

APLAC T083 Preservatives in Soy Sauce

Proficiency Testing Program

1. **Objective**

The objective of this proficiency testing program is to evaluate the competence of chemical laboratories for analyzing Preservatives in Soy sauce.

2. Organization and Coordination

This proficiency testing programme is organised by the Taiwan Accreditation Foundation (TAF) under the auspices of Asia Pacific Laboratory Accreditation Cooperation (APLAC). TAF is responsible for coordinating activities relevant to this program such as inviting participants, assigning confidential codes to participants, preparing instructions, dispatching test samples, collecting results, analysing results and preparing final report as well as the interim reports.

3. Test material

Preparation of test material was performed in accordance with ISO/IEC 17043:2010 [1]. In brief, about 20 liters soy sauce was purchased from a supermarket. Four kinds of preservatives (benzoic acid, sorbic acid, methyl p-hydroxy benzoate and butyl p-hydroxy benzoate) were added into samples in two differences in concentrations. To allow suitable and reliable proficiency testing, homogeneity and stability test will be conducted on randomly selected samples before test samples are distributed to participants. Approximate ranges of the four preservatives are as follows:

Preservatives	Approximate Conc. Range (mg/kg)
Sorbic acid	125 to 1500
Benzoic acid	125 to 1500
Methyl p-hydroxy benzoate	125 to 1500
Butyl p-hydroxy benzoate	125 to 1500

4. Homogeneity and Stability test

Not less than 10 samples were taken randomly from the final package of samples and

analyzed in duplicate for determining the sample inhomogeneity in accordance with the recommendation stipulated in ISO 13528:2005 [2]. A random sample will be analyzed in triplicate at room temperature (about 25 $^{\circ}$ C) for monitoring the stability of analytes between sample dispatch and after submission of results.

5. Test Item and Test Method

Test item: Soy sauce

Test to be conducted: Benzoic acid, Sorbic acid, Methyl p-hydroxy benzoate and Butyl p-hydroxy benzoate Test method: Ex: AOAC 983.16 (Benzoic acid, and Sorbic acid in Food); GC method(FID); LC method(UV or PDA) ; Daily-used method

6. Selection of Participants

APLAC and non-APLAC members will be invited to participate in this program; however, APLAC member has priority for participating in this program. Each APLAC member's accreditation body (AB) may nominate up to a maximum of 3 laboratories, other non-APLAC members can nominate only one laboratory. The nomination form shall be returned to TAF before the due date as stated in the invitation.

7. Result Collection and Result Analysis

Standard Result Sheets will be provided to the participating laboratories for result collection. All participating laboratories should return the completed result sheets to TAF before the due date as prescribed in the instructions. If any result is not received by the due date then TAF will contact participating laboratories and carbon copier (cc) the AB of the corresponding economy to follow up. Late return of result sheets without justified reasons may be regarded as disqualified and the corresponding data will not be included in the analysis.

Once all results are received, the data will be entered and checked by TAF. An interim report with all the received data tabulated in a well-formatted table and the consensus values of each test will be sent out to all participating AB, then kindly request the participating AB delivers the interim report to the participating laboratories. Any clarification or correction of results should be made within 2 weeks after receipt of the interim report.

After 2 weeks and resolution of any suggested changes, a draft report will be written up. The z-score approach using ISO 13528 will be used for statistical analysis of the test results. The draft report will be dispatched to APLAC Proficiency Testing Committee who will review it and forward their comments back to TAF.

The final report incorporated with the received comments will then be published. A copy of the final report will be distributed to each participating AB plus sufficient copies for each of the participating laboratories.

Performance Assessment:

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

 $z = \frac{(x_i - \overline{x})}{\sigma}$

xi= reported result of individual participant

 $\overline{\mathbf{X}} =$ robust average

 σ = standard deviation estimated from Horwitz equation

z-Score is commonly interpreted as:

(i)	z ≤ 2	Satisfactory
(ii)	2 < z < 3	Questionable
(iii)	z ≥ 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. Program Schedule

April 2011	Preparation of samples
April-June 2011	Homogeneity & stability test of samples
August 2011	Invitation of APLAC members
September 2011	Dispatch of samples
October 2011	Collection of results and statistical analysis of data
November 2011	Interim report
December 2011-January 2012	Submission of final report

9. References

1. International Standards Organization. ISO/IEC 17043:2010, Conformity assessment - General requirements for proficiency testing, ISO, Geneva,

Switzerland.

2. International Standards Organization. ISO 13528:2005, Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO, Geneva, Switzerland.