APLAC Proficiency Testing Program (APLAC T071)

Melamine in Milk

1. Organizers

Hong Kong Accreditation Service (HKAS)
Government Laboratory (HKGL)

2. Nature and Objective of the Programme

Melamine is commonly used to produce melamine resin, a durable thermosetting plastic by polymerisation with formaldehyde. With the high molar nitrogen ratio, an unethical addition of the compound to food products could mislead to a high protein measurement result using conventional methods. The incidents on melamine tainted pet food, fish feed, milk powder, diary products and food have been reported worldwide.

Considering a limited number of proficiency tests (PT) and the needs of laboratories for seeking accreditation on testing melamine in food and food products, a PT programme on melamine in milk is proposed. The programme will be organised by the HKGL with HKAS as the collaborator under the auspices of APLAC. The main objectives of this proposal are to provide an analytical forum for participants in the quantitative analysis of melamine in milk sample with the provision of reference values and to evaluate the measurement capability of participating laboratories that providing such testing services.

3. Responsibilities

3.1. The HKGL is responsible for preparing, packaging and dispatching samples, performing homogeneity and stability tests, collecting test results from participating laboratories, conducting statistical analysis of data, handling participants' queries and issuing interim and final reports. Contact information of the HKGL:

Dr. Y. C. Wong
Homantin Government Offices,
88 Chung Hau Street, Homantin, Hong Kong
Fax: +852 2194 1147
Tel.: +852 2762 4042
Email: ycwwong@govtlab.gov.hk

3.2. HKAS is responsible for inviting participants and acting as a contact point between participants and the HKGL. Contact information of HKAS:

Mr. W. W. Wong
36/F, Immigration Tower,
7 Gloucester Road, Wanchai, Hong Kong
Fax: +852 2824 1302
Tel.: +852 2829 4813
Email: wwwong@itc.gov.hk

4. Selection of Participants

APLAC members as well as other non-APLAC members will be invited to participate in the programme. Invitations will be sent to all APLAC members and other accreditation

bodies. Participating accreditation bodies will be asked to nominate laboratories to participate and indicate the accreditation status of the nominated laboratories for the test. The number of laboratories shall be preferably **limited to 100**. Therefore, a restriction on the number of participating laboratories from each accreditation body may need to be imposed.

5. Preparation of Sample

About 25 litres of UHT skimmed milk were purchased from the local market and were confirmed to be free of melamine. The milk sample was divided into two portions at approximately 12.5 L and gravimetrically spiked with different quantities of melamine standard solution. The spiked samples were gently stirred for 10 minutes and subject to freeze drying. The dried samples were ground to fine powder with the aid of high speed blenders, and then passed through 200 μm sieves. The sieved powder was placed in a 3-dimensional rotating drum for thorough mixing. The homogenized powder, in about 5 g portion each, was independently dispensed into clean nitrogen-flush amber bottles. The prepared samples (with two different melamine levels at about 0.1 to 5 mg kg^{-1} range) were disinfected by γ-irradiation at a dose of about 10 kGy and stored in dry boxes at room temperature.

Each participating laboratory will be provided with TWO independent sample bottles each containing about 5 g of dried milk powder. They will be given instruction to reconstitute the milk powder into liquid milk and are required to determine the mass fraction (in mg kg^{-1}) of melamine in the liquid milk samples. Participating laboratories should used their usual methods (either accredited, validated, in-house, etc) for the determination of melamine and the analysis is recommended to be carried out with a recommended sample size (liquid milk) of about 1 g. Test results and other technical details should be reported in the result sheets provided. If the determination has been carried out in duplicate or triplicate, laboratories can, if they wish, report all the results obtained. In such case, the mean result will be used for performance assessment.

6. Homogeneity and Stability Testing

Ten samples were taken randomly from the prepared bottles of samples and analyzed in duplicate for determining sample inhomogeneity in accordance with the recommendation stipulated in APLAC PT002 Testing Interlaboratory Comparisons. Throughout the course of the PT, a random sample will be regularly taken and analyzed in triplicate at room temperatures (about 25 ºC) for monitoring the stability of the level of analytes until the results from all participant laboratories are received.

7. Statistical Analysis

Performance of the participating laboratories is assessed using z-score which is calculated as:

$$z = \frac{x_i - x}{sd}$$

where x_i = reported mean of individual participant
x = reference values from gravimetric spike
sd = standard deviation estimated from the Horwitz Equation

Performance of participating laboratories is interpreted as follows:

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

Laboratories having a |z| score equal to or larger than 3 shall thoroughly investigate their results for the discrepancy and those having a z-score in the range $2 < |z| < 3$ are also encouraged to review their results.

8. Issuance of Reports

Upon completion of data analysis, the HKGL will issue an interim report to participating accreditation bodies via HKAS for their comment. A draft final report will then be prepared and submitted to APLAC PT Committee for review and approval. Upon approval by the APLAC PT Committee, an electronic copy of the Final Report will be distributed to the participants.

9. Programme Schedule

Event	Period	Responsible by
Preparation of sample	Feb – Apr 2009	HKGL
Homogeneity testing	Apr 2009	HKGL
Submission of proposal to APLAC PT Committee for approval	May 2009	HKAS
Stability testing	Jun – Oct 2009	HKGL
Invitation of participants	June – Jul 2009	HKAS
Dispatch of samples	Jul 2009	HKGL
Submission of results	Sept 2009	HKGL
Statistical analysis of results	Oct 2009	HKGL
Interim report	Oct 2009	HKGL
Submission of draft report to APLAC PT Committee	Nov 2009	HKGL / HKAS
Approval of draft report by APLAC Proficiency Committee	Nov – Dec 2009	HKGL / HKAS
Distribution of final report	Nov – Dec 2009	HKGL

10. Confidentiality

Information on the identities and results of the participating laboratories will be kept confidential to the respective nominating accreditation bodies and the organizers (APLAC, HKAS and HKGL). Each laboratory will be assigned with a unique identification code. This unique code will be used in the report. If laboratories submit their results through their accreditation bodies, their results may be disclosed to and released through their accreditation bodies.

Date: 1 June 2009