INSTRUCTIONS FOR PARTICIPATING LABORATORIES

1. About the Proficiency Testing Sample

The Proficiency Testing (PT) material was prepared by the Chemical Metrology Laboratory (CML), Health Sciences Authority (HSA), Singapore by fortifying cake mix flour (purchased from a local supermarket) with acesulfame potassium and sucralose. The fortified cake mix flour was then adequately homogenised by mixing the flour in a drum mixer over 14 days before being distributed into polyethylene pouches under an inert atmosphere and controlled conditions (temperature and humidity). About 140 packets of the PT samples were prepared and stored between 18 °C and 25 °C. The mass fraction ranges of acesulfame potassium and sucralose are expected to be 500 – 1400 mg/kg and 500 – 1200 mg/kg, respectively.

The PT samples were evaluated for homogeneity and stability (at storage temperature of 18 °C to 25 °C for 3 months and maximum allowable transportation of 40 °C for 1 month) with reference to ISO 13528:2005. Statistical evaluations showed that the PT samples were sufficiently homogeneous and stable for the programme. The stability of the PT samples under the storage condition will be evaluated again before dispatch.

Each participating laboratory will be provided with one packet of the PT sample (about 50 g). In addition to the PT sample, pure substance certified reference materials (CRMs) of acesulfame potassium and sucralose (about 250 mg each) from HSA will also be provided. The CRMs should be used as calibrants for the determination of the analytes. The certified mass fraction values and associated expanded uncertainties of the pure substance CRMs are shown in Table 1.

Table 1: Information on pure substance CRMs of acesulfame potassium and sucralose

Pure substance CRM	CRM Code	Certified mass fraction value (mg/g)*	Expanded uncertainty (mg/g) at approx. 95% confidence level with coverage factor, k = 2)
Acesulfame potassium	HRM-1012A	999.2	5.0
Sucralose	HRM-1015A	985.4	4.8

*A certified mass fraction value of 1,000 mg/g is equivalent to a purity value of 100 %.

2. Storage of Proficiency Testing Sample and Certified Reference Materials

The PT packages will be delivered to the participating laboratories by their Accreditation Body (AB) under non-controlled temperature conditions. A temperature strip will be pasted on each package to enable the laboratories to check if it has been exposed to a high temperature. In addition, the laboratory should inspect the packages for any physical damage, then promptly acknowledge receipt of the PT packages or report any damages to the Coordinator by returning the Sample Receipt Form For Participating Laboratory.

The PT samples and CRMs should be stored between 18 ^{o}C to 25 ^{o}C. After opening the PT sample and CRMs, the polyethylene pouch and bottles should be resealed and recapped tightly.

3. Instruction for Use and Analysis

The participating laboratories are expected to apply the methods/procedures which they would normally use for routine analysis to determine the analytes in the PT sample. The CRMs provided shall be used as calibrants.

The participating laboratories should take a minimum of three subsamples from a minimum sample size of 5 g each for analysis.

It is essential for the participation laboratories to use the CRMs provided as calibrants to ensure that their results are included for performance evaluation. The laboratories may re-analyse the PT sample using their own standards as calibrants. However, the results will only be used for comparison purposes and not for performance evaluation.

4. Reporting and Submission of Results

The participating laboratories are expected to report the mass fraction of acesulfame potassium and sucralose using a Result Proforma. All results should be reported in mg/kg.

The participating laboratories shall report results obtained from the CRMs provided. The participating laboratories may report results obtained from their own standards but these results must be reported separately in the Result Proforma.

The uncertainty of measurement should be reported with a confidence level of approximately 95 %. The expanded uncertainty is to be reported to 2 significant figures and the results rounded off to the same number of decimal place(s) as the expanded uncertainty.

The participating laboratories are required to provide technical details of their methodologies in the Result Proformas. The completed Result Proformas must be returned to the Coordinator before the given deadline.

5. Safety and Handling

The PT sample and CRMs are intended for chemical analyses only. Disposal of the PT sample and CRMs should be carried out in accordance with existing regulations on waste management in the participating laboratories' country.

6. Evaluation of Performance of Participating Laboratories

The performance of the participating laboratories will be primarily assessed using the *z*-score. For laboratories which report the uncertainties of their results, the ζ-scores will also be calculated. The calculations of *z*-score and ζ-score are as follows:

(a) *z*-score: $$z = \frac{x - X}{\sigma_{PT}}$$

where, x is participating laboratory's result, X is the assigned value determined by HSA CML and σ_{PT} is the standard deviation for proficiency assessment.

A *z*-score with absolute value of: $|z| \leq 2.0$ is satisfactory

$2.0 < |z| < 3.0$ is questionable

$|z| \geq 3.0$ is unsatisfactory

(b) ζ-score: $$\zeta = \frac{x - X}{\sqrt{u_x^2 + u_X^2}}$$

where, u_x is the standard uncertainty of the participating laboratory's result x and u_X is the standard uncertainty of the assigned value X determined by HSA CML.

A ζ-score with absolute value of: $|\zeta| \leq 2.0$ is satisfactory

$2.0 < |\zeta| < 3.0$ is questionable

$|\zeta| \geq 3.0$ is unsatisfactory

The ζ-score provides an indication of whether a participating laboratory's estimate of uncertainty is consistent with the observed deviation from the assigned value. It is complementary to the *z*-score in the assessment of laboratory performance.

The σ_{PT} for both analytes will be determined using the robust standard deviation of the results reported by all the participating laboratories, calculated using Algorithm A in ISO 13528:2005.

Both z-score and ζ-score for the reported result(s) obtained using the CRMs provided and the participating laboratories' own standards will be calculated. However, only the results obtained from the use of the CRMs provided will be used to evaluate the performance of the participating laboratories.

7. Results Confidentiality

Each participating laboratory will be assigned a unique and confidential laboratory code. Both the identity and the code will only be known to staff of the Singapore Accreditation Council involved in the PT programme and the AB of the participating laboratory.

8. Collusion and Falsification of Results

It is the responsibility of the participating laboratory to avoid collusion of results. Collusion between participating laboratories will lead to the exclusion of the laboratories' results from the reports. In addition, a laboratory found to be falsifying results will have its results excluded from the reports.

9. Late Submission of Result Proforma

Participating laboratories should return their completed Result Proforma by the specified deadline in order to ensure that their results are included in the data analysis and report. Performance evaluation will not be carried out on results received after the reporting deadline.

10. Re-submission of Results

Re-submission of the Result Proforma due to modification of any information will not be accepted. Participating laboratories should ensure that all information in the Result Proforma are thoroughly checked before submission and submitted information are considered final.

11. Fees Payable

There is no participation fee for this PT programme for the participating laboratories nominated by their ABs.

12. References

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

13. Contact Details of Coordinator

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