COMPARISON OF EPA APPROVED ODOUR MEASUREMENT METHODS

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Author: Trevor Bardsley

SUMMARY

EPA replaced an earlier odour measurement method with a more up to date method in October 2001. Since then, there has been a generally held perception that the new method would give different results from the old and, therefore, a correction factor would have to be applied to the results from the new method to make them comparable with the old.

This paper demonstrates that because the uncertainty associated with the old method was so great, measurements with the new method will always be within the likely range of results from the old. As the results of both methods have been found to be equivalent within confidence intervals, conversion factors unnecessary and should not be used.

BACKGROUND

On 16 October 2001 EPA adopted the Australian Standard (AS 4323.3, 2001) for odour measurement, replacing EPA Method B2, which was subsequently revoked by EPA on 30 June 2002. EPA licence monitoring for odour must now be conducted according to this method and the requirements of the amended guidelines for sampling and analysis of air emissions (EPA Publication 440).

During the development of the Australian Standard odour method from 1997 to 2001, EPA conducted a number of tests to investigate the relationship between B2 and the draft method for AS4323.3 (Bardsley and Demetriou, 1997 and 1999). A range of pure compounds and environmental samples were assessed. These studies showed a wide variation in the ratio between results generated using the two methods. The studies also indicated that the repeatability precision of the draft Australian Standard AS4323.3 was approximately a factor of 2 better than for the B2 Method. The better precision of AS4323.3 was attributed to the increased number of panellists, quantitative selection and screening procedures for odour panellists and the rigorous performance testing of odour assessment equipment (olfactometer). The introduction of retrospective screening of individual panellists results in AS4323.3 also improved the consistency of measurements. Where an individual panellist showed a large variation from the mean result of the panel, that panellist’s results were removed from the calculations.

Generally, EPA studies showed that there may be a bias towards higher mean values with AS4323.3 compared to B2, however other comparisons between odour measurement methods have not shown any scientifically rigorous differences from one method to another (Watts, 2000).

As the results of the EPA studies only represented measurements conducted by the EPA odour laboratory, a further study involving all Victorian NATA
accredited laboratories and major New South Wales odour laboratories was conducted in 2001. The study objectives were to evaluate the interlaboratory precision of both methods and provide guidance for EPA’s future odour measurement program.

RESULTS

Interlaboratory Odour Study

The interlaboratory study on odour measurements was conducted using EPA Method B2 and AS4323.3 on two standard odorants (n-butanol and hydrogen sulfide). The results were used to assess individual laboratory repeatability\(^1\) precision and reproducibility\(^2\) between laboratories.

Five Victorian odour laboratories determined odour thresholds using the EPA Method B2 and three NSW and one Victorian laboratory performed odour measurements on the same standard gases using AS4323.3. Each participating laboratory received the same certified standard odorants. Each standard odorant was analysed five times at separate panel sittings on different days. The laboratories were chosen because they were experienced with the method to be used. Only one Victorian laboratory had experience with AS4323.3 and no NSW laboratories were experienced with B2.

Graphical results of the mean odour threshold concentrations and range of results for the interlaboratory study are shown for the standard odorants n-butanol and hydrogen sulfide in Figures 1 and 2.

Within laboratory (repeatability) precision

EPA’s experience with the B2 Method has indicated that internal repeatability precision of ±50% was achievable with experienced panellists. For the interlaboratory study some laboratories using the B2 Method demonstrated repeatability precision better than ±50%, however, others failed to achieve this. Table 1 shows a measure of the relative variability in measurements within each laboratory.

Results for EPA Method B2 show a large variability in the internal repeatability precision, with some laboratories having very poor repeatability precision. By comparison, the laboratories using AS4323.3 demonstrated more consistent, and better, repeatability precision than those using the B2 Method.

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1 Repeatability precision involves tests being performed under conditions that are as constant as possible. i.e. same sample, same operator, same laboratory and same equipment. Note that individual panellists may have varied between tests.

2 Reproducibility precision involves tests on the same samples being carried out in different laboratories with different operators, panellists and equipment.
Table 1. Coefficient of within laboratory variation of odour threshold results for EPA Method B2 and AS4323.3

<table>
<thead>
<tr>
<th>Vic Lab No</th>
<th>N-butanol (%)</th>
<th>Hydrogen sulfide (%)</th>
<th>NSW Lab No</th>
<th>N-butanol (%)</th>
<th>Hydrogen sulfide (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>74</td>
<td>36</td>
<td>1</td>
<td>13</td>
<td>36</td>
</tr>
<tr>
<td>2</td>
<td>13</td>
<td>9</td>
<td>2</td>
<td>10</td>
<td>16</td>
</tr>
<tr>
<td>3</td>
<td>30</td>
<td>24</td>
<td>3</td>
<td>21</td>
<td>15</td>
</tr>
<tr>
<td>4</td>
<td>50</td>
<td>19</td>
<td>4</td>
<td>19</td>
<td>34</td>
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<tr>
<td>5</td>
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<td>35</td>
<td>5</td>
<td>29</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>97</td>
<td>81</td>
<td></td>
<td></td>
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</tr>
</tbody>
</table>

N-Butanol Threshold Values
Individual laboratories' odour measurements - n-butanol

Figure 1. Mean threshold and range of interlaboratory odour threshold results for the same n-butanol standard gas
Inter laboratory (reproducibility) precision

Interlaboratory results in Figures 1 and 2 show the mean odour threshold concentrations and range of results for n-butanol and hydrogen sulfide. From these figures, it is clear that all AS4323.3 odour threshold results lie within the range of results reported for the same odorant using the B2 Method.

The reproducibility precision observed for the B2 Method in the study was of concern. For n-butanol, the variation between laboratories was approximately a factor of 7 and for hydrogen sulfide a factor of 10. This compares with a factor of 2 for n-butanol and 2.5 for hydrogen sulfide with AS4323.3. With this level of variability for B2 odour measurements between NATA accredited laboratories, little confidence could be had in comparison of results between laboratories.

An assessment of the reproducibility precision for both methods was conducted based on the 95% confidence intervals3 of the interlaboratory results. Results showed that measurements performed with AS4323.3 fell within or overlap the broad confidence intervals of B2 results (Table 2). This means there is no rationale for a conversion factor to adjust results between methods to produce equivalent results.

3 A 95% confidence interval means that there is a 95% probability that the true value lies within the reported range.
COMPARISON OF EPA APPROVED ODOUR MEASUREMENT METHODS

Table 2. Odour threshold results and 95% confidence intervals for standard odorants n-butanol and hydrogen sulfide

<table>
<thead>
<tr>
<th></th>
<th>EPA Method B2</th>
<th>AS4323.3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vic Lab No</td>
<td>N-butanol (ppb)</td>
<td>Hydrogen sulfide (ppb)</td>
</tr>
<tr>
<td>1</td>
<td>20 ≤ 90 ≤ 340</td>
<td>0.2 ≤ 0.3 ≤ 0.6</td>
</tr>
<tr>
<td>2</td>
<td>110 ≤ 140 ≤ 190</td>
<td>2.5 ≤ 2.9 ≤ 3.5</td>
</tr>
<tr>
<td>3</td>
<td>30 ≤ 70 ≤ 140</td>
<td>0.7 ≤ 1.1 ≤ 1.7</td>
</tr>
<tr>
<td>4</td>
<td>30 ≤ 70 ≤ 170</td>
<td>0.4 ≤ 0.8 ≤ 1.7</td>
</tr>
<tr>
<td>5</td>
<td>10 ≤ 20 ≤ 40</td>
<td>0.2 ≤ 0.9 ≤ 3.5</td>
</tr>
<tr>
<td>6</td>
<td>3 ≤ 30 ≤ 320</td>
<td></td>
</tr>
</tbody>
</table>

Table 3 shows the coefficient of variation of the between laboratory results and further demonstrates the superior reproducibility precision of AS4323.3 compared with B2.

Table 3. Coefficient of between laboratory variation of odour threshold results for EPA Method B2 and AS4323.3

<table>
<thead>
<tr>
<th></th>
<th>EPA Method B2</th>
<th>AS4323.3</th>
</tr>
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<tbody>
<tr>
<td>N-butanol (%)</td>
<td>Hydrogen sulfide (%)</td>
<td>N-butanol (%)</td>
</tr>
<tr>
<td>70</td>
<td>79</td>
<td>33</td>
</tr>
</tbody>
</table>

In light of the interlaboratory odour results, EPA has adopted AS4323.3 as the EPA approved method for odour measurement. Laboratories conducting licence monitoring for odour must now comply with the requirements of AS4323.3 and the guidelines specified in EPA Publication 440, which have been included to further enhance consistency of odour measurement between laboratories.

CONCLUSIONS

The level of variability for B2 odour measurements between NATA accredited laboratories gave little confidence that comparison of results between laboratories could be made. The wide range of results indicates that the application of any conversion factor to translate B2 Method results to AS4323.3 results is inappropriate.

From the results of the interlaboratory study and the observed confidence intervals of measurement methods, it was also concluded that results for AS4323.3 consistently fall within or overlap the confidence intervals of B2 measurements and therefore results are deemed to be equivalent. Laboratories using AS4323.3 showed better within laboratory repeatability for odour threshold.
determinations than laboratories using B2, but more importantly, the reproducibility of measurements between laboratories was dramatically greater than that achieved with the B2 Method.

Adoption of AS4323.3 and the amended guidelines for sampling and analysis for odour (EPA Publication 440) will produce:

- greater confidence in results and improved precision due to better quality assurance in the operation of the olfactometer and selection and use of panellists.
- results which are more reproducible and enable licence holders to better understand and control their odorous emissions.
- more precise and reproducible results for computer modellers to use which will enhance modelling and better reflect real world situations.
- odour measurements which will be consistent with those laboratories adopting Australian/ISO Standards both nationally and internationally.

**REFERENCES**


Bardsley T. B and Demetriou J., (1997) *Odour Measurements That Don’t Stink* Odour Workshop University of New South Wales


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