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IMPROVED MATERIAL TESTING PROCEDURES

Final Report to the Department of the Environment

DWI 3899/2
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Authors: I M Wilson

Contract Manager: I M Wilson

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Any enquiries relating to this report should be referred to the Contract Manager at the following address:

WRc plc, Henley Road, Medmenham, Marlow, Buckinghamshire SL7 2HD.
Telephone: 01491 571 531

EXECUTIVE SUMMARY

The Drinking Water Inspectorate operates on behalf of the Secretary of State for the Environment and the Secretary of State for Wales a statutory approval scheme for materials used in contact with water supplies and chemicals used in water treatment. A number of EU and other countries operate approval schemes which differ in a number of aspects from the UK scheme and from each other. This means that suppliers often need to submit their products to similar testing and approval procedures in each country in which they wish to sell their products.

The Construction Products Directive (89/106/EEC) (CPD) was introduced to ensure an open market. It specifies that water supply products must satisfy Essential Requirements (ER), including a requirement for no adverse effect on the quality of water supplies. The European Committee for Standardisation (CEN) is drafting standards for testing to demonstrate compliance with the ER. The ER apply to construction works and not to its individual components. The product standards for components are required to ensure that the finished works will meet the ER. The intention is that the European standards will, eventually, replace national standards.

This contract was established in September 1990 to provide information on current approval schemes in other countries and on the developments within CEN, and to provide experimental data and technical advice to enable the adoption of the most appropriate UK and CEN test procedures and approval schemes.

The major approval schemes for materials used in contact with drinking water operated in Europe and the USA have been assessed and an Inventory of European Approval Systems has been published.

WRc has provided technical advice on the drafting of CEN standards for testing the effects of materials on drinking water including participation in drafting meetings and preparation of discussion documents.

WRc also participated in two interlaboratory studies organised by CEN, i.e. an extensive validation study on a leaching test procedure for factory-made products (TC164/WG3/AHG2), and a smaller study on the determination of acrylamide monomer in polyacrylamides (TC164/WG9/TG8).

For products which are applied on site, the available data from laboratory and field leaching tests has been collated and additional laboratory studies have been performed to assess the effects of various test conditions on the results. Dynamic tests on *in situ* lined pipes under controlled conditions have been proposed to provide realistic and reliable information.

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Investigations have been undertaken on the effects of test conditions on the sensitivity and accuracy of the test for microbiological growth (BS6920 Section 2.4). The results have demonstrated that reducing the head space in the test vessel and increasing the ratio of surface area of the test sample to the volume of the test water produced the greatest improvement in sensitivity without apparent loss of accuracy.

Developments in CEN, and also in the UK, have uncovered many new problems including the conversion of test results to field situations, effects of disinfection of new installations on leaching, testing of site-applied products, microbiological tests and wider aspects of approval criteria and the operation of a harmonised European approval system. The work under this contract has contributed to solving some of these problems. EC-supported co-normative research should deal with some others, but a large amount of work is still needed before the European system or, as an alternative, mutual acceptance of national approvals can be used in practice.

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1. BACKGROUND

Section 53 of the Water Act 1989 (now consolidated in Section 69 of the Water Industry Act 1991), gave the Secretary of State powers to make regulations controlling processes, substances and products used in the treatment and provision of public water supplies. Regulation 25 of the Water Supply (Water Quality) Regulations 1989, which came into force on 1 September 1989, and were amended by the Water Supply (Water Quality) Amendment Regulations 1989 and the Amendment Regulations 1991, concern the introduction of substances and products for use in the treatment and provision of public water supplies.

Consequently, apart from certain exceptions as set out in Regulation 25, approval by the Secretary of State is a statutory requirement for products used in public water supply. The Committee on Chemicals and Materials of Construction for use in Public Water Supply and Swimming Pools (CCM) advises the Secretary of State on approval matters. The Drinking Water Inspectorate (DWI) provides the Secretariat and chairs the Committee.

When considering an application for approval the Committee must be satisfied that use of the substance or product is unobjectionable on health grounds and unlikely to give rise to any other adverse effect on water quality. Data from laboratory and field leaching tests form an important part of the supporting scientific information on which the assessment is made. It is therefore important that, for a range of different products and usages, appropriate test conditions are devised, validated and well documented and that the quality and quantity of the data obtained from the tests are adequate to allow realistic assessment.

A number of EU and other countries operate approval schemes which differ in a number of aspects from the UK scheme and from each other. There are differences in test conditions, parameters which are measured, assessment of the test results, products covered by the schemes, nature of approval bodies, and other aspects. Consequently, an approval in one country usually does not enable the product to be used in another country.

The Construction Products Directive (89/106/EEC) (CPD) was introduced to ensure an open market. It specifies that water supply products must satisfy Essential Requirements (ER), including a requirement for no adverse effect on the quality of water supplies. The European Committee for Standardisation (CEN) is drafting standards for testing to demonstrate compliance with the ER. The ER apply to construction works and not to its individual components. The product standards for components are required to ensure that the finished works will meet the ER. The intention is that the European standards will, eventually, replace national standards.

Working Group 3 of the Technical Committee 164 (Water Supply) (TC164/WG3) and its ad hoc groups are preparing standards relating to the effects of materials on water quality. Working Group 9 of TC164 and its task groups are drafting standards for chemicals in water treatment. Other CEN groups are also developing standards which are of relevance to water supply. It has been recognised that any proposed improvements in testing and assessment procedures in the UK need to be synchronised with developments in CEN.

The Department of the Environment has supported experimental work on developing and validating test procedures as well as other activities providing information necessary to promote procedures for material testing and approval, which would be acceptable both in the UK and in other EU countries.

2. OBJECTIVES AND PROGRAMME OF WORK

2.1 Objectives

The objectives of the project specified by the Department of the Environment were:

1. To ensure the development of the best possible EU approval schemes.

2. To review implications of CEN/EC developments and implementations for UK materials' testing and approval.

3. To establish testing needs, devise, validate and recommend modified testing procedures.

4. To advise on action needed to maintain the use of existing substances/products and to advise on checks needed on imported products.

5. To participate in collaborative tests organised by CEN, as required and agreed with the Department of the Environment.

2.2 Programme of work

The programme of work has evolved during the course of the project, responding to fresh needs arising from developments in the testing and approval of materials for use in contact with drinking water both in the UK and in Europe. Different areas of work were covered in parallel throughout the duration of the project. The resulting programme is described under headings representing the areas of work and therefore is not necessarily in chronological order.

The specific areas of work were:

1. Technical advice in the development of CEN standards concerning the assessment of the effects of materials on water quality.

2. Participation in tests arising from CEN activities.

3. Critical appraisal of EU/EFTA testing and approval schemes for products for use in contact with drinking water.

4. Development and validation of testing procedures.

3. CONTRACT ARRANGEMENTS

The initial two-year contract, which started in September 1990, was extended in April 1993 to continue until 31 March 1995. The total cost of the contract is some £255K. The distribution between the financial years is shown in Table 3.1.

Table 3.1 Out-turn of the contract

Financial year	Cost (£)
1990/91	14 018
1991/92	64 712
1992/93	63 367
1993/94	44 674
1994/95	68 000*
Total	254 771*

* Estimated out-turn

4. SUMMARY OF ACTIVITIES AND RESEARCH RESULTS

4.1 Technical advice on the development of CEN standards

Within TC164/WG3 (effect of material on water quality), five ad hoc groups have been set up:

- AHG1 Organoleptic assessment
- AHG2 Migration from non-metallic products
- AHG3 Microbiological assessment
- AHG4 Positive lists
- AHG5 Metallic products

The technical advice supported by this contract was concerned with the work of AHG2 and AHG4 and included participation at the meetings and preparation of reports on the meetings for the Department, commenting on relevant CEN documents and preparation of supporting documents. The developments within AHGs 2 and 4 are described in the six-monthly **Progress reports to the Department (Section 7.1)**.

From January 1991 WRc have been involved with the following topics:

4.1.1 Leaching tests for factory-made products

Initially, AHG2 drafted a test procedure intended to be used as a general leaching (migration) test for all non-metallic products. Later, in response to a UK suggestion, it was agreed to limit the basic procedure to the testing of factory-made products such as plastic pipes and fittings, products made of rubber and coatings and to draft separate, more appropriate, procedures for testing other types of products, such as site-applied products, ion exchange resins, etc.

Before TC164/WG3 and its ad hoc groups had been established CEN TC155 'Plastic pipes systems' set up a working group, TC155/WG2, to develop the harmonised test methods it needed to assess effects on water quality. The CEN Technical Board (BT Committee) accepted the overlapping programmes put forward by TC 164 and TC 155 but requested co-ordination between the two Technical Committees and their Working Groups.

TC155/WG2 set about developing a method for assessing migration from plastic pipes and fittings etc., and TC164/WG3/AHG2 then started to draft methods for migration tests from non-metallic products in general, including plastic pipes, etc. There was a need for a close collaboration by the convenors involved to avoid unnecessary duplication. Even though the convenors were from the same organisation the required collaboration was ineffective in the case of the method already underway in TC155/WG2.

The **DoE Report 3304, February 1993,** reviewed the differences between the test procedure drafted by TC164/WG3/AHG2 and the prEN 852-1, 'Test method for migration from plastic pipes', drafted by TC155/WG2 and discussed the likely effects of the discrepancies on the test results. Since then there have been further modifications of both drafts, which removed most of the differences.

With other standards, where TC155/WG2 had not already made a start there has been reasonable collaboration (e.g. Organoleptic Assessment). Although the existence of the overlapping programmes is basically flawed, it is leading to a sharing of tasks, which helps with the excessive workload.

During the first half of 1992 the leaching procedure drafted by AHG2 was tested in an interlaboratory study (Section 4.2). Since then there have been several re-draftings of the text, though the test conditions remained virtually the same.

Recently, serious disagreements have emerged in defining the basic principles of the migration tests. From the first draft the test method was based on three consecutive (72-hours) stagnation periods. This principle is now being questioned, following discussion both within the AHG2 and TC155/WG2, on the interpretation of the test results in relation to long-term exposure, conversion factors, etc. The problem has been referred to TC164/WG3.

4.1.2 Leaching tests for site-applied products

Site-applied products differ from factory-made and factory-applied products in two main aspects:

a) they are usually brought in contact with water supplies sooner, often immediately the recommended curing/drying period is completed; and

b) conditions of the application are more difficult to control (e.g. temperature, preparation of the surface).

TC164/WG3 and its AHG2 have recognised that these aspects need to be reflected in the conditions of the leaching tests in order to obtain realistic estimates of the potential exposure of consumers to contaminants arising from such products.

The UK has probably more experience than any other country in assessing the leaching from such products and has, therefore, had a major input in drafting the test procedures and providing valuable supporting data when considering the appropriate test conditions (Section 4.4).

At present AHG2 is seeking from EU countries further information on the type of site-applied products, methods of their application and use and experimental data on leaching under various test conditions.

4.1.3 Testing of cementitious products

The testing of cementitious products was to be included in the standard tests for factory-made or site-applied products, as appropriate. However, leaching from such products is markedly affected by the nature of the water, particularly its aggressivity. It has become apparent that there was no consensus regarding the nature of the test water. In addition, there was little background information on the effects of pre-treatment and conditioning of the test sample on the tests results, and their relevance to field conditions.

The drafting of a standard test for cementitious products was therefore put on a standstill. Test conditions for cementitious products, together with those for metallic products (where the leaching is also strongly affected by the nature of the test water), are now a subject of co-normative research undertaken jointly by CRECEP, and LHRSP in France, KIWA in The Netherlands, DVGW in Germany and WRc in the UK. The three-year project, after administrative delays, is in its early stages, finalising work plans and project management.

4.1.4 Testing of membranes and membrane filters

In France membranes and membrane filters are tested under dynamic conditions. TC164/WG3 has agreed that AHG2 should prepare a separate leaching test procedure for this type of products. However, until then, they are to be tested in accordance with the basic procedure for factory-made products.

4.1.5 Testing of ion exchange resins

There are already several leaching test procedures for ion exchange resins, such as a DVGW standard method in Germany and the procedure specified in Annex H of the UK DWI Guidance Note on the Approval of Substances and Products. A report reviewing and comparing the existing methods is being prepared in Germany.

4.1.6 Conversion factors for results from leaching tests

The results from the laboratory leaching tests need to be adjusted, using conversion factors (that depend on the product and its use), to estimate the actual concentration in field use and thus, the potential exposure of the consumer to the contaminant. A number of conversion factors, derived from those used in the existing national approval schemes, has been proposed and the options are being discussed within CEN.

The concept of the conversion factors is primarily being developed for pipes by TC155/WG2. TC164/WG3/AHG2 is following the developments and will, when the basic concept is agreed, extend it to other factory-made products and modify it for other leaching tests as appropriate.

A report has been prepared for the Department, reviewing the situation, in CEN, on conversion factors at the end of 1993 (**WRc Report DoE 3642, March 1994**). The report also reviews wider aspects of the assessment of test results as practised by the main national approval schemes in Europe and the USA, including the basis on which acceptable concentrations of the contaminants are set.

4.1.7 Positive lists and other approval criteria

Approval systems in a number of European countries operate positive lists, which specify the chemicals, such as monomers and additives, which may be used in the manufacture of materials that are to be in contact with drinking water. CEN TC164/WG3/AHG4 has compiled an inventory of existing positive lists, for polyethylene, polyvinylchloride and rubber, based on lists existing in Germany, The Netherlands and, to a lesser extent, Italy and Denmark. For some of the chemicals the list also includes their permissible concentrations in the material and migration limits.

In addition, AHG4 has prepared a position paper on future European approval criteria, which was presented by the Group's convenor at the CEN/EUREAU Seminar on 'Materials and Chemicals in Contact with Drinking Water', which was held in Vienna in March 1994. At its last meeting, in June 1994, the Group identified and discussed areas of work essential for the development of a European approval scheme which were not covered either by the current CEN structure or by the European Commission, in particular the following:

- defining and establishing Positive List(s);
- specifications of testing requirements for type, product and audit testing for different categories of products, e.g. where to use screening tests, microbiological tests, determination of specific contaminants, general scan gas chromatography-mass spectrometry (GC-MS) etc.;
- specifications on analytical performance and quality control appropriate to leaching tests;
- drafting standard analytical methods where needed, such as group determinands or general scan GC-MS; and
- defining procedures for assessing the results of general scan GC-MS analysis and new products containing chemicals not listed in Positive Lists.

These, and other problems have been referred to TC164/WG3, the Commission and national regulatory bodies.

As a part of this contract the UK delegates have prepared a paper on the benefits of using general scan GC-MS to assess leaching potential of materials (**WRc Report DoE 3314, February 1993**).

4.2 Interlaboratory tests for CEN

WRc has participated in two CEN interlaboratory studies as a part of this contract. One was organised by TC164/WG3/AHG2 to assess and validate the Group's proposed standard procedure for leaching tests for factory-made products. The other was organised by TC164/WG9/TG8 to evaluate proposed standard methods for the determination of acrylamide monomer in polyacrylamides used as coagulants in water treatment.

4.2.1 Leaching tests for factory-made products

Eight European testing laboratories participated in the interlaboratory study:

- CRECEP, Centre de Recherche et de controle de Eaux, Paris, France;
- CRPAM, Centre de Recherche de Pont-à-Mousson, Pont-à-Mousson, France;
- LHRSP, Laboratoire Hygiene Recherche Sante Publique, Vandoluvre les Nancy, France;
- DTI, Danish Teknologisk Institut, Aarhus, Denmark;
- DVGW, Forschungsstelle am Engler-Bunte-Institut, Karlsruhe, Germany;
- Kiwa NV, Research Division, Nieuwegein, The Netherlands;
- VTT, Technical Research Centre of Finland, Espoo, Finland;
- WRc Medmenham, UK.

The following products were tested:

- epoxy resin coating;
- grey PVC-U pipe, 32 mm diameter;
- grey PVC-U pipe, 75 mm diameter;
- glass-reinforced polyester (GRP) pipe; and
- EPDM rubber.

The tests were performed in accordance with the relevant conditions of the draft leaching test method 'Water supply - Influence of materials on water quality; Part 2: Influence due to migration' sixth draft, November 1991, prepared by the AHG2 of TC164/WG3. Leachates from all the tested products were submitted for the determination of total organic carbon (TOC) and general scan GC-MS analysis, as well as one or more determinands, specific to each material, which were known to be leaching into water. The GC-MS analysis was included at WRc's request as it is a standard requirement for UK approvals. WRc supplied the analytical protocol and standard solutions of deuterated internal standards for spiking the test samples. The products were tested with unchlorinated test water, under conditions relating to cold water applications.

Testing took place between January and April 1992. The test methods and the analytical determinands were described in the **Progress Report DoE 3004, January 1992** and details of the analytical methods used were given in the **Progress Report DoE 3302,**

January 1993. WRc results for the specific determinands were reported in the **Progress Report DoE 3154, July 1992** and a full WRc test report was included in the **Appendix A of the Progress Report DoE 3302, January 1993.**

Results from the participating laboratories were tabulated by the convenor of AHG2 and later statistically evaluated by Kiwa NV. Results for the specific determinands and TOC showed a fair degree of agreement but also some major differences. Since the study was time consuming and costly, many laboratories could not follow the study plan in all details, particularly they were not be able to provide sufficient data for spiked samples and analytical controls. It was therefore difficult to identify with any certainty, whether the differences were due to inconsistent calculation and reporting of the results, analytical errors, test conditions or variation in leaching characteristics between the test samples. The contract enabled WRc to produce the most complete set of data of all the participants.

Only five of the laboratories were able to undertake the general scan GC-MS analysis. The complexity of the compounds detected was similar for all the products tested, increasing in the order from PVC-U pipes (no peaks), epoxy resin coating, and GRP pipe to EPDM rubber (multitude of unidentified compounds). However, the results and their interpretations were inconsistent, partly because some laboratories did not fully follow the protocol and because the method was not described in sufficient details and did not include appropriate quality controls.

4.2.2 Determination of acrylamide monomer in polyacrylamides

During the period January to July 1992, WRc analysed samples of polyacrylamides, distributed to the participants by TC164/WG9/TG8. The analytical methods were based, in accordance with a draft (prEN) standard method, on extracting the samples with organic solvents followed by the determination of acrylamide monomer in the extracts by High Performance Liquid Chromatography (HPLC). Three types of polyacrylamide coagulants were analysed, i.e. anionic, non-ionic and cationic. Anionic and non-ionic samples were extracted with 80% methanol/water solvent. Cationic samples were extracted with 80% acetone/water solvent. The extractions were performed in triplicate and the extracts analysed by HPLC five times.

The work has been described in the **WRc Report DoE 3148, 1992.** The report also made suggestions for improving the standard methods. The main improvements related to the filtration method, the criteria for evaluation of the performance and separation efficiency of the HPLC column.

TC164/WG9/TG8 has concluded that the method was suitable and the accuracy of the analytical results, including those from WRc, was within an acceptable range. Most of the suggested improvements in the method have been incorporated into the draft standard.

4.3 Review of EU/EFTA testing and approval schemes for products for use in contact with drinking water

WRc prepared two reviews of current approval systems, one at the beginning of the contract in 1991 and the other was published as an external publication in November 1994 (**Fielding, Rogers and Wilson 1994**).

For the initial review information on major approval systems in The Netherlands, Germany, France and the USA was gathered and evaluated under the following headings:

- organisation;
- information required from applicants;
- the use of positive lists;
- basis of test procedures;
- criteria for assessments; and
- published information and listing.

The systems were compared to that operating in the UK. The findings were reported in **Progress Report 2835-M, June 1991**.

The review found that the UK system was generally more stringent, with the materials having passed a wider range of tests and thorough toxicological assessment by the CCM. The European use of measurements in leachate of total organic carbon (TOC) was recommended as a useful, inexpensive determinand. The European systems also all used positive lists for materials and the use of these is being considered by CEN.

Information gathered for this review has also formed a basis for a review of the main European approval systems, presented at the Vienna Seminar (**Fielding 1994**).

The main objective of the second, more extensive review, was to provide advice to manufacturers and suppliers on how to apply for approvals in EU and EFTA countries. The information was obtained from a WRc questionnaire sent out by the Drinking Water Inspectorate to representatives of the national regulatory bodies, standards organisations, testing laboratories, certification bodies and some manufacturers. Where insufficient or conflicting information was received further details were obtained by follow up correspondence and discussions and from available brochures and documents. The final entry for each country was sent for confirmation to the organisations listed therewith.

The report covers the following main aspects:

- background information on regulatory aspects, organisations involved and existing approval systems, concerned with the effects of materials on water quality, within each country;
- scope of each approval system;
- identity of bodies carrying out testing and issuing approvals/certificates;
- information on testing requirements and the basis for awarding approval; and
- arrangements for recognising approvals issued in other countries.

4.4 Development and validation of testing procedures

The contract included three experimental studies, of which one was concerned with leaching tests for site-applied epoxy resin linings and the other two investigated factors affecting the test for microbiological growth in accordance with BS6920, Section 2.4. A summary of the work undertaken and conclusions reached are given in the two following subsections.

4.4.1 Leaching tests for site-applied epoxy resin linings

For epoxy resin linings more extensive testing is required in the UK than in most of Europe. The present UK CCM assessment method requires data from field trials resembling real situations. Three sections of mains pipe are lined with the resin and water that has flowed through it is analysed for particular resin and hardener components. Experimental evidence was required to support the UK proposal to TC164/WG3/AHG2 for more extensive testing of such products than the standard leaching tests. This has been provided by WRc.

To support the CCM and WRc view that *in situ* application is more prone to variations in product composition and hence in leaching rates, data was obtained on the effects of different resin:hardener ratios on migration rates, measured in laboratory plate tests. The results show considerable variations in concentrations of resin components in the extracts of particular leachates, due to the different resin:hardener ratios. As an extreme, a 10% excess of resin caused a 500% increase in migration of one resin component.

The initial draft CEN TC164/WG3/AHG2 standard for testing site-applied products involved subjecting pipes which have been lined *in situ* to the basic migration tests (three 72-hour stagnation periods) after removal to a laboratory. During the period July 1991 to June 1992, work was done at WRc on a comparison of different testing regimes for two-part epoxy resin mains re-lining systems.

A length of pipe (about 60 m) was lined *in situ* with an epoxy resin by a contractor. Immediately after lining two segments from each end of the pipe were cut off, cured at 10 °C for 16 hours and tested in the laboratory in accordance with the proposed draft CEN procedure. The leachates from the three 72-hours stagnation periods were submitted for analysis to determine the concentrations of TOC, total nitrogen (TN), two compounds originating from the resin part and two from the hardener.

The results were compared with those obtained, for the same epoxy resin, from a laboratory plate test and a field trial undertaken, during the same period, for the resin manufacturer.

The results indicated that laboratory plate tests were inadequate to assess leaching rates as they gave incorrectly high concentrations of hardener components and TOC in the extracts. The results from the laboratory pipe tests were broadly in line with those of the field trial, except for the initial transient high concentration observed with the field trial.

Available data for replicate samples from field trials and laboratory plate tests, together with the results obtained from the laboratory tests on the *in situ* lined pipe were compared for variability of the results.

The variation in results from field trials for the triplicate pipes was larger (50 to 70%) than that from laboratory plate or pipe tests (20 - 50%). It was not possible to identify whether this was due to the application technique, flow control, temperature variation or other factors. Though the field tests provide the most realistic estimates of consumers' exposure, there are also practical problems in using field trials for standard testing.

The study concluded that to provide realistic leaching data for site-applied pipe linings, with reasonable accuracy, the tests should be carried out:

a) on *in situ* lined pipes;

b) under dynamic flow conditions relevant to field situation; and

c) under controlled conditions of curing and test temperature and flow (residence time) in a 'yard' test or on a laboratory test rig.

Details of the study are given in **Progress Reports DoE 3004, January 1992 and DoE 3154, July 1992.**

Some of the results of this study were presented at an International Conference on Coatings and Linings for the European Water Industry in Birmingham in October 1993 (**Wilson 1993**).

4.4.2 Growth of aquatic micro-organisms test (BS6920 Section 2.4)

In the UK products for use in contact with drinking water have to pass a test for microbiological growth in accordance with BS6920:1990, Section 2.4 with additions and amendment given in the documentation of the Water Byelaws Scheme (WBS) Tests of Effect on Water Quality (Instruction No:Admin 7). The standard method has been considered by AHG3 of TC164/WG3, together with microbiological methods developed in The Netherlands and Germany. Initially, the Group agreed that a part of the UK standard, where the extent of growth is measured by determining the Mean Dissolved Oxygen Difference (MDOD), should go forward as a Pr-EN.

However, following an interlaboratory test, the Group came to a conclusion that further research was needed to address problems with certain aspects of the tests. A co-normative research proposal was submitted to the Commission in March 1995 by collaborators in AHG3. They are awaiting a response on the success of their proposal.

A recent study carried out for the American Water Works Association (AWWA) recommended some changes to Section 2.4 of BS6920 to improve the accuracy of the test. Briefly these changes were to increase the head space used in the test vessel, and to standardise the inoculum used in the test. The American study had implications for the acceptance of the specified test conditions in the future European Standard.

The Department of the Environment therefore required an independent study to confirm the American results and asked WRc to investigate the influence of head space on the results obtained from the MDOD test. Two consecutive studies were carried out under this contract. The first study was undertaken in the first half of 1994 and the work is described in **WRc Report DoE 3696/1, 1994.** A follow up study was undertaken during the last quarter of 1994 and the first quarter of 1995 and the work is described in **WRc Report DoE 3892, 1995.**

During the first study, several materials, which would normally be expected to give pass, fail and borderline responses, were tested using a range of head spaces, which included the current BS6920 value of 15% and the AWWA suggestion of 35%.

The results of the study did not confirm the recommendations of the AWWA study, and produced the following conclusions:

1. Increase in head space reduces the sensitivity of the MDOD test.

2. There is no evidence that reproducibility is reduced at 15% head space.

3. Different acceptance criteria are required for different head spaces.

4. Not all materials produce a plateau of DOD, therefore there is a concern over a fixed period for MDOD determination.

5. Water change period affects MDOD result.

The results of the study have demonstrated that the MDOD test in BS6920 Section 2.4 would not be improved by adopting the recommendations of the AWWA study. The conclusions have been taken into account during the recent update of the WBS Tests of Effect on Water Quality and are incorporated in proposed changes to BS6920.

The WRc study, whilst not confirming the results of the AWWA study, has suggested several ways in which the current MDOD method may be modified to improve its sensitivity. Methods being developed in The Netherlands and Germany already have greater sensitivity and, if a more stringent acceptance criteria was to be agreed for the European Standard, then the current UK MDOD test would no longer be appropriate.

The first study suggested the three following ways in which sensitivity could be improved:

1. Reduction of the head space.

2. Increase in the time between water changes (increased contact time).

3. Increase in the ratio of surface area of material to volume of test water.

The Department therefore asked WRc to investigate further the effects of the three proposed modifications in the follow up study.

The research programme of the follow up study involved two phases. In the first phase the three options for improving sensitivity were investigated independently, so that an assessment of their individual impact could be made. In the second phase the most promising combinations of these three options were investigated in an attempt to produce an optimum sensitivity.

The results of this study have demonstrated that the sensitivity of the MDOD test can be improved by reduction of the headspace and by increasing the surface area to volume ratio (S/V) **(WRc Report DoE 3892, 1995)**. Increasing the time between water changes had a less marked effect. A combination of increased S/V ratio and reduced head space had an additive effect on improvement to sensitivity.

5. DISCUSSION

5.1 General

The European Standards drafted by CEN are intended to replace national standards and other established test procedures. It is therefore important that the new test and approval procedures provide satisfactory safeguards to water quality and consumers health without restricting the development of new materials and products and imposing unnecessary testing costs on the manufacturer.

The basic objective of CEN, i.e. to harmonise the various standards in European Member States, is admirable since its achievement would lead to an 'open market' and enable Europe to compete on equal terms with other large homogenous markets such as the USA and Japan. However, the experience of the last few years in the area of assessment of effects on drinking water quality has indicated that there are major problems, some of which threaten this basic objective.

The overall timescale for the production of the required CEN standards and implementation in the UK is much longer than was originally estimated. The replacement of the national standards is many years away, assuming that such a substitution is indeed possible. This is because the effort needed to achieve agreement on technical and non-technical issues has proved very demanding in relation to the available resources. But more fundamental problems exist. In particular, the difficulties of harmonising those aspects of EU test/approval schemes that fall under the regulators authority, i.e. limit values and positive lists, have not been anticipated by CEN. In retrospect, it was probably unwise to proceed too far developing the purely technical aspects of harmonised test methods without achieving a clear understanding of how these essential regulatory-related aspects would be included. Linked to this problems is how CEN harmonisation deals with the various national restrictions, usually based on health considerations, on particular products, materials and chemicals.

Many of the harmonised test methods that are emerging are clearly 'hybrid' methods that are largely untested but there is no obvious CEN plan to support such testing prior to the methods becoming CEN standards that have to be implemented in the UK.

Some of the problems raised jeopardise the basic aim of CEN. Consequently, a rigorous reappraisal of the objectives, targets and progress is needed. It may be that the original objective, a complete harmonised EU approval procedure, is no longer feasible.

Because of the complex task that has been requested of and CEN has set itself, the work programme needs to reflect all the perceived problems associated with developing harmonised standards and this needs to be rigorously monitored and revised in the light of an increasingly better understanding of the difficulties of solving some of the problems and of the underlying timescale.

CEN is run by people and success can only be achieved through genuine collaboration. At times there is evidence of a 'polarisation' of manufacturers and regulators (and their representatives) which is unfortunate. As mentioned already, some problems can only be solved by effective collaboration between convenors of CEN committees with related interests, but there is also evidence that such collaboration is not effective, even when the convenors come from the same organisation.

5.2 Leaching tests for factory-made products

TC164/WG3/AHG2 has progressed furthermost with the test method for factory-made products. In the interlaboratory tests the results for relatively simple determinations, such as TOC or lead, showed less discrepancies than the more difficult determinations of specific organic compounds and the general scan GC-MS. This would indicate that any problems in reproducibility are more likely to be caused by analytical errors than by variations between test samples or by difficulties in controlling test conditions.

WRc, under a separate contract for the Department, has now drafted a more detailed method for the general scan GC-MS analysis, including a range of performance specifications and checks, and the method has been validated by CRECEP, Kiwa NV, the Water Quality Centre and WRc in an interlaboratory test.

Apart from achieving an agreement on the appropriate conversion factors and related issues, there is still an outstanding question of the possible effects of disinfection of new installations, as practised in the field, on the nature and leaching rates of the contaminants. A proposal to the EC, under the Framework Programme IV, is being prepared for co-normative collaborative research to investigate the likely effects and to define test conditions for the disinfection stage, if its inclusion is shown to be necessary.

5.3 Leaching tests for site-applied products

In order to provide realistic estimates of the potential effects on water quality and consumers' exposure to the contaminants, the conditions for leaching tests on site-applied products will need to be more flexible, depending on the chemical nature, application and use of the different types of site-applied products. Compared to the factory-made products, there is less experience within Europe in testing such products, and not much data on the relevance of the test results to field conditions. The UK is probably leading in this field. Experimental data obtained by WRc under this and other contracts show that, for some products such as two-part *in situ* applied pipe linings or solvent cements for plastic pipes, dynamic testing under controlled flow conditions would be more appropriate than standard static tests.

A well designed interlaboratory study for such tests would be especially important as the effects of the variability of the test samples and test conditions are expected to be substantially greater than with factory-made products.

5.4 Approval criteria

Over the past few years of work within CEN a number of shortcomings and obstacles have come to light, which may delay the introduction of a workable harmonised approval system for several years. The lack of consensus and insufficient experimental data to support any particular view, resulted in putting the drafting of standards on a standby, awaiting further information from the participating countries or the outcome of a co-normative research (e.g. cementitious and metallic products, microbiological tests, effects of product disinfection).

Even more than the test methods, the feasibility of an European approval system will depend on other aspects essential in the operation of any approval system, but which have not yet been decided, or even considered, for the European system.

CEN could deal with some of the tasks, such as the co-ordination between the test and product standards, defining analytical requirements and other outstanding tasks listed in Section 4.1.7. Other aspects, particularly those concerning toxicological assessment (e.g. establishing positive lists, setting acceptable concentrations for unregulated contaminants, etc.), are beyond the competence of the current CEN committees but they are likely to be critical factors in establishing the European system.

As an alternative, several countries are considering a mutual acceptance of national approvals. However, full acceptance of an approval from another country is also unlikely to be agreed in the short term, particularly where the approval/certification is based on national standards or where accreditation of testing laboratories and product/manufacturer audits are integral parts of the approval system. Nevertheless, even a partial mutual acceptance of similar tests or test results for assessment by an expert committee, could eliminate some of the current re-testing of products.

5.5 BS6920 test for microbiological growth

The test has been in routine use for a number of years and is internationally recognised (e.g. in Australia and the USA). Nevertheless, there have been some questions regarding its sensitivity, accuracy and suitability for testing a wide range of products, which were preventing it to being accepted outright as the European Standard. The investigations undertaken under this contract have provided valuable additional contribution to understanding of the effects of the test conditions. Conditions have been found, which would offer sufficient sensitivity, comparable with the alternative methods, which are being developed in Germany and The Netherlands.

6. FURTHER NEEDS

6.1 Co-normative research

TC164/WG3 has recognised several problems in developing its standards that need research to be carried out. To date this research is being covered by existing or proposed EC contracts for the following identified problems:

- leaching test conditions for cementitious and metallic products;
- further development and validation of tests for microbiological growth; and
- effects of disinfection of newly installed products on the nature and leaching rates of contaminants.

6.2 Leaching test procedures

Even in the UK, where the testing of site-applied products, testing under dynamic conditions and other specialised testing, is probably more advanced than in other countries, more information and data is needed on some products in order to specify leaching test conditions which would provide realistic and reliable results. The following needs have been identified in this report, but others are likely to emerge from future developments in CEN or UK test procedures:

- information on the chemical nature, methods of application and use of main types of site-applied products;
- investigations to establish the main factors affecting the leaching from different site-applied products;
- investigations to establish the main factors affecting the leaching from organic admixtures in cementitious products (not included in the programme of the current co-normative research project);
- investigations of the feasibility and test conditions of dynamic testing of membrane filters and other products, where appropriate;
- validation, including interlaboratory tests, of emerging leaching test methods for site-applied products, ion exchange resins, etc.

6.3 Approval criteria and related work

A substantial amount of work is needed in this area. In addition to the tasks, where an input from toxicologists or regulatory bodies is required (e.g. setting up positive lists, acceptable concentrations, deciding the basis of conversion factors, etc.), some other outstanding tasks, relating to the determination of contaminants can be identified:

- assessing, for known contaminants of interest, priorities in needs for method development and validation;
- identifying where standard mandatory analytical methods would be appropriate;

- defining the required analytical method performance;
- developing specifications for appropriate quality controls;
- developing specifications for the acceptance of leaching test results.

6.4 Mutual acceptance of national approvals

Should mutual acceptance of national approvals be agreed as a practical temporary measure, while the problems in establishing a harmonised European approval system are being solved, detailed assessment of the relevant national systems would be needed to identify common grounds and differences, in particular for the following factors:

- types of tests;
- test conditions and likely effects on test results;
- selection of specific contaminants for which leaching data is required;
- basis on which maximum acceptable concentrations (MAC) are set;
- relationships between the test results and the MACs;
- conditions of approvals, such as flushing regimes after installation and limitations on applications;
- conditions for accreditation of testing laboratories; and
- required quality control and auditing.

7. REPORTS PREPARED

7.1 Progress reports to the Department of the Environment

Approved Material Testing Procedures (DWE 9534). Progress Report July to December 1990, DoE 2660-M, December 1990.

Improved Material Testing Procedures (DWE 9007). Progress Report January to June 1991, DoE 2835-M, June 1991.

Improved Material Testing Procedures (DWQ 9007). Progress Report July to December 1991, DoE 3004, January 1992.

Improved Material Testing Procedures (DWQ 9007). Progress Report January to June 1992, DoE 3154, July 1992.

Improved Material Testing Procedures (DWQ 9007). Progress Report July to December 1992, DoE 3302, January 1993.

Improved Material Testing Procedures (DWQ 9007). Interim Report September 1990 to February 1993, DoE 3334, February 1993.

7.2 Other reports to the Department of the Environment

Ling, K. and Harding, L. (1992) CEN TC164/WG9 Collaborative Study on the Determination of Acrylamide Monomer in Polyacrylamides. WRc Report DoE 3148.

Wilson, I. (1993) A Review of Draft prEN 852-1 Test Method for Migration for Plastic Pipes Developed by CEN TC155. WRc Report DoE 3304.

Wilson, I. (1993) The Benefits of using General Scan GC-MS to Assess Potential Leaching from Materials into Water Supplies. A paper prepared for CEN TC164/WG3/AHG4. WRc Report DoE 3314.

Wilson, I. (1994) Conversion Factors for Leaching Tests. WRc Report DoE 3642.

Jago, P.H. (1994) Impact of Head Space on Test for Microbiological Growth (BS6920 Section 2.4). WRc Report DoE 3696/1.

Jago, P.H. (1995) Investigation of options to improve the sensitivity of BS6920 Section 2.4. WRc Report DoE 3892.

7.3 External publications

Wilson, I.M. (1993) Assessment of the effects of in situ epoxy resin lining of water distribution pipes on drinking water quality. Proc. Coatings and Linings for the European Water Industry, Birmingham, UK, Independent Technical Conferences Ltd.

Fielding, M., Rogers, H.R. and Wilson, I.M. (1994). Effects of materials on water quality. Inventory of approval systems in EU and EFTA countries for products for use in contact with drinking water. WRc Publication NS 104.

Fielding, M. (1994) Products in contact with drinking water - The approval authority/certifier viewpoint. CEN-EUREAU Materials and Chemicals in Contact with Drinking Water, Seminar Papers, Vienna, March 1994.