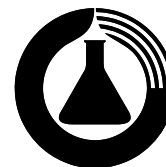




APLAC T069 Proficiency Testing Programme



“Melamine in Fish Feed”

1. Coordinators

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

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. Recently, incidents on melamine tainted pet food, fish feed, milk powder and diary products have been reported.

Considering the limited proficiency programme (PT) and the needs of laboratories for seeking accreditation on testing melamine in food and food products, a PT programme on melamine in fish 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 fish feed sample 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 as soon as this proposal is approved by the APLAC PT Committee. 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 is **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 10 kg of fish feed were purchased from the local market and were confirmed to contain certain quantity of melamine. The sample was oven dried, powdered, sieved (through 250 µm sieve) and homogenized. The fine fish feed powder, in about 15 g portion each, was independently dispensed into clean amber bottles. The prepared samples (about 300 bottles) were disinfected by γ-irradiation at a dose of about 1 kGy for preventing microbial growth. Approximately melamine in the fish feed was found to be in the range of 1 to 15 mg/kg.

Participating laboratories will be provided with one bottle containing about 15 g of sample and are required to determine the mass fraction (in mg/kg) of melamine in the sample as received. Participating laboratories should use their preferred methods (accredited, validated, in-house, etc) for the determination of melamine and the analysis is recommended to be carried out with a recommended sample size of 0.5 g to 1 g and should be conducted in triplicate. Test results and other technical details should be reported in the result sheets provided.

6. Homogeneity and Stability Testing

Ten samples were taken randomly from the prepared bottles and analyzed in duplicate for determining sample inhomogeneity in accordance with the recommendation stipulated in APLAC PT002 Testing Interlaboratory Comparisons and guidelines in QM/MWI/PT1 of the HKGL. Regularly, a random sample will be taken and analyzed in triplicate at two different temperatures (ca. room temperature of about 25 °C and elevated temperature of about 37 °C) for monitoring the stability of the level of analytes until the results from all participant laboratories are received.

7. Statistical Analysis

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

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

where x_i = reported mean of individual participant
 \bar{x} = consensus mean
sd = standard deviation estimated from Horwitz Equation

Consensus mean (\bar{x}) is determined using pooled data (arithmetic mean of three independent analytical data from each participating laboratories by robust statistic and

the standard deviation (sd) is estimated using 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 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 laboratories and/or accreditation bodies via HKAS for comment. After that, a draft final report will be prepared and be submitted to APLAC PT Committee for approval. With the official endorsement from APLAC PT Committee, an electronic copy of the Final Report will be distributed to the participating laboratories.

9. Proposed Time Schedule

Event	Period	Responsible by
Preparation of sample	Dec 2008 – Jan 2009	HKGL
Homogeneity testing	Feb 2009	HKGL
Submission of proposal to APLAC PT Committee for approval	Feb 2009	HKAS
Stability testing	Mar – Jul 2009	HKGL
Invitation of participants	Mar – Apr 2009	HKAS
Dispatch of samples	May 2009	HKGL
Submission of results	Jun 2009	HKGL
Statistical analysis of results	Jul 2009	HKGL
Interim report	Jul 2009	HKGL
Submission of draft final report to APLAC PT Committee	Aug 2009	HKGL / HKAS
Approval of draft final report by APLAC Proficiency Committee	Sept 2009	HKGL / HKAS
Distribution of final report	Sept 2009	HKGL

10. Confidentiality

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