Estimation of Uncertainty in Olfactometry

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Background
- Measurement of odour is a crucial element of odour management and regulation.
- Odour regulation based on the measurement of “odour index,” a sensory index of odour measured by the triangular odour bag method, was introduced in the Offensive Odour Control Law in 1995 in Japan.
- However, some problems related to the interpretation of measurement results have been reported by the municipalities.
- In order to solve the problems, the estimation of uncertainty in odour measurement seems to be effective.

Objectives
- To discuss the feasibility of estimation of measurement uncertainty in olfactometry, especially the measurement of odour index by the triangular odour bag method.
- To propose the estimation procedure of measurement uncertainty in olfactometry.

Triangular Odour Bag Method

Odour Index
- Odour index
  - Odour index = 10 log (Odour concentration)
- Odour concentration
  - Dilution ratio when odorous air is diluted by odorless air until the odour becomes unperceivable

Measurement Uncertainty
- Measurement uncertainty is a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand.
- Estimation of measurement uncertainty is essential to the interpretation of the results.
- The Guide to the expression of uncertainty in measurement (GUM), published by ISO (1995), provides a methodology for evaluating the measurement uncertainty associated with a result from a model of the measurement process.
Procedure for Estimation of Measurement Uncertainty

- Specification of measurement process
- Expression of measurement model and all influences
- Evaluation of standard uncertainty of each component
- Calculation of combined standard uncertainty: \( u(y) \)
- Determination of coverage factor: \( k \)
- Calculation of expanded uncertainty: \( U(y) = k u(y) \)

Application to Olfactometry (1)

- In air quality measurements, ISO 20988: 2007 provides comprehensive guidance and specific statistical procedures for uncertainty estimation.
- Experimental data for uncertainty estimation can be provided either by a single experimental design in a direct approach or by a combination of different experimental designs in an indirect approach.

Application to Olfactometry (2)

Direct approach
Input data are obtained in a single experimental design that provides information on deviations and bias by comparison with one or more reference values of the measurand.

Indirect approach
Input data are obtained in different experimental designs for the different input quantities of the method model equation used to calculate the result of measurement.

Application to Olfactometry (3)

- Experimental designs including simple random sampling and interlaboratory comparison of identical measuring systems are mentioned in ISO 20988.
- On the other hand, a very wide range of standard test methods is subjected to collaborative study in accordance with ISO 5725-2: 1994.

Application to Olfactometry (4)

- The triangular odour bag method is implemented in the form of a single detailed procedure described in the Notification No. 63 of the Japan Environment Agency.
- The sensory measurement of odour mainly consists of sample collection, panel selection and judgment of odour.
- Reference odour may influence the bias if quality control process using reference odour is implemented.
Estimation of Uncertainty

- Collaborative study such as interlaboratory comparison of olfactometry is conducted in order to collect basic data for the establishment of a quality control procedure and for the determination of quality criteria for olfactometry.
- These data yield performance indicators of measurement method including repeatability and reproducibility.
- Therefore, the use of collaborative test results for measurement uncertainty estimation according to ISO/TS 21748 and ISO 20988 is effective and reasonable.

Interlaboratory Tests (1)

<table>
<thead>
<tr>
<th>Year</th>
<th>2003</th>
<th>2004</th>
<th>2005</th>
</tr>
</thead>
<tbody>
<tr>
<td>Odour sample</td>
<td>Ethyl acetate (2000 ppm) + m-Xylene (94 ppm)</td>
<td>Dimethyl sulfide (3 ppm)</td>
<td>Dimethyl disulfide (3 ppm)</td>
</tr>
<tr>
<td>Number of laboratories</td>
<td>120</td>
<td>93</td>
<td>93</td>
</tr>
<tr>
<td>Repetition of measurement</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Repeatability</td>
<td>0.866</td>
<td>1.05</td>
<td>1.23</td>
</tr>
<tr>
<td>Reproducibility</td>
<td>1.86</td>
<td>2.45</td>
<td>3.34</td>
</tr>
</tbody>
</table>

Interlaboratory Tests (2)

<table>
<thead>
<tr>
<th>Year</th>
<th>2006</th>
<th>2007</th>
</tr>
</thead>
<tbody>
<tr>
<td>Odour sample</td>
<td>Field odour at sludge thickener of sewage treatment plant</td>
<td>Photogravure-like odour ( \text{Toluene (100 ppm) + 2-Propanol (55 ppm)} ) Ethyl acetate (60 ppm) + 2-Butanone (65 ppm)</td>
</tr>
<tr>
<td>Number of laboratories</td>
<td>86</td>
<td>116</td>
</tr>
<tr>
<td>Repetition of measurement</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Repeatability</td>
<td>0.936</td>
<td>1.01</td>
</tr>
<tr>
<td>Reproducibility</td>
<td>3.29</td>
<td>1.53</td>
</tr>
</tbody>
</table>

Statistical Analysis (1)

The simplest model underlying the statistical analysis of collaborative study data is given in Equation (1):

\[
y = m + B + e_r
\]

where, 
- \( m \): expectation for \( y \); 
- \( B \): laboratory component of bias under repeatability conditions, assumed to be normally distributed with mean 0 and standard deviation \( \sigma_L \); 
- \( e_r \): random error under repeatability conditions, assumed to be normally distributed with mean 0 and standard deviation \( \sigma_r \).

Statistical Analysis (2)

Since \( \sigma_B \) and \( \sigma_r \) are estimated by the repeatability standard deviation \( s_r \) and the interlaboratory standard deviation \( s_L \) obtained in an interlaboratory test, respectively, Equations (2) and (3) are obtained:

\[
u(B) = s_L \tag{2}
\]

\[
u(e_r) = s_r \tag{3}
\]

The combined standard uncertainty \( u(y) \) is given in Equation (4):

\[
u^2(y) = u^2(B) + u^2(e_r) = s_L^2 + s_r^2 \tag{4}
\]

Statistical Analysis (3)

The reproducibility variance \( s_R^2 = s_L^2 + s_r^2 \) can be substituted for \( s_L^2 + s_r^2 \) in Equation (4) and Equation (5) is obtained:

\[
u^2(y) = s_R^2 \tag{5}
\]

The reproducibility standard deviation is used for estimating the standard uncertainty and the expanded uncertainty is given in Equation (6) using a coverage factor \( k \):

\[
U(y) = k \cdot u(y) = k \cdot s_R \tag{6}
\]
Uncertainty Estimation Results

<table>
<thead>
<tr>
<th>Year</th>
<th>2003</th>
<th>2004</th>
<th>2005</th>
<th>2006</th>
<th>2007</th>
</tr>
</thead>
<tbody>
<tr>
<td>( u(B) = s_L )</td>
<td>0.866</td>
<td>1.05</td>
<td>1.23</td>
<td>0.936</td>
<td>1.01</td>
</tr>
<tr>
<td>( u(y) = s_R )</td>
<td>1.86</td>
<td>2.45</td>
<td>3.34</td>
<td>3.29</td>
<td>1.53</td>
</tr>
<tr>
<td>( k )</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>( U(y) = k \ u(y) )</td>
<td>3.7</td>
<td>4.9</td>
<td>6.7</td>
<td>6.6</td>
<td>3.1</td>
</tr>
<tr>
<td>( = k \ s_R )</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Conclusions

• The use of collaborative test results for measurement uncertainty estimation is effective and reasonable.

• Measurement uncertainty of the triangular odour bag method was estimated using interlaboratory comparison data, and the expanded uncertainty of odour index ranged between 3.1 and 6.7 (\( k = 2 \)).

• Based on the establishment of the estimation procedure of uncertainty, the coherent interpretation of measurement results and more effective and practical quality control of olfactometry will be available.