Proficiency Testing Programme (APLAC T068)

Determination of Polycyclic Aromatic Hydrocarbons in Sediment

1. Organizations

The programme will be 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. Introduction

Polycyclic aromatic hydrocarbons (PAH) are ubiquitous organic pollutants that are known to exhibit carcinogenic and mutagenic properties. These compounds are produced anthropogenically under high temperature combustion of fossil fuels in automobile engines, cooking stoves, power plants, refineries and various industrial activities. Phenanthrene (PHE), fluoranthene (FLT), benzo(a)anthrancene (BAA), benzo(a)pyrene (BAP) and benzo(ghi)perylene (BGP) are chosen as representative PAHs, in terms of their high mass emission or high toxicity equivalence, for the present programme. The HKGL had participated in the Consultative Committee for Amount of Substance (CCQM) inter-laboratory comparisons for the same analytes in organic solution (CCQM-P31a) in 2004 and in soil/sediment (CCQM-P69 and CCQM-K50) in 2005 and 2007 respectively. Analytes in the three programmes were determined using gravimetric isotopic dilution gas chromatography mass spectrophotmetry (ID-GCMS), and the results obtained were comparable with those of the national metrology institutes. The main purpose of this proficiency testing programme is to critically evaluate the performance of laboratories for analysing the above five PAHs in sediment based on the reference values provided by the HKGL (using gravimetric ID-GCMS).

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 October to November 2008.

4. Number and identity of prospective participants

Participating accreditation bodies (AB) 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

(i) Sample selection and preliminary processing:

About 20 kg of sediment samples were collected from the local contaminated coast. The sample material was air dried and immersed with acetone for removal of microbes. The dried samples were grinded and passed through 250 μm sieves, and the fine powder was collected and thoroughly mixed in a commercial rotating drum. Aliquots of about 15 g of the homogenized powder were packed into clean and air-tight amber bottles with PTFE-inlayed screw caps. A total of about 400 bottled samples was prepared.

(ii) Homogeneity testing and stability monitoring:

Not less than ten samples were taken randomly from the sample log and analyzed using validated methodology in duplicate for homogeneity tests by the HKGL. Random samples will be taken and analyzed in triplicate for stability testing at room temperature (~25 ºC) and at elevated temperature (37 ºC).

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

(iii) Approximate concentration range of PAHs:

Analyte	Approximate Concentration Range (mg/kg)
PHE	1 – 15
FLT	1 – 15
BAA	1 – 15
BAP	1 – 15
BGP	1 – 15

(iv) Testing methodology:

Each participating laboratory will be given one sample containing about 15 g of dried sediment powder. The laboratories will be required to determine the quantity of PHE, FLT, BAA, BAP and BGP in the samples according to their accredited methods, validated methods and/or in-house methods. Analysis should be conducted with a minimum of triplicate; and test results, associated measurement uncertainty and other technical information should be reported in the Result Proforma provided and returned electronically to the HKGL on or before the set deadline.

(v) Dispatch of samples and instructions to accreditation bodies and participants:

Test samples together with sample receipt form, instructions to accreditation bodies and participating laboratories, result proforma and other necessary documents will be dispatched to participating AB around December 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, result proforma, etc. should be distributed to participating laboratories as soon as practicable. Participating laboratories would have about eight weeks (depending on the duration of their 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	Apr to Jul 2008	HKGL
Homogeneity testing	Aug 2008	HKGL
Submission of proposal to APLAC PT Committee for approval	Sept 2008	HKGL/HKAS
Invitation of participants	Oct – Nov 2008	HKAS
Dispatch of samples	Nov/Dec 2008	HKGL
Stability testing	Sept 2008 – Mar 2009	HKGL
Deadline for result submission	31 Jan 2009	HKGL
Statistical analysis of pooled data	Feb 2009	HKGL
Interim report	Feb – Mar 2009	HKGL
Preparation of draft report	Mar 2009	HKGL
Submission of draft report to APLAC PT Committee for approval	Mar 2009	HKGL/HKAS
Distribution of final report	Apr 2009	HKGL

8. Performance assessment

Assigned values of the programme are determined by the HKGL using ID-GCMS 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

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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:
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27th Aug 2008