APPROVAL OF PRODUCTS FOR USE WITH DRINKING WATER

TEST PROTOCOLS FOR DESIGNATED LABORATORIES

Leaching of substances from products used in contact with water intended for human consumption

Protocol 1
Reporting requirements

Version 3.5 – June 2016

INTRODUCTION

This test protocol is one of a series prepared by the Drinking Water Inspectorate (DWI) to provide guidance to test laboratories on procedures to be used in evaluating the suitability of products for use in the treatment and distribution of water intended for human consumption. These procedures are designed to ensure a consistent approach to testing by the designated test laboratories.

The protocols currently available are listed below.

Number	Title
0	Designated test laboratory requirements
1	**Leaching of substances from products used in contact with water intended for human consumption : Reporting requirements**
2	Leaching of substances from products used in contact with water intended for human consumption: General Method
3	Leaching of substances from products used in contact with water intended for human consumption : Admixtures for cementitious products
4	Leaching of substances from metallic products used in contact with water intended for human consumption: General method – *provisional*
5	Leaching of substances from products used in contact with water intended for human consumption : Water treatment membranes
6	Leaching of substances from non-metallic products used in contact with water intended for human consumption : Filter media and ion exchange resins

IMPACT OF EUROPEAN TECHNICAL REQUIREMENTS

Currently a whole series of test methods are being drafted within CEN in support of the approval of products used with water intended for human consumption. As these are published any conflicting national test protocols will have to be withdrawn.
It is currently anticipated that published EN standards will become available for most of the areas covered by these protocols during the next few years.

AVAILABILITY

Copies of these test protocols, together with information requirements for applicants, can be freely downloaded from our website – http://www.dwi.gov.uk/drinking-water-products/advice-and-approval/index.htm

CONTACT

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Revision notes –
Version 2.0 – inclusion of BS EN 12873-1 & addition of documents previously issued separately – Annexes A to D; v 2.1 – inclusion of BS EN 12873-2; deletion of Annex D (now included in Test Protocol 2); v 2.2 – clarification of reporting requirements for other reports for consideration by the former CPP (Annex D) and minor revision of Section 1.6; v 2.3 – minor amendments and clarifications (section 1.5 and 1.6)
Version 3.0 – revisions to reflect new approval processes and requirements, together with addition of annexes D and E; v 3.1 – new section 1.7 (Confidentiality); v 3.2 – new section 3, v 3.3 – updating of regulatory requirements. V 3.4 address change, V3.5 change to England and Wales Regulations.

CONTENTS

GENERAL DEFINITIONS (for use with all Test Protocols)

The relevant regulations (for public drinking water suppliers)
The following regulations apply to the approval of substances and products used in the provision of public water supplies within the United Kingdom:

a) England - Regulation 31 of The Water Supply (Water Quality) Regulations 2016 (Statutory Instruments 2016 No 614) – http://www.legislation.gov.uk/uksi/2016/614/contents/made
b) Wales – Regulation 31 of The Water Supply (Water Quality) Regulations 2010 (Welsh Statutory Instrument 2010 No 994 (W.99) - http://www.legislation.gov.uk/wsi/2010/994/contents/made (and Amendment Regulations 2016 No. 410 (W. 128))
c) Scotland – Regulation 33 of The Public Water Supplies (Scotland) Regulations 2014 – http://www.legislation.gov.uk/ssi/2014/364/contents/made
d) Northern Ireland – Regulation 30 of The Water Supply (Water Quality) (Amendment) Regulations (Northern Ireland) 2009 (Statutory Rules of Northern Ireland 2009 No.246) - http://www.opsi.gov.uk/sr/sr2009/nisr_20090246_en_1

Where reference is required to specific regulatory requirements, these are given in footnotes.

REPORTING REQUIREMENTS : FORMAT & CONTENT OF LABORATORY TEST REPORTS

0. INTRODUCTION

The DWI advises the U.K. Authorities on the approval of products and processes to be used in contact with public water supplies (see Section 1 of Advice Sheet 1). Products submitted for approval are usually subjected to testing to assess their potential for leaching of toxic substances into water. This protocol sets out test report requirements based upon the needs of the DWI, following discussions with the test laboratories.

1. TEST REPORT

This section sets out the reporting requirements associated with analysis carried out on leachates from the test product.

1.1 Executive summary

Test reports shall include a separate executive summary, in plain, concise English, covering the following issues:

- The product tested
- Reference to the test methods used
- The outcome of the testing
- Any specific issues or results that are likely to be of concern
- Description and justification of deviations from the prescribed method and explanation of any unusual results

1.2 Test Sample/Specimen details

These shall include the requirements as set out in BS 6920-4:2001, BS EN 12873 parts 1 and 2 or any other relevant test protocol (including other DWI protocols in this series).
Aspects covered by the report shall include, as a minimum -

- Product name
- Manufacturer
- Location of production facility
- Product code and batch code ("date of manufacture" would be a suitable acceptable alternative, if batch code is not available)
- Description of test sample including dimensions and methods of preparation
- Volume of test water
- Product surface area exposed to test water
- Surface area to volume (S/V) ratio expressed as dm^{-1}
- For laboratory prepared samples, the actual temperature of the substrate at the time it is coated prior to placing in a temperature controlled environment and the temperature range experienced during the cure.
- For site applied products the details of actual temperatures, relative humidity and duration of curing
- A chain of custody if test samples have been prepared by a third party or off-site

1.3 Leachate preparation

This shall be in accordance with BS 6920-4:2001, EN 12873, parts 1 and 2 or any other relevant test protocol (including other DWI protocols in this series, or test protocols developed by the test laboratories[1]).

Aspects covered by the report shall include, as a minimum -

- Summary of method used with full details of test conditions and leaching periods
- A reference to the test method
- Deviations from the prescribed procedure and reasons for any deviations
- Surface area of test sample
- Volume of test water in contact with the test sample
- S/V ratio
- Source and methods of purification of test water
- Date and time of commencement of each leaching period
- Date and time of collection of each leachate
- Chlorine concentrations in bulk chlorinated test water at the beginning of each leaching period and, for each leachate and procedural control sample, measured at the end of leaching periods

1.4 Leachate analysis

These shall be in accordance with BS 6920-4:2001, BS EN 12873 parts 1 and 2 or any other relevant test protocol (including other DWI protocols in this series, or test protocols developed by the test laboratories[1]).

Aspects covered by the report shall include, as a minimum -

- A reference to the written leaching test specification from the DWI
- Summary description of analytical methods
- References to methods used[2]
- Statement of the performance characteristics (limit of detection, calibration range, trueness and recovery) with supporting data
- Date and time of extraction
- Date and time of instrumental analysis
- Deviations from the prescribed procedure and reasons for any deviations
- Stability of analyte in water, if known

1.5 Test results

The test report shall include:

- Results in tabular form – reported in accordance with any specific requirements of the test protocol used – normally this will be the concentration and absolute values of the various leachate determinands (µg/l) against time intervals, giving individual results of any replicates, rather than means
- Tabular and graphical plots of leaching concentrations (µg/l) and migration rates (µg/dm²/d) against time (h)
- Information on the level of confidence in the measurements presented, including details of the performance characteristics of the methods of analysis

[1] Reference should be made to published and un-published methods e.g. in-house methods. In the latter case the laboratory must be prepared to provide a copy if requested

[2] Full performance characteristics may not be available for non-routine determinands – in these situations the DWI may accept less comprehensive evidence for performance of the method of analysis, depending on the level of risk posed by the analyte in question.

- A brief statement about whether the results have been adjusted in any way e.g. blank correction
- Details of any unanticipated event during the test procedure[3] and analysis
- Dates of extraction and injection

More detail on reporting concentrations is given in annex E.

1.5.1 *Raw Data*

The raw data appendices should include the following:

- Raw data (chromatograms, spectra etc.) - these should be clearly labelled to identify the test product, the leachate and the test water type, e.g. blank or product name, leachate number, chlorinated test water etc
- Details of procedural blanks and controls used, with information on whether analytical results have been blank corrected. Where possible, the origin of the artefact or contamination should be stated
- AQC results from the batch and the criteria for acceptance of test results. Graphical presentation of data, such as the peak areas of the internal standards and Shewhart style control charts, are required
- Adequate means of cross referencing between the discussion/reporting sections and the actual peaks in spectra and chromatograms etc. It is also important that a cross reference (time or peak number) between each spectrum and its annotated chromatographic peak is provided
- Chromatograms and the relevant blanks or laboratory standards are conveniently presented on a single sheet for easy comparison. Likewise, the spectra of unknowns should be printed on the same sheet as the best library match(es)

1.6 Report format

The test report shall be submitted electronically (E-mail attachment(s) or CD ROM) to the DWI as a single document including all contributions from the participating test and analytical laboratories. The co-ordinating laboratory shall include an executive summary describing which contractors carried out the various parts of the work, and providing an overall summary of the results.

All the pages of the report shall be numbered (x of y pages format, including the annexes and the spectra etc. Each annex may start at page 1). All tables, graphs and chromatograms shall be uniquely numbered. The DWI requires a declaration that the testing laboratory has followed the DWI's instructions.

Submit the results of testing in the following formats –

a. An electronic report (MS Word or pdf file format), including all the information set out in this protocol and any other relevant DWI Test Protocol of method (including in-house methods), with the exception of chromatograms, charts and computerised analytical print-outs; this report will be made available to the DWI
b. A full report, including all supporting chromatograms, charts and computerised analytical print-outs; this report will be made available to the analytical expert members of the DWI. Include a completed copy of the AQC checklist with each full report– see **Annex A** of DWI **Test Protocol 2**. Please submit this final report electronically (MS Word or Adobe Acrobat pdf file format). The electronic raw data files for the GC-MS analysis should be provided on a CD or DVD ROM.

[3] An example of an unusual result might be the finding in the blank an unanticipated substance – such a finding, unless adequately explained, could undermine confidence in the credibility of the test data.

1.7 Confidentiality

Some specific determinands requested by the Inspectorate will have been provided in strict confidence, as part of the application process, by suppliers to the applicant; their identities will not normally be known to the applicant and all reasonable measures must be taken to ensure the security of this information.

No information shall be included in any report that would allow any possible identification of anonymised compounds. This exclusion will include such information as outline details of the test methods used or developed, together with supporting validation data and results, as well as the identity and potential source of these compounds. Where this has been requested, submit this information, in confidence, separately to the Inspectorate. This information must be sent to the Inspectorate in a secure, traceable manner.

2. COMMENTARY AND ADDITIONAL INFORMATION

This section contains additional DWI requirements for reporting.

2.1 Toxicological report & assessment

The DWI ultimately needs to assess whether the concentrations of substances reported in leachates are of significance to the health of consumers. The issues of confidentiality dictate that the test laboratories, rather than the applicant, are likely to have access to commercial-in-confidence data relating to the product. The laboratories are therefore well placed to provide information that will assist the DWI in its risk assessment.

Whilst it is not a mandatory requirement that a laboratory offers this service, if it does so, it is essential that the applicant is made aware of the likely costs that will be involved. It is important also that the applicant understands that when leaching test data indicates significant leaching, this will inevitably raise questions to be answered by the applicant or the laboratory. These questions need to be resolved before any recommendation for approval can be made.

The areas where test laboratories can assist include:

- Providing references/summaries of relevant toxicological studies for substances exhibiting long-term leaching, including hard copies of all references cited
- Reporting on the relationship between the concentrations indicated by the test data and the concentrations expected under practical exposure conditions in water supply systems
- Submitting calculations that indicate the worst case leaching rates for specific analytes

If the development of suitable test methods for specific determinands proves to be impractical, then a risk assessment, based upon toxicological considerations, may be appropriate. This assessment should be presented in five sections -

1) Introduction – this should include a summary of the method development work undertaken for the specific determinand (including mention of the various methods considered and tried) together with the limits of detection achieved and calibration range, trueness and recovery
2) exposure assessment
3) toxicity data
4) toxicity assessment
5) overall risk

The risk assessment should include full bibliographic details of all references cited and a hard copy of each supporting reference or review should be submitted to the DWI .

For further advice see **Annex C**.

2.2 Discussion

This part of the report should include a comprehensive discussion and interpretation of the leaching test results and the AQC. The focus of the discussion should be to enable the DWI to assess the risk posed by the product.

Comments should be made on:

- the significance of any variations from prescribed test conditions and on any unusual or unexpected results.
- any results that are likely to be contentious and where appropriate, justify their acceptance - this may include consideration of the extent to which the laboratory test conditions adequately represent the worst-case conditions to consumer exposure.
- any toxicological data that is relevant to the test results.

2.3 Conclusions

Unlike the executive summary, this may include technical matters as well as a summary of results.
It should draw together the discussion and present:

- A concise summary of what was found
- Summaries of additional work elucidating an unusual finding
- The salient factors relating to each leachate found at higher than 1µg/l concentration
- Relevant comparisons with any other materials or test method (especially when a sample has been submitted for re-testing)

3 PAYMENT FOR TESTING

The designated test laboratory shall be responsible for recovering the costs for carrying out any testing they have undertaken in respect of requirements specified by the Inspectorate. The Inspectorate cannot assist designated test laboratories in recovering payment from applicants for testing.

It is the responsibility of the designated test laboratory to ensure that it has received full payment for all test work undertaken before the relevant test report(s) is submitted to the Inspectorate for final product assessment.

ANNEX A : REPORTING OF PROCEDURAL BLANK RESULTS

A.1 INTRODUCTION

There are three types of solution created and analysed during a leaching test:

- The *leachates* are the experimental results from which we gain information on the product being tested
- The *standards* (including an analytical blank) are prepared independently as part of the chemical analysis (and independently of them, AQC standards to check the validity of the analysis)
- The *procedural blank* is test water treated in exactly the same way as the leachate except that it is never in contact with the test piece

The detection of compounds that could be toxic at 1µg/L is analytically demanding and contamination is an ever-present problem.

The standard solutions confirm that if a compound is in the leachate it can be detected above a known threshold and ensure a reasonable estimate of its concentration can be obtained.

The procedural blanks should identify whether any substances of interest are present in the test water, or are introduced as contaminants, e.g. from the laboratory during the test procedure. External effects on the procedural blank include:

- General changes in water chemistry
- Contamination that can be eliminated from consideration
- Degradation of reagents
- Loss of volatile components
- A myriad of other failures in the test and analytical procedures

It follows that any and all differences between the leachates and procedural blanks must be investigated. The DWI needs to see this has been done and if the differences are thought to be due to anything other than compounds leaching from the test piece, reasonable proof is required that this is the case. The level of proof required varies with the toxicity of the compound.

A.2 REQUIREMENTS

BS6920-4:2001 describes the preparation of the *procedural blank* (clause 6.2) and indicates how it is to be used. But standards have not been set which would assist in interpreting the results, although the note on page 11 of this standard gives criteria for deciding how to present results. It is difficult to be prescriptive because of the wide range of things that can go wrong during a leaching test and the subsequent analysis. The following points indicate what the DWI looks for when scrutinizing a laboratory report –

A.2.1 The results from the procedural blanks

These should be reported in the same way as the leachates – without corrections for recovery, etc. If blank corrected results are reported, they should be in addition to tables of the raw results.

A.2.2 The background “noise” in chromatograms

This should be the same for leachates and procedural blanks – including both the size and retention times of peaks due to laboratory contaminants. Any apparent difference in magnitude ought to be due solely to the auto-scaling of the chromatogram to the largest peak, although stochastic variation is expected, especially at low levels and for airborne contaminants.

A.2.3 Excessive low peaks in the Blank

The sensible procedure when there are a lot of low peaks in the blank is to run the solvent direct from the bottle (after concentration) to see if it is indeed the extractant creating the problem. One injection for each bottle should suffice unless contamination is suspected during the bottle's life.

A.2.4 The large variation in peak height

This is common experience and raises another issue when concentrations in the procedural blank and the leachate are both high. The accepted procedure is to subtract results for the procedural blank from the leachate result to arrive at the amount contributed by the test piece. We are concerned that this is likely to be very inaccurate. It is even possible for there to be a real effect when the leachate readout is lower than the procedural blank. Although there may be no statistically significant difference in these circumstances, we look at a worst case based on the absolute values and the variance evident from the test solutions. If there is a possibility that an amount of toxic significance could be present, we are likely to request further work.

The general chemistry of the water is unlikely to change during a leaching test on most organic materials. The chlorinated water leachate from the test sample may well contain a different (lower) concentration of free-chlorine than a procedural blank, but otherwise pH, conductivity and the major ions (analyte) will be unaffected. The same is not necessarily true of membranes, adsorbants and ion exchange resins. Many of these parameters are not routinely measured, but the laboratory would be expected to undertake additional analyses if general water chemistry might change during the leaching tests, and this will include the procedural blank as a reference.

ANNEX B : ESTIMATION OF "WORST-CASE" LEACHING FROM PRODUCTS

B.1 INTRODUCTION

When considering applications for approval the DWI needs to assess whether migration of contaminants from materials into water poses a risk to the health of consumers. The DWI's assessment is based on the results of leaching tests in which the products under test are exposed to water under controlled conditions.

Under certain circumstances it may not be possible, or even necessary, to obtain experimental leaching data:

a. The method of analysis may not be sufficiently sensitive
b. The current technology may not provide any analytical method for the determination of the contaminant in water
c. When the substance under consideration is a minor constituent of the product, there may not be any need for analytical determination

In such cases, some form of "worst-case" estimate of the likely concentration must be obtained.
This Annex outlines the approaches in estimating the likely consumers' exposure to the contaminants from the experimental leaching data and suggests principles to be adopted when carrying out estimation of leaching in perceived "worst-cases" where adequate experimental data is not available. It is based on current practices and considers some typical cases.

Whether adequate experimental leaching data is available, or not, the methods of estimating the consumers' exposure will depend on a number of factors and assumptions made. It will also vary for different products and different conditions of use.

In some cases it will be impossible to develop suitable test methods for compounds of interest requested by the DWI. In this case an assessment of potential risk to the health of consumers should be undertaken in accordance with the guidelines given in **Annex C**.

B.2 GENERAL PRINCIPLES OF EXPOSURE ESTIMATION

B.2.1 Factors affecting exposure

The exposure of consumers to a contaminant arising from products used in contact with water supplies is derived from its estimated concentrations in water at the tap. This concentration will mainly depend on the following major factors:

- Surface area of the material in contact with a specified volume of water (**S/V ratio**)
- How long the water is in contact with the material (**contact time**)
- The **leaching rate** of the contaminant from the material into water, that is the amount of the substance leaching from a specified surface area over a specified time (e.g. µg dm^{-2} day^{-1})

The leaching rate depends on a number of factors, particularly the following:

- Physical and chemical properties of the contaminant (polarity, solubility, etc.) and the material (porosity, density, etc.)
- Amount of the contaminant in the material
- Chemical characteristics of the water (such as pH or hardness)

- Temperature
- Time; usually the rate decreases with age of the product. This decrease depends on the length of time since its installation and conditions of its use, in particular the total volume of water that has passed over the surface

Ideally the "worst-case" exposure would be determined by measuring the concentrations of the contaminant directly under field conditions, over a period of time, at the highest S/V ratio and the longest contact time likely to be used in practice. With the exception of water filtration and electrodialysis units (see Test Protocols 5.1 and 5.2) leaching rates for products are calculated from the results of laboratory leaching tests under standard test conditions and converted to the likely "worst-case" exposure concentrations, taking into account the S/V ratio and contact time relevant in practice for the product under test.

Under certain circumstances it may be sufficient to estimate the potential maximum consumers' exposure to a contaminant from a product for specified use without obtaining experimental data, after taking into consideration the factors listed above. Such estimates would be undertaken to show whether the maximum possible daily leaching rates of the contaminant at the tap would be either:

a. Well below exposure levels generally regarded as insignificant as far as consumers health was concerned (equivalent to ~1µg day^{-1})
b. Below levels judged by the DWI to be acceptable on the basis of toxicity data provided by the applicant – see also **Annex C**

The basis of the calculations of exposure concentrations from experimental leaching data is summarised below. The situations where adequate leaching data is not available or not needed are considered in Annex B3.

B.2.2 Laboratory leaching tests

B.2.2.1 General test conditions

Test Protocol 2 and BS EN 12873, parts 1 and 2 describe the conditions (S/V test ratio, and number and duration of the leaching periods) usually employed for leaching tests on a variety of products. Leaching of a substance from a product is taken to be directly proportional to the surface area exposed to water as well as to the contact time. Hence the results of a laboratory leaching experiment under one set of conditions can be used to predict the concentrations of contaminants under other conditions, more typical of actual usage of the product.

B.2.2.2 Pipes

Pipes up to 100mm diameter are usually tested under the "worst-case" S/V ratio, i.e. the test is carried by filling a pipe with the test water, using the narrowest diameter which is to be used in contact with drinking water. Contact times of the three stagnation periods are also taken as the "worst-case" conditions and, therefore, no conversion is required.

B.2.2.3 Reservoirs, tanks, etc.

For many other products, such as large diameter pipes, coatings, sealants, filtration membranes, etc., the conditions of the test may differ widely from those in practical use. Reservoir coatings, sealants and similar products are used in situations where the contact time (or residence time) between the product and the water is generally of the order of a few days. The concentrations of leaching contaminants determined in the test are, therefore, assumed to reflect the actual exposure concentrations, after allowance for the different S/V ratio in the two situations.

B.3 EXPERIMENTAL LEACHING DATA NOT AVAILABLE

As a general rule, for contaminants that are likely to leach into water, analytical methods are needed which are capable of detecting ~1µg l^{-1} in the test water. The potential contaminants could be residual monomers and oligomers, antioxidants, stabilisers, diluents, reactions by-products, etc., i.e. substances which are not regulated by the Water Supply (Water Quality) Regulations, are not measured in water as a matter of routine, and for which suitable analytical methods may not be available.

It is not always possible, within a reasonable time, to develop a suitable analytical method, because either:

- The required limit of detection cannot be achieved
- An analytical standard, i.e. a pure sample of the contaminant, is not available (e.g. for reaction products)
- Attempts to measure the contaminant in water have failed (e.g. it cannot be extracted from water, does not give any response to GC or HPLC detectors, cannot easily be derivatised to give compounds which can be analysed etc.)

Under certain circumstances, depending on the nature of the contaminant, the product and conditions of its use, it can be shown, without providing experimental data, that the substance would not pose a risk to consumers.

”Worst-case” calculations can help in the assessment of products, provided the assumptions made are stated and justified and the limitations of the calculation are realised. Examples of the principles involved in such calculations are given below.

B.3.1 Analytical limit of detection too high

The leachates should still be analysed for the contaminant using the available analytical method. If no concentration is detected, the estimated limit of detection of the analytical method is then taken as the “worst-case” concentration and the exposure concentration is then calculated allowing for differences in S/V and contact time as outlined in Annex B2.

The results would need to show that the concentration at the tap would be negligible, even when calculated using the assumed “worst case” concentration in the leaching test. As a general guidance, the calculated concentration would need to be below 1µg l^{-1}, assuming 24 hours contact time. This approach would be unlikely to be acceptable for small diameter pipes and other products where the test and field conditions are similar.

Alternatively, appropriate toxicological evidence would need to be provided to show that the calculated “worst-case” exposure concentration would not pose a health risk to the consumer.

B.3.2 No analytical method available

On occasions it proves very difficult to set up an analytical method that would allow specific determination of the substance in water at all. The “worst-case” calculation then needs to be based on indirect measurements. Depending on the substance, these could be group determinations in the leachates, such as, total organic carbon (TOC), total organic nitrogen (TON), total phenols, etc., or the determination of metals for organo-metallic substances, or, in exceptional cases, maximum leaching rates from similar materials could also be used. In these circumstances the DWI will expect the test laboratory to provide details of the test methods considered or evaluated and the reason(s) for their rejection.

B.3.2.1 Group determinations

Total organic carbon (TOC) will detect most organic substances in water, though only at relatively high concentrations (approximately 400 to 800µg l^{-1}). As a rough estimate it is assumed that organic substances contain 50% of organic carbon (for a specific contaminant it can be calculated accurately). Therefore, "worst-case" exposure concentrations would be calculated in a usual manner using twice the TOC concentration in the leachate (after correcting for a blank). This could well be an over estimate as the TOC value determined would also include any other organic contaminant present in the leachate. When no TOC increase is detected in the leachate, the concentration of twice that of the limit of detection (usually about 100µg C l^{-1}) would be used for the calculations.

A similar approach can be used for total organic nitrogen (TON) or the determinations of metals, taking into account the levels and variations in procedural blanks as well as the nitrogen or the metal content of the substance.

When using more specific analytical methods for group determinations, such as those for total phenols or aromatic amines, experimental evidence would need to be provided on the sensitivity of the method for the particular substance of interest. Any concentration determined in the leachate would be taken as arising solely from the substance.

B.3.2.2 Comparison with similar materials

There are considerable variations in leaching rates between products. However, if the highest observed rate for a similar material is used for the calculation of the "worst-case" exposure concentration and the results are insignificant (see Annex B2.1), it may provide sufficient reassurance to satisfy the DWI without leaching tests.

Thermoplastic materials – e.g. polyethylene, polypropylene, polybutylene, polyacetals, polyamides; polyesters, nylon, etc. Leaching of monomers, additives and other contaminants from these materials is not usually very extensive; measured concentrations of contaminants in leachate after the first leaching period using an S/V ratio of ~1cm^2 ml^{-1} seldom exceed 100µg l^{-1}.

Thermosetting materials – e.g. glass-reinforced polyesters (GRP), epoxy resins, polyurethanes, and polymer-modified cementitious coatings. Leaching rates are dependent, to a major extent, on the conditions of curing or setting; measured concentrations of contaminants in the first leachate after curing, using an S/V of lcm^{-1} may be up to 10mg l^{-1}, with a "worst case" of about 100mg l^{-1} day^{-1}.

All of the above approaches could have a large margin of error. The assessment using `worst-case' calculations based on indirect measurement would therefore be suitable only for products with very small actual S/V ratio or short contact time, or for substances for which comprehensive toxicological data indicate that relatively high exposure concentrations would not pose a health risk – see **Annex C**.

B.3.2.3 Minor components/ingredients

When the total amount of a potential contaminant in the product is minute compared to the volume of water passing through or over the product it can be assumed that either:

- It would be completely leached out of the product over a relatively short time, e.g. less than one month, if the leaching rates are high
- The exposure concentrations would be negligible (though present over longer periods), if the leaching rates are low

The maximum exposure concentration of the contaminant that can be produced at the tap can be estimated assuming that all of the contaminant present leaches out into water over a

short defined period, such as the first week of usage. This can be calculated by dividing the total amount (µg) of the contaminant' by the minimal total volume of water (l) in contact with the product over that period. The assumption will usually be a considerable exaggeration of the short term leaching rate of an ingredient such as an anti-oxidant or stabiliser that is designed for long-term incorporation into a product. However it is probably less exaggeration for residual by-products or traces of processing aids.

Alternatively, the length of time (days) it would take to leach all of the contaminant from the product, assuming a certain exposure concentration, can be estimated by dividing the total amount of the contaminant (µg) by the minimum volume of water (l) in contact with the product over 24 hours, and by the assumed concentration (e.g. the minimum concentration of interest, such as 1 µg l^{-1}). The value of this concentration will vary for different substances depending on their toxicity and other properties, such as odour or flavour.

If these “worst-case” calculations show that any exposure to the contaminant would be negligible, no experimental leaching data would be needed. This approach is likely to be applicable to products such as filtration units or other small parts in contact with large volumes of water, such as reservoir repair materials, which nevertheless are still considered under the full requirements of the relevant regulations.

B.4 SUMMARY

- When considering a product for approval for use in contact with water supplies, the DWI need realistic estimates of the likely “worst-case” exposure of consumers to contaminants leaching from the product into water.
- Usually the exposure concentrations are estimated from laboratory leaching tests, but the exposure concentrations need to take into account the differences in S/V ratios and contact times during the test and in practice – see Test Protocol 2 and BS EN 12873, parts 1 and 2.
- Experimental leaching data for specific contaminants that might leach into water is preferable, wherever possible. However estimations of the exposure concentrations from non-specific group determinations, or by calculations, may suffice under certain circumstances. TOC can provide a useful check on the maximum leaching rates where a more specific method is not available.
- Experimental data is not needed for minor contaminants where “worst-case” calculations can show that the total content of the contaminant in the product would give rise to negligible exposures under the conditions of actual use.
- Test reports should contain full details of the derivation of any estimation of “worst-case” leaching from products.
- Where appropriate, a risk assessment may also be carried out (see Annex **C)** and submitted to the DWI for further consideration.

Note : This Annex is based upon WRc Report “Estimation of “Worst-case” Leaching from Products” by Fielding, Hegarty & Wilson, 1995, prepared for the Drinking Water Inspectorate.

ANNEX C : RISK ASSESSMENTS

C.1 REFERENCE SOURCES

Risk assessments should always be done with sight of original documentation unless for particular reasons this is not feasible, and this documentation should be available for submission to the DWI.

Acceptable reasons for absence of documentation are that it is not accessible either because it is "commercial in confidence" material from another supplier or has been published in a language and journal that means that it is not easily obtainable. In such cases it would be reasonable to cite and provide a study summary that had been prepared by an independent reputable body that had had sight of the 'in confidence' material or a translation of the obscure text. Examples of such bodies are the:

- United Kingdom's Committee on Toxicity of Chemicals in Food
- Consumer Products and the Environment or its sister committees on Mutagenicity and Carcinogenicity
- Joint FAO/WHO Committee on Food Additives (JECFA)
- toxicological committees either of the Organisation for Economic Co-operation and Development (OECD) or of the European Commission (e.g. the former Scientific Committee on Food).

It would also be perfectly satisfactory to cite, use and provide the outcome of an entire risk assessment by such a body, provided that the exposure section was appropriate, i.e. considered ingestion exposure, as this is most likely to be relevant to the DWI.

C.2 ABSENCE OF INFORMATION

It is accepted that for many of the compounds found in the leachate analyses submitted to the DWI there are few relevant toxicological, clinical or epidemiological studies, particularly ones that would be considered of a reasonable standard for a risk assessment. In these circumstances it is the exposure data that become of great importance. The DWI would consider well-reported experimental studies supporting an upper limit for potential human exposure. If appropriate the DWI would consider an argument, by structural analogy, to a "closely" related compound if there were available data on the hazard of the analogue compound but not for the compound identified in the leachate.

In all cases, it should be considered appropriate to provide a properly referenced document supported by copies of the originals of quoted articles. It will certainly accelerate the DWI's consideration as it will mean that the risk assessment can be considered in the light of the cited papers.

C.3 RISK CHARACTERISATION

Risk characterisation, the final step of the scientific part of the risk assessment process (i.e. excluding risk management and some of risk communication), has been considered in a

document published in the September 2003 issue of Food and Chemical Toxicology [4] - although this is food related the general principles are equally applicable to water related uses.

[4] Renwick,A.G.; Barlow,S.M.; Hertz-Picciotto,I.; Boobis,A.R.; Dybing, E.; Edler,L.; Eisenbrand,G.; Greig,J.B.; Kleiner,J.; Lambe,J.; Müller,D.J.; Smith,M.R.; Tritscher,A.; Tuijtelaars,S.; van den Brandt,P.A.; Walker,R.; Kroes,R. (2003) Risk characterisation of chemicals in food and diet, *Food Chem. Toxicol.* **41**(9) 1211-1271

ANNEX D : REQUIREMENTS FOR OTHER REPORTS, CERTIFICATES OF ANALYSIS AND CONFORMITY

D.1 INTRODUCTION

The earlier part of this test protocol is primarily concerned with the reporting requirements to be used for the results from leachate studies carried out at the request of the DWI. On many occasions, however, applicants include other test reports and Certificates of Analysis in support of their submission for approval under the relevant regulations.

The following advice and guidelines has been provided to help both Applicants and the DWI to avoid rejection of these supporting reports and certificates. Reports and Certificates which do not include all the relevant information set out below may not be acceptable to the DWI.

D.2 BASIC REQUIREMENTS FOR TEST REPORTS

To enable the DWI to consider these they must contain the following minimum information. If the laboratory or organization is accredited (eg ISO 9001, ISO 17025) this should be stated in a covering letter if it is not apparent from the report.

D.2.1 Executive summary

Test reports shall include a separate executive summary, in plain English, covering the following issues:

- The product tested
- Reference to the test methods used
- The outcome of the testing
- Any specific issues or results that are likely to be of concern
- Description and justification of deviations from prescribed methods and explanation of any unusual results

D.2.2 Test Sample/Specimen details

These shall include any test requirements specified in the test method protocols used. Aspects covered shall include, as a minimum -

- Product name
- Manufacturer
- Location of production facility
- Product code and batch code ("date of manufacture" would be a suitable acceptable alternative, if batch code is not available)
- Description of the test water
- Description of test sample including dimensions and/or methods of preparation, together with the volume of test water (if appropriate)
- Product surface area exposed to test water (if appropriate)
- Surface area to volume (S/V) ratio expressed as dm^{-1} (if appropriate)
- A chain of custody if test samples have been prepared by a third party or off-site

D.2.3 Analytical sample preparation

This shall include, as a minimum -

- Summary of method used with full details of test conditions and leaching periods (if appropriate)
- A reference to the test method used
- Deviations from the prescribed procedure and reasons for any deviations.
- Source and methods of purification of test water
- Date and time of collection of the test solution/leachate

D.2.4 Analysis

These shall include, as a minimum -

- Summary description of analytical methods
- References to methods used
- Statement of the performance characteristics (e.g. limits of detection, trueness and recovery) with supporting data
- Deviations from the prescribed procedure and reasons for any deviations
- Date and time of analysis

D.2.5 Test results

The test report shall include:

- Results in tabular form – reported in accordance with any specific requirements of the test protocol used
- Tabular and graphical plots of leaching concentrations (µg/l) and migration rates (µg/dm²/d) against time (h) if appropriate
- Information on the level of confidence in the measurements presented, including details of the performance characteristics of methods of analysis (more detail in annex E)
- A brief statement about whether the results have been adjusted in any way e.g. blank correction
- Details of any unanticipated event during the test procedure

D.2.5.1 Raw Data

If raw data appendices are supplied these should include the following:

- Raw data (chromatograms, spectra etc.) - these should be clearly labelled to identify the test product, the leachate and the test water type etc. as appropriate
- Details of procedural blanks and controls used with information on whether analytical results have been blank corrected
- AQC results from the batch and the criteria for acceptance of test results.
- Adequate means of cross referencing between the discussion/reporting sections and the actual peaks in spectra and chromatograms etc

D.3 CERTIFICATES OF ANALYSIS AND CERTIFICATES OF CONFORMITY (Test/Approval Certificates)

These can only be considered by the DWI if supported by appropriate test reports providing the minimum information set out in Annex D.2 (above) of this Test Protocol.

ANNEX E : CONTROLLING AND REPORTING UNCERTAINTIES IN GC-MS GENERAL SURVEY ANALYSIS

E.1 INTRODUCTION

The estimation of the concentration of compounds in leachates as described in BS 6920 part 4 is based upon the following assumptions:

- All compounds have the same response on a TIC chromatogram
- A compound has the same recovery efficiency as the internal standard it is measured against
- The chromatographic process collects the total amount of the compound into a single peak. A chromatographic peak may or may not be a single compound
- All the ions detected in the MS have a charge of 1

All of these conditions are very broad approximations and as a result there maybe widely different responses, which can be expressed as a recovery or a bias of the analytical process.

It is evident from experience over the years that the internal standards have different responses, with phenol being exceptionally poor. Consequently the concentration reported in the leachate will depend on the internal standard chosen for comparison. It also follows that if a recovery is measured from injecting spiked solutions, it will relate to a specific standard. There maybe no logical way of choosing the appropriate internal standard to give the most accurate result.

The internal standards are various hydrocarbons, with the exception of phenol and chlorobenzene. Most of the chemicals of concern have functional groups which may, like phenol, have varying effects on the analytical response. Physical properties like volatility and partition coefficient with the extractant and the stationary phase are also important determinants of the systematic error.

E.2 SOURCES OF ANALYTICAL UNCERTAINTY

In addition to variances from sampling, storage, pipetting etc. the instrumental procedure introduces variances from:

a. **Extraction:**
   Unless the partition coefficient is very favourable to full extraction under the conditions of the method, there will be a significant random error introduced at this stage. If the coefficient is unfavourable the leached material is unlikely to be detected.
b. **Recovery:**
   The largest source of uncertainty is likely to be the analytical recovery of the compound. This can only be estimated by injecting a pure standard of the identified compound, which is impractical for routine work. Experience indicates that recovery may be as low as 10% (or zero if not detected). In theory it could exceed 100% (higher recovery than the internal standard); most often it is much less than 100%. Part of this error is systematic, as described above, but there also seems to be a significant random element.
c. **Internal standard response:**
   This is readily calculated for any run and the standard deviation calculated from a series of runs in the usual way. It is advisable to maintain a Shewhart control chart of the IS response; either the area or the concentration per unit area.

d. **Between batch:**
If a standard or spike of the determinand is injected for comparison there is an additional inter-batch variance. This is often minimized by adjusting the result by the ratio of the response from an internal standard in both samples.
e. **Purity of chromatographic peak:**

E.3 REPORTING THE UNCERTAINTY IN RESULTS

In trying to quantify the leached compounds there are two situations. Either a reasonable match has been found or the compound remains unknown.

E.3.1 Library match

- Estimate the concentration from the five internal standards used for quantification.
- If a standard or spiked solution of the compound has been run and the recovery efficiency is known, report the result as well as the above,.
- Report the library and leachate MS scans on the same page at the same scale
- Report the purity of fit of the closest compound(s)
- If knowledge is known about the recovery efficiency of the compound (or similar substances), report it.
- Calculate the combined uncertainty from the standard deviation of the internal standards and all other uncertainties expected.
- Report the concentration, uncertainty and recovery (best guess)

E.3.2 Unknown

- Estimate the concentration from the five internal standards used for quantification.
- Report the retention time and the four largest MS peaks in order of size (m/z)
- Report any conclusions that can be made regarding the molecular weight, functional groups, hetero-atoms etc.
- Report library matches with a purity of fit greater than 50% up to a maximum of three.
- Make an estimate of the analytical recovery
- Calculate the combined uncertainty from the standard deviation of the internal standards and all other uncertainties expected for the compound.
- Report the concentration, uncertainty and recovery (best guess)

E.4 SUMMARY

It follows that all semi-quantitative results should be reported as four elements:

- Either the range of estimates of concentration (uncorrected) from comparison with the five internal standards,
  Or the estimate (uncorrected) based on a standard of the pure compound.
- The combined uncertainty (without expansion by a coverage factor).
- The bias (it is acceptable to write “unknown” if there is no information).
- The recovery (it is acceptable to put in a guess based on what is known about the determinand, but it should be marked as such).

Qualitative results should be reported as two elements:

- Either the identity of the compound and the closest match(es) from the library,
  Or the four largest ions from the MS spectrum and any possible matches from the library search
- The purity of fit metrics for the compounds reported

There may, in addition, be a commentary on any characteristics of an unknown compound that can be inferred from the analytical results e.g. possible molecular weight, possible functional groups, the type and number of heteroatoms that may be present.

Test Standards

BS 6920-4:2001	Suitability of non-metallic products for use in contact with water intended for human consumption with regard to their effect on the quality of the water — Part 4: Method for the GCMS identification of water leachable organic substances
BS EN 12873-1:2003	Influence of materials on water intended for human consumption – Influence due to migration – Part 1: Test method for non-metallic and non-cementitious factory made products
BS EN 12873-2:2005	Influence of materials on water intended for human consumption – Influence due to migration – Part 2: Test method for non-metallic and non-cementitious site-applied materials