|  |  |  |  |
| --- | --- | --- | --- |
|  | **CIML online ballot and comments** | | |
| Comments on: | **R 100 Atomic absorption spectrometer systems for measuring metal pollutants in water** | |
| Country/Liaison Organisation: | **NMO United Kingdom** | Date: 08 July 2013 |
|  | Comments accompanying the UK’s ‘Abstention’ vote | |

| **Member State/ Liaison** | **Item** | **Comment** | **Response** |
| --- | --- | --- | --- |
| UK | 8.3.2 | (i) Is this requirement in addition to, or instead of, regular QC samples placed at intervals throughout the instrument run, which would normally be recommended in longer runs? Perhaps it would it be more useful to specify a maximum number of test samples between successive QC checks of this kind; for example, at least one reference standard solution should be run per 10 test samples, with not less than one such QC at the beginning and end of each measurement run.  (ii) Ideally, QC materials should be prepared completely independently from those used in calibration, since using one of the calibration standards in a dilution sequence will not pick up incorrect preparation of stock solution. Perhaps a note could be included to stress this best practice advice if it is not practical to include it as a formal requirement?  **We still believe that this paragraph be re-drafted along the lines of our comments.** |  |
| UK | 8.3.3 | A performance test of this kind would normally be something akin to a system suitability check, perhaps involving a single well-characterised test material run 3-5 times to check precision before calibrating or a simple check on LOD. If so, it would be useful to provide an indication of what tests were to be done (for example a statistical test of whether the precision had genuinely deteriorated), or at least provide some warning as a Note, so that the limited amount of data (cf. Box 1 above) did not lead to unnecessary servicing or adjustment of the instrument.  **We still believe that this point needs addressing as the current**  **wording of this paragraph is not sufficiently specific.** |  |
| UK | A1.1 | Drying of pure materials certified for calibration of instruments should normally follow supplier recommendations for use rather than a fixed prescription. We suggest adding ‘unless otherwise directed by the supplier of the material’.  **We still believe that this comment needs to be taken on board as it compromises metrological principles otherwise.** |  |
| UK | B4.5.1-4.5.3 | We still believe that there seems to be a potentially serious mismatch between required precision and required r2 here. Comparison with literature [Accred Qual Assur (2006) 11: 146-152] on available standard error of prediction, which is dominated by the residual SD, indicates that r2 equal to 0.98 based on five mean values of ten observations each would be associated with a residual standard deviation well over 5 % of the mean value of the response. This would not be sufficient to meet the metrological requirements of Tables 1 and 2 and para 5.5. If the linear fit is to as few as three values, the situation becomes worse. To address this, r2 would (based on the tables in the reference cited) need to be better than 0.99 and should be calculated from the independent data points rather than the mean values. If calculated from the mean values, r2 would probably need to be above 0.999 to provide satisfactory precision at the mid-point. While we have checked this via a brief simulation, we would strongly suggest that the r2 criterion be carefully reviewed and tested to ensure that it is consistent with the required precision. We have picked-up comments from our network of contacts in the UK who have significant experience and expertise in the analysis of waters for metal pollutants. This network agrees strongly with our view on Sections B4.5.1-4.5.3, and is of the opinion that such an approach is not in compliance with European Union guidelines and practice. They also believe strongly we should be encouraging the use of more modern technology that is more sensitive and less susceptible to interference. |  |
| UK | Annex B | In Annex B, there is no Section B.3. The text jumps straight from B.2 to B.4, and section  B.4.3.1 references B.3.3.1. |  |