

EXECUTIVE SUMMARY

To reinforce the UK's case for the inclusion of an analysis using gas chromatography-mass spectrometry (GCMS) to detect unsuspected compounds in migration waters from non-metallic construction products used in contact with public water supplies, the performance of the existing UK test method (BS6920:2001 Part 4) has been assessed by three of the laboratories approved by the UK regulatory body (the Committee on Products and Processes (CPP)). This performance test was supervised by WRc-NSF and the laboratories that participated were WRc-NSF, Law Laboratories and Intertek.

The performance testing exercise had two components. Firstly, three batches of migration waters (unchlorinated and chlorinated) prepared from different products were circulated to the participating laboratories. As the true concentrations of the compounds detected in real migration waters are not known, the results from these samples were used to check the comparability of the data produced by the different laboratories. Each laboratory analysed five samples (test water, chlorinated test water, migration water, chlorinated migration water and a laboratory blank) for each of three products. The second component of the exercise involved an assessment of the semi-quantitative performance of the method, which was checked by analysing samples to which known compounds (fifteen) had been added at known concentrations. Each laboratory analysed three batches of four samples (test water spiked at a low concentration, chlorinated test water spiked at a low concentration, test water spiked at a high concentration, chlorinated test water spiked at a high concentration).

The results from the analysis of real samples indicated that the three laboratories produced broadly comparable data. For two batches of migration waters, all laboratories reported very few compounds at low concentrations. For the other batch of migration waters, where the concentrations of the compounds detected were very high (hundreds of $\mu\text{g l}^{-1}$), the variation between the highest and lowest concentrations reported for the same compounds by the different laboratories was high (by factors of $2\times$ to $3\times$). Potential reasons for this variation include the use of different GCMS instruments, GC columns and internal standard solutions by each participant.

By comparing the relative responses of the internal standards (to one of these, d_{10} -phenanthrene), it is possible to determine the relative standard deviations of the responses for eight of the nine internal standards for each batch of migration waters analysed. It is also possible to quantify the remaining standards on the basis of their relative responses compared to d_{10} -phenanthrene, and obtain an indication of the uncertainty of measurement for the overall procedure, as the true concentrations of all of the internal standards are known. It is suggested that each laboratory could maintain a record of their performance on this basis, which may satisfy UKAS' concerns regarding this method.

The data from the analysis of the spiked test water and spiked chlorinated test water samples containing known compounds at known concentrations indicated that there may be some bias due to chlorination, but this is small in relation to the precision of the analysis. The results from the samples spiked at low concentrations ($0.86 - 2.04 \mu\text{g l}^{-1}$) confirm that the ability of the approved laboratories to detect and report the presence of

these determinands is relatively good, with two of the participating laboratories reporting the concentrations for thirteen of the compounds present. The compounds that caused problems were tetrahydrofuran (THF), which is very volatile, N-methylpyrrolidinone which is polar and basic and bis-phenolA diglycidyl ether (BADGE) which would normally be determined using a HPLC-based method, rather than GCMS. These three compounds were included in the spiking mixtures because they were known to be “difficult” compounds to analyse using BS6920:2001 Part 4. The results from the samples spiked at high concentrations ($8.6 - 20.4 \mu\text{g l}^{-1}$) were satisfactory, with variation (% relative standard deviation) being less than 25% for most compounds. The exceptions were the three “difficult” compounds noted earlier. With respect to bias (i.e. the deviation between the reported concentrations and the true concentrations) the results from two laboratories consistently exhibited negative bias, while those from the other laboratory were predominantly positively biased. This may be due to the use of biased internal standard solutions, which could be addressed if internal standard solutions from a single source were available.

The relative GCMS responses of the compounds used in the spiking mixtures was determined by WRC-NSF and, on a per nanogram basis, there was a five-fold variation between the most and least responsive compound (BHT and BADGE, respectively). Given that there is also variation in the responses of the individual internal standards which are used to produce the quantitative estimates of the compounds detected in migration water samples, the results from the spiked samples are better than expected, with the bias for the samples spiked at low concentrations in the range from +150% to -100%, and in the range -20% to +15% for the samples spiked at high concentrations.

Compared to the performance achieved during the EU 5th Framework programme CPDW project, where six of the seven laboratories that participated had no previous experience of applying BS6920:2001 Part 4, the results of this current exercise are an improvement. This suggests that, as expected, experienced users can achieve better performance from the method than inexperienced users, and reinforces the view that following incorporation of this GCMS method into the EAS other European laboratories would be able to utilise the method and obtain similar performance data to the UK laboratories.

It is recommended that:

- the supply of isotopically-labelled internal standards from a single commercial source should be investigated;
- consideration should be given to modifying BS6920:2001 Part 4 to include specifications for the GC column to be used and the temperature programme to be used for the GCMS analysis; additional information on these points will emerge from DG Enterprise-funded work which will commence in April 2005;
- any compounds of interest which have a boiling point lower than that of d_6 -benzene (79°C) should be analysed as specified compounds using, for example, purge and trap GCMS rather than the procedures specified in BS6920:2001 Part 4.