Introduction

In these days, importance of metrologically traceable reference materials for ensuring reliability of chemical analyses has been recognized. National Metrology Institute of Japan, National Institute of Advanced Industry, Science and Technology (NMIJ/AIST) and Chemicals Evaluation and Research Institute (CERI) have been supplying many types of reference materials. In 2015, the demand of traceable certified reference materials (CRMs) for drinking water analyses rose as an amendment of a notification under the Waterworks Act of Japan. In this presentation, NMIJ’s activities such as supplying organic CRMs and providing calibration services for purity assessment of organic compounds are outlined. Especially, efforts to improve efficiency of reference material supply by applying new techniques such as quantitative NMR (qNMR) are described.

CRM supply and Japan Calibration Service System (JCSS)

NMIJ provides the services and issues calibration certificates for domestic reference material producers. NMIJ’s calibration services of purity assessment (2018) (1) are as follows:

- qNMR & FID: 23 pesticides
- qNMR with verification by HPLC: 46 pesticides and 2 vitamins
- FID with verification by GC: 4 pesticides and 8 hydrocarbons etc.
- qNMR & titrimetry: 22 amino acids

Some of the pesticides are the Complementary items.

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Calibration service (purity assessment)

To realize rapid calibration services for purity assessment,

- Quantitative NMR is mainly applied.
- Continuous scan method by differential scanning calorimetry (DSC) is applied instead of time consuming other FPD methods.
- Area percentage method with GC or HPLC is used instead of the strict mass balance approach.

NMIJ provides the services and issues calibration certificates for domestic reference material producers.

Examples of high purity organic NMIJ CRMs

<table>
<thead>
<tr>
<th>Item</th>
<th>Characterization</th>
<th>Certified value ± U</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,4-Dichloro</td>
<td>Adriatic calyminety (A-MAA)</td>
<td>(0.9995 ± 0.0002)</td>
</tr>
<tr>
<td>Bromoacetone</td>
<td>Differential scanning calyminety (A-MAA)</td>
<td>(0.9993 ± 0.0012)</td>
</tr>
<tr>
<td>Trichloroacetic acid</td>
<td>Differential scanning calyminety &amp; Titrivity</td>
<td>kg/L</td>
</tr>
<tr>
<td>17F-Evaporation</td>
<td>NMIJ &amp; QNMR</td>
<td>kg/L</td>
</tr>
</tbody>
</table>

Some of these are the JCSS standard solutions.

Direct characterization of standard solutions for more efficient standard supply

NMIJ provides calibration services for CERI to characterize the JCSS specified standard solutions. To realize more efficient organic standards for safe water supply in Japan (51 items) allows use of them as the JCSS standards. A new notification (Notification of the Ministry of Health, Labour and Welfare No.56, March 2015) allows use of standard solutions which are traceable to national standards based on the Measurement Law of Japan.

![Diagram](https://www.nmij.jp/english/)

Conclusions

NMIJ supplies organic reference materials to end users directly or via the JCSS traceability scheme. In the latter case, high purity CRMs are supplied as primary standards for calibration of standard solutions. To realize more efficient organic standard supply, NMIJ has also provided calibration services of purity assessment for reference material producers by adopting the qNMR and other techniques. Furthermore, NMIJ has investigated methods for the direct characterization of target compounds in standard solutions. As a result, NMIJ has started calibration services for the designated calibration laboratory, CERI to directly characterize the JCSS specified standard solutions by qNMR/chromatography and post-column reaction GC.

Acknowledgements

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