Introduction

Sweet soy sauce, made from fermented black soy beans, is a condiment widely used in traditional Indonesian cuisine. Commercial sweet soy sauce usually contains preservatives such as sodium benzoate and methyl paraben in order to prevent spoilage and to increase the shelf life. The levels of preservatives that can be added to foodstuffs are regulated in Indonesia. The metrology institute of Indonesia (SNSU-BSN) provides an accuracy-based Proficiency Testing (PT) programme with traceable assigned value to International System of Units (SI) for the food testing laboratories in order to assess the measurement capability of the participating laboratories, to evaluate the reported measurement uncertainties and to spread awareness of the comparability of measurement result.

Sample Preparation

Commercial sweet soy sauces as raw material (containing sodium benzoate)  
Confirmation the presence of benzoic acid in the raw material  
Spiked with methyl paraben and n-butyl paraben  
Homogenization process  
Checked %RSD of five subsamples; Bottling and packaging  
Stored at room temperature (19 - 25°C)

Assigned Values and Statistics

The determination of the assigned values ($X_{PT}$) of benzoic acid, methyl paraben and n-butyl paraben were done using the exact-matching Gas Chromatography Isotope Dilution Mass Spectrometry (GC-IDMS). The assigned values are metrologically traceable to SI through the use of certified reference material provided by Health Sciences Authority (HSA, Singapore). These assigned values were used to provide an assessment of participant performance.

In this PT scheme, the standard deviations for proficiency assessment ($\sigma_{PT}$) were set using the Horwitz equation. The participants’ performance was mainly evaluated based on a metrologically traceable assigned value using $z$-score, as the $u(X_{PT})$ of each parameter was greater than $0.3\sigma_{PT}$. The $z$-scores was also provided as additional information in the report. The median and standard deviations (SD) of the participants’ result for each parameter was also determined as a comparison to the assigned value, except for the n-butyl paraben as there was only one measurement result.

Participants

There were seven testing laboratories in Indonesia participated in the PT scheme. Each participant received one sample which was packaged in a dark glass bottle and related documents such as the technical instruction, sample condition form, and results report form. The participants were asked to provide information about their uncertainty budget for their measurements, analytical method, as well as the calibrants used for measurement.

PT Results

The determination of the assigned values ($X_{PT}$) of benzoic acid, methyl paraben and n-butyl paraben were done using the exact-matching Gas Chromatography Isotope Dilution Mass Spectrometry (GC-IDMS). The assigned values are metrologically traceable to SI through the use of certified reference material provided by Health Sciences Authority (HSA, Singapore). These assigned values were used to provide an assessment of participant performance.

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Summary

All laboratories measured the sample by using HPLC and reported results for the three measurands investigated. Most of the participants (> 85%) obtained satisfactory $z$-scores for the three measurands. In addition, there was a good agreement between the nominal assigned values and the median of the reported results. All laboratories reported expanded measurement uncertainties, however some laboratories reported very small (underestimated) values, which need to be re-evaluated.

Acknowledgement

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Table 1. Summary of assigned values and statistics

<table>
<thead>
<tr>
<th>Parameter</th>
<th>$X_{PT}$</th>
<th>$u(X_{PT})$</th>
<th>$\chi_{PT}$</th>
<th>Median</th>
<th>SD (MCV)</th>
<th>Unit</th>
<th>Traceability to SI through</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzoic Acid</td>
<td>493</td>
<td>34</td>
<td>31</td>
<td>490</td>
<td>22 (4.4%)</td>
<td>mg kg$^{-1}$</td>
<td>HBM-1002A</td>
</tr>
<tr>
<td>Methyl Paraben</td>
<td>90.0</td>
<td>6.7</td>
<td>7.3</td>
<td>85.8</td>
<td>11.3 (13%)</td>
<td>mg kg$^{-1}$</td>
<td>HBM-1003A</td>
</tr>
<tr>
<td>n-Butyl Paraben</td>
<td>76.7</td>
<td>7.0</td>
<td>6.4</td>
<td>-</td>
<td>-</td>
<td>mg kg$^{-1}$</td>
<td>HBM-1004A</td>
</tr>
</tbody>
</table>

Notes: Satisfactory: Questionable