taken from each of 11 or 12 bottles was carried out. The relative standard deviations of the results obtained from 12 and 11 bottles were 0.29 % and 0.23 % for Cd and Pb, respectively, which are satisfactory for the present proficiency testing.

(2) Sample storage

- The proficiency test sample should be kept sealed in its original bottle and stored under room temperature conditions.
- Opening of the sample bottle should be carefully planned to avoid contamination and deterioration of the sample.
- For safety considerations, the proficiency test sample should be handled with care to prevent inhaling the sample powder and getting into eyes. In the case of accidental exposure to sample, wash the exposed areas with plenty of water and consult physicians when necessary.
- For this proficiency testing programme, it is not required to return remaining sample.

(3) Guidelines for sample preparation and dry-mass correction

- Sample preparation for analysis:
  - Sample handling in a laboratory with well-controlled humidity and temperature is recommended. Any kind of contamination should be avoided.
  - Sampling for the analysis should be carried out at the same time as sampling for the dry-mass correction.
  - Digest the wheat flour material completely, if the method used requires digestion.
  - The proficiency test sample should be mixed thoroughly before taking an aliquot of sample from a bottle.
  - The analysis should be conducted with a recommended sample size of at least 0.5 g.
  - Participants are recommended to perform at least triplicate measurements and report the mean and associated measurement uncertainty for each analyte as specified in the Result Report. Participants should estimate the measurement uncertainty using their own practice.
- Dry-mass correction:
  - Participants should also carry out the dry mass correction.
  - Sampling for the dry mass correction should be carried out at the same time as the sampling for analysis.
  - At least three separate portions from a sample bottle (with a recommended sample size of about 0.5 g for each portion) should be taken as dry mass correction samples and placed over P_2O_5 in a desiccator at room temperature for 7 days (168 hours).
  - Calculate the moisture content from the mass change observed in the three aliquots and use it for dry-mass correction.

(4) Measurement method

- Participants are expected to use the test method of their choice, which should be consistent with their routine procedures. If the laboratory is accredited, it is recommended to use the method listed in the scope of its accreditation.

2. Reporting and submission of results

Participants should complete the Result Report (Annex B). The manners of reporting test results are as follows:

- For each analyte, the mean value of at least three independent measurements and its associated expanded uncertainty with 95 % level of confidence should be reported on a dry mass basis;
- Report the mass fractions of analytes in mg/kg for cadmium and lead; and
- Participants should provide information about the method of analysis.

Participants should be aware that any submitted results are considered final and accordingly such results and units should be thoroughly checked before submission. Participants should submit the Result Report electronically to the coordinator of the proficiency testing programme (E-mail: aplacpt.inorg@kriss.re.kr) before the deadline, **4 September 2015**. Results submitted after the deadline will not be accepted. Participants are reminded that the ability to report results in the specified unit and within the given time scale are part of the proficiency test.

The proficiency testing programme is conducted in the belief that participants will perform the analysis and report results with scientific rigour. Collusion and falsification of results are clearly against the spirit of the proficiency testing programme.

3. Contact

Participants may wish to contact the coordinator of the proficiency testing programme for any enquires (E-mail: aplacpt.inorg@kriss.re.kr):

Dr. Sook Heun Kim
Senior Research Scientist
Center for Inorganic Analysis
Division of Metrology for Quality of Life
Korea Research Institute of Standards and Science (KRISS)
267 Gajeong-Ro, Yuseong-Gu, Daejeon 305-340, Republic of Korea
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Fax: +82-42-868-5801

Annex A

Sample Receipt Form
(Participating Laboratories)

Institute/
Laboratory: _____

Postal address: _____

Contact person: _____ _____ _____
Title Given name Surname

E-mail: _____

Date of Receipt: _____

Confirmation of Package Content

Please choose the state of the sample: □ intact
□ broken
□ others: _____

Please write the serial numbers of the sample bottle:_____

Please complete this form and return it to Dr. Sook Heun Kim, KRISS (E-mail: aplacpt.inorg@kriss.re.kr) by email immediately after receipt of the sample.

Annex B

Result Report

Institute/ Laboratory: _____

Postal address: _____

Contact person: _____ _____ _____

Title Given name Surname

E-mail: _____

Laboratory accreditation: YES (based on ISO 17025/ ISO 9000 series/ GLP/ under the law of your economy (please specify)) /NO

If yes, please specify the name of accreditation body.

1. Analytical results

Analyte	Analytical result (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor *k* (95% level of confidence)	Expanded uncertainty (mg/kg)

Notes: (i) Report the analytical results and associated uncertainties in the unit mg/kg; (ii) Report the analytical results on a dry mass basis.

1) Results from individual aliquot (dry-mass corrected)

number	Cd (mg/kg)	Pb (mg/kg)
1		
2		
3		
4		
Average		
Standard deviation		
Standard deviation of the mean		

Please insert more lines for more determinations.

2) Dry-mass Correction

Moisture content (% in mass):_____

2. Methods of analysis

1) Analyte: Cadmium

a) Sample size used for analysis : _____ g

b) Digestion technique: Microwave-assisted digestion / Wet digestion / Dry ashing / Others (please specify):

c) Digestion medium: HNO_3 / HCl / HF / H_2SO_4 / $HClO_4$ / H_2O_2 / Aqua regia / Others (please specify):

d) Matrix separation: YES / NO

e) Calibration method: External calibration / Internal calibration / Standard additions / Isotope dilution mass spectrometry

f) Source(s) of calibration standard(s):

g) Use of internal standard(s): YES (please specify): / NO

h) Analytical instrument(s): ICP-MS / ICP-AES / Flame AAS / Graphite AAS
Others (please specify):

i) Correction for recovery: YES (please specify recovery (%)): / NO

j) Method validation: YES(please specify): / NO

k) Additional information:

2) Analyte: Lead

a) Sample size used for analysis: g

b) Digestion technique: Microwave-assisted digestion / Wet digestion / Dry ashing
Others (please specify):

c) Digestion medium: HNO_3 / HCl / HF / H_2SO_4 / $HClO_4$ / H_2O_2 / Aqua regia
Others (please specify):

d) Matrix separation: YES / NO

e) Calibration method: External calibration / Internal calibration / Standard additions / Isotope dilution mass spectrometry

f) Source(s) of calibration standard(s):

g) Use of internal standard(s): YES (please specify): / NO

h) Analytical instrument(s): ICP-MS / ICP-AES / Flame AAS / Graphite AAS
Others (please specify): _____

i) Correction for recovery: YES (please specify recovery (%)): _____ / NO

j) Method validation: YES(please specify): _____ / NO

k) Additional information: _____

Print name of responsible person:

Date: ____ / ____ /_____.
dd mm yyyy