Proficiency Testing Programme (APLAC PT065)

Determination of Cadmium and Lead in Herbal Sample

1. Organizations

The programme is organized by the Government Laboratory of Hong Kong (HKGL), in collaboration with Hong Kong Accreditation Service (HKAS) under the auspices of the Asia-Pacific Laboratory Accreditation Cooperation (APLAC).

2. Objective of the programme

Heavy metals are contaminants that occur as residues in herbal materials from the environment/industrial activities. Excess intake of these elements could induce adverse effect and hence their maximum residual in foodstuffs have been regulated by respective national health authorities. As such, heavy metal residues testing in herbal materials and their products is commonly monitored and is carried out extensively by laboratories worldwide. The main purpose of this proficiency testing programme is to evaluate the performance of laboratories for analysing cadmium and lead in herbal sample (*Herba Desmodii Styracifolii*) through inter-laboratory comparison.

3. Selection of participants

APLAC members as well as non-APLAC members will be invited for the programme. Invitations will be sent to all APLAC members and other accreditation bodies in May to June 2008.

4. Number and identity of prospective participants

Participating accreditation bodies will be asked to nominate laboratories to participate and indicate whether the nominated laboratories are accredited or not. Preference should be given to laboratories that are accredited for this test. A restriction on the number of participating laboratories from each accreditation body may need to be imposed. The number of participating laboratories shall be limited to not more than 100.

5. Application fee

Free of charge.

6. Preparation of PT sample

Sample selection and preliminary processing:

About 1 kg of herbal sample (*Herba Desmodii Styracifolii*) were purchased from local market. The herbal samples were rinsed with distilled water to remove dirt and foreign matters, and then freeze dried. The dried samples were grinded to powder and passed through 100 μm sieves. The fine powder (≤ 100 μm) was collected and thoroughly mixed in a commercial mixer. Aliquots of about 5 g of the homogenized powder were packed into clean and air-tight amber bottles, and then disinfected by γ-irradiation at a dose of about 1 kGy to prevent microbial growth. A total of about 150 bottled samples were prepared.

Homogeneity testing and stability monitoring:

Not less than ten samples will be taken randomly from the prepared powder sample and analyzed using validated methodology in duplicate for homogeneity tests by HKGL. Regularly, random samples will be taken and analyzed in triplicate for the stability testing at

room temperature and elevated temperature (eg. 37 ºC). The period of stability test should be long enough in order to ensure the measurands in the samples are stable over a reasonable timeframe.

The preparative procedures and statistical analysis are in accordance with the recommendations stipulated in ISO/IEC G43-1:1997, ILAC G13:2007, ISO13528:2005 and APLAC PT002 Testing Inter-laboratory Comparisons.

Testing methodology:

Each participating laboratory will be given one sample containing about 5 g of dried herbal powder. The laboratories will be required to determine the quantity of cadmium and lead in the samples according to their accredited methods, validated methods and/or in-house methods. Analysis should be conducted in triplicate; and test results, associated measurement uncertainty and other details should be reported in the result sheets provided and returned electronically to HKGL (email: ycwong@govtlab.gov.hk) on or before the deadline.

Dispatch of samples and instructions to accreditation bodies and participants:

Test samples together with sample receipt form, instructions to accreditation bodies and participating laboratories will be dispatched to AB around August 2008. Upon receipt of the sample pack, AB should check the samples for damage during transportation, complete and return the receipt form to the organizer. The samples, instructions and results sheets should be distributed to participating laboratories as soon as possible. Participating laboratories might have about eight weeks (depending on the duration of respective custom clearance) to analyze the samples and are required to report the test results as well as their measurement uncertainties before the scheduled deadline.

7. Proposed schedule of the programme

Event	Period	Responsible by
Preparation of sample	Aug to Sept 2006	HKGL
Homogeneity testing	Oct 2006	HKGL
Submission of proposal to APLAC PT Committee for approval	May 2008	HKGL/HKAS
Invitation of participants	May - June 2008	HKAS
Dispatch of samples	July 2008	HKGL
Stability testing	Nov 2006 – Sept 2008	HKGL
Deadline for result submission	31 August 2008	HKGL
Statistical analysis of pooled data	Sept 2008	HKGL
Interim report	Sept 2008	HKGL
Preparation of draft report	Oct 2008	HKGL
Submission of draft report to APLAC PT Committee for approval	Oct 2008	HKGL/HKAS
Distribution of final report	Nov 2008	HKGL

8. Performance assessment

Assigned values of the programme are determined by the HKGL using isotope dilution inductively coupled plasma-mass spectrometry (ID-ICP-MS) technique; and the standard deviations will be calculated based on Horwitz Equation. Each participating laboratory is recommended to report three sets of test data and the respective mean value. The performance of each laboratory is assessed by the z-score which will be presented in tabular form and histograms.

The z-score is commonly interpreted as follows:

(a)	$\|z\| \leq 2$	Satisfactory
(b)	$2 < \|z\| < 3$	Questionable
(c)	$\|z\| \geq 3$	Unsatisfactory

Laboratories having a |z| score larger than 3 shall investigate their results.
Laboratories having a z-score in the range $2 < |z| < 3$ are encouraged to review their results.

9. Issuance of reports

The HKGL shall prepare (i) an interim report to the participating laboratories and/or AB for comments, (ii) a draft final report to the APLAC PT Committee for review. Upon approval of the draft report, the electronic copy of the final report will be forwarded to participating accreditation bodies for distribution to their respective participating laboratories.

10. Confidentiality

Each laboratory will be assigned with a unique identification code. This unique code will be used throughout the programme. All information on the identities and results of participating laboratories will be kept confidential and not be disclosed. If participating laboratories submit the results through their accreditation authorities, their results may be disclosed to and released through their accreditation authorities.

11. Further Enquiries

Please contact:
Proficiency Testing Advisory Board (PTAB),
Hong Kong Government Laboratory
Email: ycwong@govtlab.gov.hk

PTAB, HKGL

21st May 2008