

**WRc**

730/1

# **Improved Material Testing Procedures (DWQ 9007)**

*Progress Report to the Department of the Environment  
July to December 1992*

DoE 3302  
JANUARY 1993



## **IMPROVED MATERIAL TESTING PROCEDURES (DWQ 9007)**

Progress Report to the Department of the Environment  
July to December 1992

Report No: DoE 3302

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## SUMMARY

The Drinking Water Inspectorate operates a statutory approval scheme for materials used in contact with water supplies and chemicals used in water treatment processes. A new EC approval scheme is under discussion and the European Committee for Standardisation (CEN) is drafting standards for testing procedures for the scheme. This contract was established 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 test procedures and approval schemes.

During the last six months the evaluation of the results from the CEN TC164/WG3/AHG2 interlaboratory study on migration tests on factory-made products, has been completed and the results, which have been reported to the AHG2, are presented in this report.

Within CEN committees, two draft standards are in preparation for leaching tests on plastic pipes. One, specific to plastic pipes, has been developed by TC 155/WG2 (Plastic Pipes) and the other, which also covers other products, is being drafted by TC 164/WG3/AHG2. The two proposed procedures have been reviewed and the likely effects of the different conditions on test results assessed in a separate document (DoE 3304, January 1993)

Other developments within CEN Committees on the testing and approval of products for use in contact with drinking water are summarised in this report.

## **1. BACKGROUND**

Organic and inorganic impurities from products in contact with water supplies may leach into water and pose a threat to consumers and impair water quality generally. In the UK the Drinking Water Inspectorate operates a statutory scheme which assesses and, where appropriate, gives approval (on behalf of the Secretary of State) to the use of chemicals used in water treatment processes and to materials used in constructing water treatment works and distribution systems. Data from laboratory and field tests are used by an expert committee (CCM) to assess whether any effects on water quality resulting from the use of a product would be unacceptable. It is therefore important that, for a range of different products and various usage, 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 EC 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 the approval bodies and other aspects. Consequently, an approval in one country usually does not usually enable the product to be used in another country.

The Construction Products Directive (89/106/EEC) (CPD), aimed principally at building and construction products, was introduced to ensure an open market and set minimum requirements, including those concerning health and the environment. The elimination of technical barriers to trade is to be achieved by utilising harmonised European Standards. The European Committee for Standardisation (CEN) has been given a mandate by the Commission to draft appropriate harmonised standards. Working Group 3 of the Technical Committee 164 (Water Supply) (TC164/WG3) is preparing standards relating to the effects of materials on water quality and Working Group 9 of the same Committee is drafting standards for chemicals in water treatment. It has been recognised that any proposed improvements in testing and assessment procedures in the UK, need to be synchronised with developments in CEN.

## **2. OBJECTIVES AND PROGRAMME OF WORK**

This contract has the following objectives:

1. To assess the approval schemes used in EC and other countries and identify features which should be incorporated into a UK scheme.
2. Review implications of CEN/EC developments for UK materials' testing and approval.
3. To establish testing needs and devise, validate and recommend modified testing procedures.

### **3. OBJECTIVES FOR THE PERIOD COVERED**

The objectives of the work performed between July and December 1992 were:

1. To complete the evaluation of results from the CEN interlaboratory study to assess the suitability of the proposed standard migration test procedure for factory-made products and report the results to AHG2.
2. To continue involvement in CEN work, concentrating on providing experimental data and advise on drafting migration test procedures and on the interpretation of test results in relation to conditions of approval.
3. To review Draft pr EN852-1 'Test method for migration for plastic pipes' developed by CEN TC155 and compare it with the draft standard which has been produced by TC 164 WG3 AHG2.
4. To continue gathering information on approval schemes in other countries.

### **4. PROGRESS**

#### **4.1 Introduction**

This report presents WRc results of the interlaboratory study and summarises recent progress in the development of CEN standards, in accordance with objectives 1 and 2. A separate report has been prepared (DoE 3304, January 1993) on the Draft pr EN 852-1 (Objective 3). Since there has been no work during this reporting period in gathering further information on approval schemes in other countries (Objective 4), this aspect is not covered by this report.

The last Progress Report (DoE 3154, July 1992) outlined possible experimental work for this period under review, in particular, providing the necessary data to AHG2 to enable the selection of realistic migration test conditions for site-applied products and participating in further CEN interlaboratory studies on leaching tests for other products, such as cementitious and site-applied products. However, no such work has yet been agreed by AHG 2.

#### **4.2 CEN TC 164/WG3/AHG2 Interlaboratory Study**

##### **4.2.1 Objectives of the study**

Within the AHG2 a standard migration test for factory-made products has been drafted. Before forwarding the draft for acceptance, information was needed on the comparability of results from the proposed test. For this purpose, an interlaboratory study was undertaken in which different products were tested in accordance with the proposed test method and the leachates analysed for determinands agreed by the AHG2. A list of the

products and the determinands is given in a previous Progress Report (DoE 3004, January 1992). It was not possible before the study was completed, to agree on criteria by which the test method could be judged as suitable or not. The following eight testing institutes agreed to participate in the study:

- CRECEP, Centre de Recherche et de contrôle de Eaux, Paris, France
- CRPAM, Centre de Recherche de Pont-à-Mousson, Pont-à-Mousson, France
- Danish Teknologisk Institut (DTI), Aarhus, Denmark
- DVGW - Forschungsstelle am Engler-Bunte-Institut, Karlsruhe, Germany
- KIWA NV, Research Division, Nieuwegein, The Netherlands
- LHRSP, Laboratoire Hygiène Recherche Santé Publique, Vandoulvre les Nancy, France
- VTT, Technical Research Centre of Finland, Espoo, Finland
- WRc Medmenham, UK

#### **4.2.2 Test samples**

The samples were provided by two of the participating laboratories, KIWA and Engler Bunte Institute. The samples were delivered to WRc over a period of three months and upon receipt stored at ambient temperature in the dark. The migration study on the epoxy resin (metal plates coated with epoxy resin) was begun on the 20 January and the migration studies on the PVC pipes, GRP pipe section and EPDM rubber sheets was begun on the 2 April 1992. Information on the test samples are summarised in Table 4.1.



**Table 4.1 Test samples for CEN TC 164/WG3/AHG2 interlaboratory study**

Product	Test samples	Received from	Date received	WRc Ref:
Epoxy Resin coating	4 fully coated metal plates (20 x 15 cm)	KIWA	14 Jan 1992	CEN/EP
Grey PVC pipe 75 mm o.d.	2 lengths of pipe	Engler Bunte Institute	3 April 1992	CEN/LP
Grey PVC pipe 32 mm o.d.	2 lengths of pipe	Engler Bunte Institute	3 April 1992	CEN/SP
Glass-reinforced polyester pipe	1 pipe segment + 1 pipe segment coated with clear resin	KIWA	March 1992	CEN/GRP/A
		KIWA	March 1992	CEN/GRP/B*
Rubber	5 square sheets	Engler Bunte Institute (20 x 20 cm)	3 April 1992	CEN/RUB

\* Sample of GRP coated with clear resin.

The samples of GRP represented sections of large diameter pipes. In practice the outside and edges of the pipe would not come into contact with potable water. To prevent the outside and edges from leaching they were coated with an inert silicone putty (provided by KIWA).

Because of the limited quantity of test samples provided, it was not always possible to test at the recommended sample surface area to volume of test water ratio (S/V) of 1 cm ml<sup>-2</sup>. Detailed information on the samples and S/V ratios tested are given in Appendix A.

#### 4.2.3 Methods

The tests performed in accordance with the relevant conditions of the draft migration test method 'Water supply - Influence of materials on water quality; Part 2: Influence due to migration' sixth draft, November 1991, prepared by the Ad Hoc Group 2 of TC164/WG3. The test method is for factory made or factory applied products. The products tested were for cold water systems and the test protocol was three successive periods of 72 hours stagnation in deionised activated carbon filtered water.

The products were tested under conditions suitable for factory-made products for cold water systems (test temperature  $23 \pm 2$  °C), but only with unchlorinated test water, either by immersing test samples in test water in glass vessels or, in case of narrow pipes, filling lengths of the pipe with the test water.

The following analytical methods were used for the analysis of the leachates for the specified determinands:

- (a) Total organic carbon, TOC: routine determination by persulphate/UV oxidation instrumental method
- (b) Monohydric phenols: standard 4-Aminoantipyrine (pH 10) SCA method
- (c) Secondary aliphatic amines - spectrophotometric method provided by KIWA
- (d) Primary aromatic amines - spectrophotometric method provided by KIWA
- (e) Styrene, alkylbenzenes and dimethylphthalate were determined by GC-MS in the extracts used also for 'general scan' GC-MS analysis
- (f) Epoxy resin constituents diglycidylethers of bisphenol A and bisphenol F - an HPLC based method developed in WRc
- (g) Cobalt - routine determination by inductively coupled plasma spectrometry
- (h) Lead - routine determination by atomic absorption
- (i) 'General scan' GC-MS - established WRc semiquantitative method based on extraction of acidified samples, internal standards added with DCM and GC-MS examination of the concentrated extracts.

The interlaboratory study specified the required detection limit for each determinand and listed the recommended type of the analytical method to be used. The KIWA methods c) and d) (together with analytical standards) and the WRc methods f) and i) were circulated to all participants at the beginning of the study. For the other determinands in-house methods were used.

The new methods were set up and validated before the test commenced. Quality control analytical samples were analysed with each batch of leachates for each determinand, where appropriate.

Details on test samples, sample storage and test conditions for each product are given in Appendix A.

#### **4.2.2 Results**

A summary of the WRc results (migration values) for the specific determinands was already presented in the previous Progress Report (DoE 3154, July 1992). Detailed results

were reported to the Convenor of AHG2 before the last meeting of the Group in October and an updated version of the report is enclosed in Appendix A. (A copy of the updated version has also been sent to the Convenor of AHG2).

Seven of the eight participating laboratories sent at least some results to the Convenor before the last meeting of the Group. However only WRc provided all the required data in the agreed format. A preliminary summary of the available results was circulated at the meeting, indicating a fair degree of agreement but also some major differences. Any discussion on the outcome of the study and reasons for the differences was postponed until the next meeting in February, before which the other laboratories were asked to bring their data up to the WRc's standard.

#### **4.2.5 Discussion**

From the factory made products, the GRP pipes and other large diameter pipes where the outside surface (which is not in contact with drinking water) is from different material than the inner surface, present the main problem with test samples. The draft standard recommends several arrangements on how to test such products, though none of these is regarded as entirely satisfactory. Another approach, recommended by KIWA, was used for this study, i.e. the parts of the test sample not in contact with drinking water were covered with a silicone putty to prevent leaching of interfering compounds and the pipe segment was immersed in a test vessel. However this covering material itself could give rise to undesirable leaching. The results did show elevated TOC levels in the GRP procedural blanks and several compounds originating from the putty were detected by GC-MS though the levels, relatively to those in the leachates, were low. There was no interference with the specified determinands. It is not yet known what test arrangements have been used by the other participating laboratories nor how the results compare.

Results for spiked control samples were not very satisfactory for some of the specified determinands, particularly those determined by GC-MS (recoveries  $\leq 50\%$ ). Human error in preparing the standard solutions cannot be excluded even though the records were correct. Inadequate calibration of the GC-MS system or losses from the sample itself during the stagnation periods are other possible explanations.

According to the draft standard, all analysis should be performed after each stagnation period on duplicate test samples with two test waters. For each test temperature this represents 12 samples of leachates and three to six procedural blanks for each parameter, including GC-MS analysis. In view of the costs of the GC-MS analysis, particularly the cost of identification of the peaks, and bearing in mind the purpose of including the general scan GC-MS (as a safety step to detect unsuspected compounds, that may leach), it should be considered how best to specify the GC-MS requirements to provide adequate information at reasonable cost. During this study all extracts were prepared, as specified, but for all products (except the epoxy resin coating) only the extracts from the first and the third stagnation periods were submitted for peak identification.

The GC-MS analysis of the PVC pipes leachates did not detect any significant compounds even after the first stagnation period. Considerable leaching of benzyl alcohol

was detected in the leachates from the epoxy coating, which was consistent with the high TOC values, though it did not account for all the TOC concentrations found. For GRP pipes the specified determinand dimethyl phthalate was not detected, but the general scan GC-MS detected the presence of dibutyl phthalate. Complex chromatograms of unknown peaks were obtained from the rubber leachates. Without considerable additional work it was not possible to correlate the peaks for the duplicate samples and for the different stagnation period, as was done for the other products.

### **4.3 Progress in the development of CEN standards**

During the period under review there was only one meeting of TC 164/WG3/AHG2 (migration tests) and one of the AHG4 ('positive lists').

At the previous meeting of the AHG2 in June a proposal was put forward to TC164/WG3 to prepare separate standards for factory-made and site-applied products. As there was no response, due to cancellation of the last WG3 meeting, it was not possible to progress along these lines.

The convenor was to contact, via TC164/WG3, appropriate Technical Committees preparing Product Standards. This has not yet happened.

Characteristics of test waters for cementitious products were again discussed and there were doubts that the proposed low-hardness water could be prepared. It was decided to check the calculations and prepare the water in a laboratory. Other laboratories may then be asked to prepare both test waters in accordance with the proposed specifications.

An interlaboratory test on cementitious products will need to be carried out. The convenor agreed to prepare a proposal (which cements to tests, what to measure, etc) for discussion at the next meeting in February.

There was a concern about the different contents and formats of the related draft standards being produced by AHGs within TC164/WG3. A resolution was prepared for the WG3 proposing setting up a Drafting Committee to assure consistency and compatibility of the Standards.

The TC155/WG2 is preparing a proposal for correction factors for pipes. The Group felt that it should not wait any longer and should consider in detail whether the proposed approach for pipes was acceptable and, if so, extend it to other products. After the next meeting of TC155/WG2 in December, the convenor should prepare a Draft of the Part 2.2 for discussion at the next meeting.

At the meeting of the AHG4, the discussion concentrated on the following; an updated inventory of positive lists, future positive lists, future European approval criteria (in particular whether to have one universal list, as for materials in contact with food, or a separate list for each type of polymer), how to include migration limits for chemicals that are not starting materials and on what basis are the migration limits set in different countries.

It is still not at all clear who is to set up the European approval system, what should be the criteria for approval, how should they be set up and who should do it and, also, what would need to be tested. PC6 is planning to hold a seminar on this next year and it was thought unlikely that any real progress could be made until after the seminar.

After a lengthy discussion it was agreed that, although the AHG4 did not have a specific mandate for this, it should put forward a discussion document proposing a well-argued approval system. UK and France should also provide more written information on the experience and value of using GC-MS analysis as a part of an approval system, in particular on what is found and how are the results assessed.

## **5. INTERIM CONCLUSIONS**

1. Interlaboratory study on proposed migration tests for factory-made products has been completed and the results reported to CEN TC164/WG3/AHG2.
2. The contract enabled WRc to provide the best set of data of the eight participating laboratories.
3. Three problems, which need to be addressed in future, were encountered during the study, i.e. test arrangements for heterogeneous large diameter pipes, low recoveries of some of the specified determinands and high cost of the required GC-MS analysis.
4. The progress in drafting CEN standards for test methods is slow. This is caused, to some extent, by a slow progress in other related Groups such as TC155/WG2 and TC164/WG3 and by insufficient information on the effects of some proposed test conditions on test results.
5. It is important to set up and agree criteria for approval of products for use in contact with drinking water though no CEN Committee has so far been mandated to do this.

## **6. WORK PROPOSED AND TARGETS FOR THE FOLLOWING PERIOD**

1. A short paper will be prepared for discussion in the TC 164/WG3/AHG4 on the benefits of using GC-MS to assess potential for leaching from materials into water supplies.
2. An interim report will be produced summarising the progress of the contract from the beginning (September 1990).
3. CEN involvement will continue to concentrate on providing advice on drafting migration test procedures and on the interpretation of test results in relation to conditions of approval.

4. Any experimental work would depend on the outcome of the next meeting of CEN TC164/WG3/AHG2 on 9 February 1993. This would most likely be related to testing cementitious products.

PROJECT NUMBER: DWQ 9007

PROJECT TITLE: IMPROVED MATERIALS TEST

CONTRACT REFERENCE: PECD 7/7/370

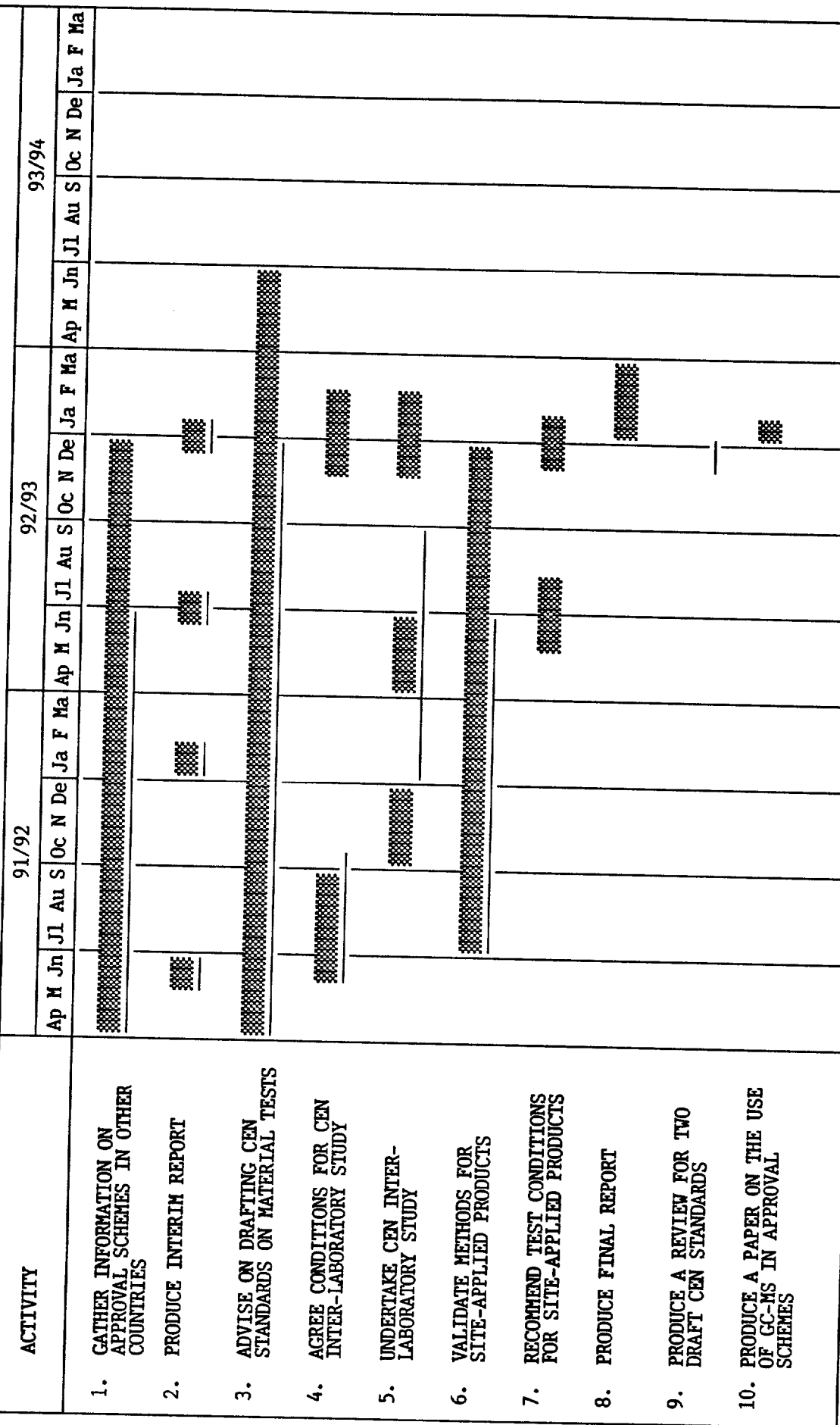
WRC CONTRACT MANAGER: I WILSON

Start Date: 01/09/90

Current End Date: 31/03/93

Revised End Date:

Research Programme Bar Chart



# **APPENDIX A - CEN TC 164/WG3/AHG2 INTERLABORATORY STUDY - TEST REPORT**

Form N° 001	Epoxy resin coating	- /1 : Test samples /2 : Test conditions /3 : Analytical results /4 : Migration values /5 : Results for control samples
002	Grey PVC pipe - 75 mm diameter	- /1 : Test samples /2 : Test conditions /3 : Analytical results /4 : Migration values
003	Grey PVC pipe - 32 mm diameter	- /1 : Test samples /2 : Test conditions /3 : Analytical results /4 : Migration values
004	Glass-reinforced polyester pipes	- /1 : Test samples /2 : Test conditions /3 : Analytical results /4 : Migration values /5 : Results for control samples
005	EPDM Rubber	- /1 : Test samples /2 : Test conditions /3*: Analytical results /4*: Migration values /5 : Results for control samples

\* GC-MS chromatograms after the first and third stagnation periods contained a number of unknown compounds (extracts after the second period were not analysed) which could not be easily correlated for the replicate samples and the different periods. The defected compounds and their approximate concentrations are, therefore, listed separately and their migration values were not calculated.



Form No: 001/1

Product: Epoxy Resin Coati

**LEACHING/MIGRATION TESTS**

Test Samples Received

Received from: KIWA N.V, The Netherlands (Mr H Van Der Jagt)

Received at WRc (Date): 14/01/92

Manufactured/prepared (Date): Before 14/01/92

Dimensions: 150 x 200 mm

Sample Quantity: 4 plates

Sample appearance: White pigmented coating on metal plates

Manufacturers Ref No: None

Sample storage before test: In paper wrapping as supplied, in desk.

Tests to be carried out: In accordance with CEN/TC164/WG3/AHG2-N28 sixth draft,  
November 1991

Determinands to be measured: On leachate from each of three, 3-day (72 h) stagnation  
periods;  
TOC  
Secondary aliphatic amines  
Alkyl benzenes  
Bisphenol A/F Diglycidyl ethers (DGEBA, DGEBF)  
GC-MS general scan

## LEACHING/MIGRATION TESTS

### Test Conditions

Test carried out in duplicate (A and B), two plates per test vessel.

Surface area for test: 1200 cm<sup>2</sup> (each plate 600 cm<sup>2</sup>)

#### Test Water

- Origin: Mains water deionized activated carbon filtered
- Conductivity: <12  $\mu\text{Scm}^{-1}$
- Chlorination: nil
- Volume during test: 1.850l
- Surface to volume ratio: 0.65 cm<sup>-1</sup>
- Test Vessel Glass tank glass top, no headspace
- Temperature °C 24.0-25.0

Form No: 001/3a

Product: Epoxy Resin Coating

**LEACHING/MIGRATION TESTS****Analytical Results - Specific Determinands**

DETERMINAND/ SAMPLE CODE	Concentration found µg/l		
	First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
<u>TOC</u>			
TEST A	16000	10700	6890
TEST B	14200	7960	5660
BLANK A	390	250	110
BLANK B	450	300	350
<u>SECONDARY ALIPHATIC AMINES</u>			
TEST A	<50	<50	<50
TEST B	<50	<50	<50
BLANK A	<50	<50	<50
BLANK B	<50	<50	<50
<u>DGEBA</u>			
TEST A	<1	<1	<1
TEST B	<1	<1	<1
BLANK A	<1	<1	<1
BLANK B	<1	<1	<1
<u>DGEBF</u>			
TEST A	<1	<1	<1
TEST B	<1	<1	<1
BLANK A	<1	<1	<1
BLANK B	<1	<1	<1

LEACHING/MIGRATION TESTS

Analytical Results - Specific Determinands

DETERMINAND/ SAMPLE CODE	Concentration found µg/l		
	First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
<u>TOLUENE</u>			
TEST A	21	9	10
TEST B	9	13	9
BLANK A	<0.1	<0.1	<0.1
BLANK B	<0.1	<0.1	<0.1
<u>O-XYLENE</u>			
TEST A	12	6	7
TEST B	1	8.5	1
BLANK A	<0.1	<0.1	<0.1
BLANK B	<0.1	0.07	<0.1
<u>M-XYLENE</u>			
TEST A	15	7	8
TEST B	2.5	4.4	2.5
BLANK A	0.1	<0.1	<0.1
BLANK B	<0.1	<0.1	<0.1
<u>ETHYL BENZENE PLUS P-XYLENE</u>			
TEST A	21	13	16
TEST B	7	11	7
BLANK A	<0.1	0.25	0.2
BLANK B	<0.1	0.26	0.3
<u>TRIMETHYL BENZENE ISOMERS-TOTAL</u>			
TEST A	14.8	2.1	4.7
TEST B	4.3	5.8	4.5
BLANK A	<0.5	<0.5	<0.5
BLANK B	<0.5	<0.5	<0.5
<u>ALKYL BENZENES- TOTAL(FROM ABOVE)</u>			
TEST A	83.8	37.1	45.7
TEST B	16.8	42.7	24.0

**LEACHING/MIGRATION TESTS**

Concentrations of compounds detected by GC-MS

COMPOUND	GC-MS Scan	Test Sample	Concentration (µg/l)		
			T1	Stagnation: T2	T3
Unknown m/z- 56,31,41,43 (glycidyl ether)	133- 160	A: B:	70 <1	61 71	<1 <1
Unknown m/z 43,56,41,73	407- 440	A: B:	290* <1	135* <1	130* 1
Unknown m/z 58,72,87	510	A: B:	<1 <1	15 46	<1 <1
Benzaldehyde	662- 663	A: B:	<1 <1	66 <1	58 29
Benzyl alcohol	790- 980	A: B:	1400* 860*	1180* 340*	560* 480*
Benzoic acid	1060- 1081	A: B:	<1 <1	10 4.2	29 62
t-butyl- cyclohexane	1167- 1186	A: B:	7.0 7.0	0.2 <1	<1 <1
Nonyl phenols	1694- 1781	A: B:	8.5 4.5	3.7 7.8	2.2 4.6

\* - GC-MS overload, approximate concentrations determined after 30 x dilution

LEACHING/MIGRATION TESTS

Migration Values

DETERMINAND Sample code	Migration values $\mu\text{gdm}^{-2} \text{ day}^{-1}$		
	First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
<u>TOC</u>			
TEST A	822	550	354
TEST B	730	409	291
BLANK A	20	13	5
BLANK B	23	15	18
<u>SECONDARY ALIPHATIC AMINES</u>			
TEST A	<2.6	<2.6	<2.6
TEST B	<2.6	<2.6	<2.6
BLANK A	<2.6	<2.6	<2.6
BLANK B	<2.6	<2.6	<2.6
<u>DEGBA</u>			
TEST A	<0.05	<0.05	<0.05
TEST B	<0.05	<0.05	<0.05
BLANK A	<0.05	<0.05	<0.05
BLANK B	<0.05	<0.05	<0.05
<u>DEGBF</u>			
TEST A	<0.05	<0.05	<0.05
TEST B	<0.05	<0.05	<0.05
BLANK A	<0.05	<0.05	<0.05
BLANK B	<0.05	<0.05	<0.05

LEACHING/MIGRATION TESTS

Migration Values

DETERMINAND Sample code	Migration values $\mu\text{gdm}^{-2} \text{ day}^{-1}$		
	First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
<u>TOLUENE</u>			
TEST A	1.1	0.5	0.5
TEST B	0.0	0.7	0.5
BLANK A	<0.1	<0.1	<0.1
BLANK B	<0.1	<0.1	<0.1
<u>O-XYLENE</u>			
TEST A	0.6	0.3	0.4
TEST B	<0.1	0.4	<0.1
BLANK A	<0.1	<0.1	<0.1
BLANK B	<0.1	<0.1	<0.1
<u>M-XYLENE</u>			
TEST A	0.8	0.4	0.4
TEST B	0.1	0.2	0.1
BLANK A	<0.1	<0.1	<0.1
BLANK B	<0.1	<0.1	<0.1
<u>ETHYL BENZENE AND P-XYLENE</u>			
TEST A	1.1	0.7	0.8
TEST B	0.4	0.6	0.4
BLANK A	<0.1	<0.1	<0.1
BLANK B	<0.1	<0.1	<0.1
<u>TRI-METHYL BENZENE ISOMERS TOTAL</u>			
TEST A	0.8	0.1	0.2
TEST B	0.2	0.3	0.2
BLANK A	<0.1	<0.1	<0.1
BLANK B	<0.1	<0.1	<0.1
<u>ALKYL BENZENES- TOTAL(FROM ABOVE)</u>			
TEST A	4.4	2.0	2.3
TEST B	0.8	2.2	1.2

LEACHING/MIGRATION TESTS

Migration values for compounds detected by GC-MS

COMPOUND	GC-MS Scan	Test Sample	Migration value M ( $\mu\text{g dm}^{-2} \text{ day}^{-1}$ )		
			T1	Stagnation: T2	T3
Unknown m/z- 56,31,41,43 (glycidyl ether)	133- 160	A:	3.6	3.1	<0.05
		B:	<0.05	3.6	<0.05
Unknown m/z 43,56,41,73	407- 440	A:	14.9	6.9	6.7
		B:	<0.05	<0.05	<0.05
Unknown m/z 58,72,87	510	A:	<0.05	0.8	<0.05
		B:	<0.05	2.4	<0.05
Benzaldehyde	662- 663	A:	<0.05	3.4	3.0
		B:	<0.05	<0.05	1.5
		B:	<0.05	<0.05	1.5
Benzyl alcohol	790- 980	A:	71.9	60.6	28.8
		B:	44.2	17.5	24.7
Benzoic acid	1060- 1081	A:	<0.05	0.5	1.5
		B:	<0.05	0.2	3.2
t-butyl- cyclohexane	1167- 1186	A:	0.4	<0.05	<0.05
		B:	0.4	<0.05	<0.05
Nonyl phenols	1694- 1781	A:	0.44	0.20	0.11
		B:	0.23	0.40	0.24



**LEACHING/MIGRATION TESTS**

Analytical Results - Control Determinands

DETERMINAND/ (Concentration added) µg/l	Concentration found µg/l		
	First Stagnation T2	Second Stagnation T2	Third Stagnation T3
Second aliphatic amines (100 µg/l)	92, 82	107.5, 124	96, 102
<u>ALKYLBENZENES</u>			
Toluene (18.8µg/l)	8, 6.8	5, 9	7, 7
Et Benz (18.8µg/l)	8, 6.9	6, 9	6.5, 7.5
O-Xyl (18.8µg/l)	8, 7.1	6, 9	7, 8
M-Xyl (18.8µg/l)	7, 6.4	6, 7.5	6, 7
DGEBA (20µg/l)	11.6, 11.4	8.2, 8.6	9.8, 13.4
DGEBF (20.4µg/l)	20.4, 15	15.3, 16.8	19.0, 26.2

Form No: 002/1  
Product: Grey PVC pipe  
(75mm diameter)

**LEACHING/MIGRATION TESTS**

Test Samples Received

Received from: Engler Bunte Institut

Received at WRc (Date): 03/04/92

Manufactured/prepared (Date): About 04/11/91

Dimensions: 75.1 mm O.D 67.25 mm I.D 1018 mm long

Sample Quantity: 2 pieces

Sample appearance: Grey plastic pipe

Manufacturers Ref No: OMNIPLAST DVGW K022 PVC n DN65 75 x 3.6  
PN 10 DIN 19532 04119130  
ONORM B5182 GEPRUFT D5 972 NS S1S 1776

Sample storage before test: In the laboratory, in the original packing

Tests to be carried out: In accordance with CEN/TC164/WG3/AHG2-N28 sixth draft,  
November 1991

Determinands to be measured: On leachate from each of three, 3-day (72 h) stagnation  
periods;  
TOC  
Lead  
GC-MS general scan

Form No: 002/2  
Product: Grey PVC pipe  
(75mm diameter)

## LEACHING/MIGRATION TESTS

### Test Conditions

Test carried out in duplicate (A and B)

Surface area for test: 2150.75 dm<sup>2</sup>

### Test Water

- Origin: Mains water deionized, activated carbon filtered
- Conductivity: <12  $\mu\text{Scm}^{-1}$
- Chlorination: nil
- Volume during test: 3.56l
- Surface to volume ratio: 0.59 cm<sup>-1</sup>
- Test Vessel The pipe itself, held upright, bottom end closed with glass stopper. Top covered with aluminium foil.
- Temperature °C 22.5-25.0

Form No: 002/3a  
 Product: Grey PVC pipe  
 (75mm diameter)

**LEACHING/MIGRATION TESTS**

Analytical Results - Specific Determinands

DETERMINAND		Concentration found ug/l		
Sample code		First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
<u>TOC</u>	PIPE A	400	<200	<200
	PIPE B	<200	290	<200
	BLANK A	<200	<200	<200
	BLANK B	<200	<200	<200
<u>LEAD</u>	PIPE A	90	80	32
	PIPE B	59	100	32
	BLANK A	<2	<2	<2
	BLANK B	<2	<2	<2

**LEACHING/MIGRATION TESTS**

**Analytical Results - GC/MS Analysis**

Sample Code	Results		
	Scan No.	Compound	Concn. ug/l
<u>Third Stagnation 72 h</u>			
PIPE A	0196	2-Pentanol-2-methyl	2
	0229	3-Pentanol-3-methyl	1
PIPE B	0194	2-Pentanol-2-methyl	2
	0227	3-Pentanol-3-methyl	1
 <u>Third Stagnation 72 h</u>			
PIPE A		No compounds detected above 1 µg/l	
PIPE B	1654	Unknown m/z 43, 179, 161, 59 No other compounds detected above 1 µg/l	2

Form No: 002/4a  
 Product: Grey PVC Pipe  
 (75mm diameter)

**LEACHING/MIGRATION TESTS**

Migration Values

DETERMINAND		Migration values $\mu\text{gdm}^{-2} \text{ day}^{-1}$		
Sample code		First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
<u>TOC</u>	PIPE A	22	11	<11
	PIPE B	<11	16	<11
	BLANK A	<11	<11	<11
	BLANK B	<11	<11	<11
<u>LEAD</u>	PIPE A	5.0	4.5	1.8
	PIPE B	3.3	6.5	1.8
	BLANK A	<0.2	<0.2	<0.2
	BLANK B	<0.2	<0.2	<0.2

Form No: 002/4b  
 Product: Grey PVC Pipe  
 (75mm diameter)

**LEACHING/MIGRATION TESTS**

Migration Values for compounds detected by GC-MS

COMPOUND	GC-MS Scan	Test Sample	Migration value M ( $\mu\text{g dm}^{-2} \text{ day}^{-1}$ )		
			Stagnation: T1	T2	T3
		75 mm A: B:	All Values Below 0.1		

Form No: 003/1  
Product: Grey PVC pipe  
(32mm diameter)

**LEACHING/MIGRATION TESTS**

Test Samples Received

Received from: Engler Bunte Institut

Received at WRc (Date): 03/04/92

Manufactured/prepared (Date): About 5/12/91

Dimensions: 32.3 mm O.D 27.25 mm I.D 1018 mm long

Sample Quantity: 2 pieces

Sample appearance: Grey plastic pipe

Manufacturers Ref No: OMNIPLAST PVC b 32 x 18 PN10 DIN 8061/62 0512.91.22

Sample storage before test: In the laboratory, in the original packing

Tests to be carried out: In accordance with CEN/TC164/WG3/AHG2-N28 sixth draft,  
November 1991

Determinands to be measured: On leachate from each of three, 3-day (72 h) stagnation  
periods;  
TOC  
Lead  
GC-MS general scan



Form No: 003/2  
Product: Grey PVC pipe  
(32mm diameter)

## LEACHING/MIGRATION TESTS

### Test Conditions

Test carried out in duplicate (A and B)

Surface area for test: 871 cm<sup>2</sup>

#### Test Water

- Origin: Mains water deionized activated carbon filtered
- Conductivity: <12  $\mu\text{Scm}^{-1}$
- Chlorination: nil
- Volume during test: 0.591
- Surface to volume ratio: 1.47 cm<sup>-1</sup>
- Test Vessel The pipe itself, held upright, bottom end closed with glass stopper. Top covered with aluminium foil.
- Temperature °C 22.5-25.0

Form No: 003/3a  
 Product: Grey PVC pipe  
 (35mm diameter)

**LEACHING/MIGRATION TESTS**

**Analytical Results - Specific Determinands**

DETERMINAND	Concentration found ug/l		
	First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
<u>TOC</u> PIPE A	<200	<200	<200
PIPE B	350	<200	<200
BLANK A	<200	<200	<200
BLANK B	<200	<200	<200
<u>LEAD</u> PIPE A	170	139 / 140*	221 / 220*
PIPE B	140	112 / 130	493 / 460
BLANK A	<2	<2	<2
BLANK B	<2	<2	<2

\* Repeat analysis

Form No: 003/3b

Product: Grey PVC pipe  
(32 mm diameter)

## LEACHING/MIGRATION TESTS

Analytical Results - GC/MS Analysis

Sample Code	Results		
	Scan No.	Compound	Concn. ug/l
<u>First Stagnation 72 h</u>			
PIPE A	1223	Propionic acid 2-methyl 2.2 dimethyl-1-(2-hydroxy-1 methyl) propyl ester	4
	1245	Propionic acid 2-methyl 3-hydroxy-2,4,4-triethyl pentyl ester	4
PIPE B		No other compound detected above 1 µg/l	
		No compound detected above 1 µg/l	
<u>Third Stagnation 72 h</u>			
PIPE A		No compound detected above 1 µg/l	
PIPE B		No compound detected above 1 µg/l	

LEACHING/MIGRATION TESTS

Migration Values

DETERMINAND		Migration values $\mu\text{gdm}^{-2} \text{ day}^{-1}$		
Sample code		First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
<u>TOC</u>	PIPE A	<5	<5	<5
	PIPE B	7.8	<5	<5
	BLANK A	<5	<5	<5
	BLANK B	<5	<5	<5
<u>LEAD</u>	PIPE A	3.8	3.1/3.1*	4.9/4.9*
	PIPE B	3.1	2.5/2.9*	10.9/10.2*
	BLANK A	<0.1	<0.1	<0.1
	BLANK B	<0.1	<0.1	<0.1

\* Repeat analysis

Form No: 003/4b  
 Product: Grey PVC Pipe  
 (32mm diameter)

**LEACHING/MIGRATION TESTS**

Migration Values for compounds detected by GC-MS

COMPOUND	GC-MS Scan	Test Sample	Migration value M ( $\mu\text{g dm}^{-2} \text{ day}^{-1}$ )		
			Stagnation:		
			T1	T2	T3
		32 mm A: B:	All Values Below 0.1		

Form No: 004/1  
Product: GRP Pipe

## LEACHING/MIGRATION TESTS

### Test Samples Received

Received from: KIWA NV

Received at WRc (Date): March 1992

Manufactured/prepared (Date): Not known to WRc

Dimensions: Surface exposed to water, 20 x 18.7 cms. Surfaces not to be exposed to water (ie the back and edges) were covered with a layer of SABA transparent silicone putty, 091212, and cured at ambient temperature for two days before commencement of test.

Sample Quantity: 2 pieces

Sample appearance: 1 piece - Glass reinforced polyester (GRP) on a section of grey composite pipe (Sample A)  
1 piece - As above but coated with unknown clear resin (Sample B)

Manufacturers Ref No: None

Sample storage before test: In the laboratory, in the original packing

Tests to be carried out: In accordance with CEN/TC164/WG3/AHG2-N28 sixth draft,  
November 1991

Determinands to be measured: On leachate from each of three, 3-day stagnation periods;  
TOC  
Cobalt  
Styrene  
Dimethylphthalate  
GC-MS general scan

## LEACHING/MIGRATION TESTS

### Test Conditions

Surface area for test: 374 cm<sup>2</sup>

#### Test Water

- Origin: Mains water deionized activated carbon filtered.
- Conductivity: <12  $\mu\text{Scm}^{-1}$
- Chlorination: nil
- Volume during test: 3.39l
- Surface to volume ratio: 0.11 cm<sup>-1</sup>
- Test Vessel Glass tank, covered with glass sheet, no headspace
- Temperature °C 22.5 - 25.0

#### Blank

Glass plate coated with SABA transparent silicone putty, 091212, and cured at ambient temperature for two days before commencement of test.

Form No: 004/3a  
 Product: GRP Pipe  
 A: uncoated  
 B: coated

**LEACHING/MIGRATION TESTS**

**Analytical Results - Specific Determinands**

DETERMINAND		Concentration found µg/l		
		First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
<u>TOC</u>	GRP A	5510	2160	1780
	GRP B	9620	3500	2230
	BLANK A	1000	400	220
	BLANK B	740	300	210
<u>Cobalt</u>	GRP A	6	<5	<5
	GRP B	<5	<5	<5
	BLANK A	<5	<5	<5
	BLANK B	<5	<5	<5
<u>Styrene</u>	GRP A	>15	22	21
	GRP B	>20	27	17
	BLANK A	<0.1	<0.1	<0.1
	BLANK B	<0.1	<0.1	<0.1
<u>DMP</u>	GRP A	<0.1	<0.1	<0.1
	GRP B	<0.1	<0.1	<0.1
	BLANK A	<0.1	<0.1	<0.1
	BLANK B	<0.1	<0.1	<0.1



Form No: 004/3b  
 Product: GRP Pipe  
 A: uncoated  
 B: coated

# **LEACHING/MIGRATION TESTS**

## Concentrations of compounds detected by GC-MS

COMPOUND	GC-MS Scan	Test Sample	Concentration (µg/l)		
			T1	Stagnation: T2	T3
Unknown m/z- 55,83,43,98 (4-methyl-pent-3-ene-2-one?)	304- 546	A:	20	ND	22
		B:	10	ND	48
4-hydroxy-4-methyl-2-pentanone	381- 384	A:	31	ND	<1
		B:	15	ND	<1
Diacetone alcohol	625- 628	A:	<1	ND	39
		B:	<1	ND	76
Acetophenone	783- 1070	A:	22	ND	10
		B:	2	ND	16
Unknown m/z 82,54,143	1281- 1580	A:	4	ND	7
		B:	<1	ND	6
Dibutyl phthalate isomers	1786- 2120	A:	9	ND	1.8
		B:	147	ND	<0.3

ND - not determined

(Outside and edges of both test samples A and B were covered with a layer of SABA silicone putty).

Form No: 004/4a  
 Product: GRP Pipe  
 A: uncoated  
 B: coated

# LEACHING/MIGRATION TESTS

## Migration Values

DETERMINAND Sample code	Migration values $\mu\text{gdm}^{-2} \text{ day}^{-1}$		
	First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
<u>TOC</u>			
GRP A	1665	650	534
GRP B	2907	1060	674
BLANK A	303	120	66
BLANK B	222	90	63
<u>Cobalt</u>			
GRP A	1.8	<1.5	<1.5
GRP B	<1.5	<1.5	<1.5
BLANK A	<1.5	<1.5	<1.5
BLANK B	<1.5	<1.5	<1.5
<u>Styrene</u>			
GRP A	4.5	6.6	6.3
GRP B	>6.0	8.2	5.1
BLANK A	<0.1	<0.1	<0.1
BLANK B	<0.1	<0.1	<0.1
<u>DMP</u>			
GRP A	<0.1	<0.1	<0.1
GRP B	<0.1	<0.1	<0.1
BLANK A	<0.1	<0.1	<0.1
BLANK B	<0.1	<0.1	<0.1

Form No: 004/4b  
 Product: GRP Pipe  
 A: uncoated  
 B: coated

# LEACHING/MIGRATION TESTS

## Migration Values for compounds detected by GC-MS

COMPOUND	GC-MS Scan	Test Sample	Migration value M ( $\mu\text{g dm}^{-2} \text{ day}^{-1}$ )		
			T1	Stagnation: T2	T3
Unknown m/z-55,83,43,98 (4-methyl-pent-3-ene-2-one?)	304-546	A:	6.0	ND	6.6
		B:	3.0	ND	14.5
4-hydroxy-4-methyl-2-pentanone	381-384	A:	9.4	ND	<0.3
		B:	4.5	ND	<0.3
Diacetone alcohol	625-628	A:	<0.3	ND	11.8
		B:	<0.3	ND	23.0
Acetophenone	783-1070	A:	6.6	ND	3.0
		B:	0.6	ND	4.8
Unknown m/z 82,54,143	1281-1580	A:	1.2	ND	2.1
		B:	<0.3	ND	1.8
Dibutyl phthalate isomers	1786-2120	A:	2.7	ND	1.8
		B:	44.4	ND	<0.3

ND - not determined

(Outside and edges of both test samples A and B were covered with a layer of SABA silicone putty).

**LEACHING/MIGRATION TESTS**

Analytical Results - Control Determinands

DETERMINAND (Concentration added µg/l)	Concentration found µg/l		
	First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
Styrene (100µg/l)	31 32	28 23	- -
Dimethyl phthalate (100µg/l)	7 7	9.6 8.2	- -

Form No: 005/1  
Product: Rubber sheet

**LEACHING/MIGRATION TESTS**

Test Samples Received

Received from: Engler-Bunte-Institut

Received at WRc (Date): 3 April 1992

Manufactured/prepared (Date): Not known to WRc

Dimensions: 20 x 20 x 0.2 cms

Sample Quantity: 5 pieces

Sample appearance: Squares of black rubber

Manufacturers Ref No: EPDM 149017

Sample storage before test: In the laboratory, at ambient. Samples had a very pronounced odour (ester-like?). The sheets were exposed laboratory air for one week, turning over after four days.

Tests to be carried out: In accordance with CEN/TC164/WG3/AHG2-N28 sixth draft, November 1991

Determinands to be measured: On leachate from each of three, 3-day (72h) stagnation periods;  
TOC  
Phenols  
Primary aromatic amines  
GC-MS general scan

**LEACHING/MIGRATION TESTS**

Test Conditions

In duplicate (A and B)

Surface area for test: 1632 cm<sup>2</sup> (two pieces each test)

Test Water

- Origin: Mains water deionized activated carbon filtered.
- Conductivity: <12  $\mu\text{S.cm}^{-1}$
- Chlorination: Nil
- Volume during test: 5.12l
- Surface to volume ratio: 0.32 cm<sup>-1</sup>
- Test Vessel Glass tank
- Temperature °C 22.5 - 22.0

Form No: 005/3a  
Product: Rubber sheet

**LEACHING/MIGRATION TESTS**

**Analytical Results - Specific Determinands**

DETERMINAND		Concentration found ug/l		
		First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
<u>TOC</u>	TEST A	4450	3280	1910
	TEST B	6090	3920	2780
	BLANK A	<200	<200	<200
	BLANK B	<200	<200	<200
<u>PHENOLS</u>	TEST A	<50	<50	<50
	TEST B	<50	<50	<50
	BLANK A	<50	<50	<50
	BLANK B	<50	<50	<50
<u>PRIMARY AROMATIC AMINES</u>	TEST A	<20	<20	<20
	TEST B	<20	<20	<20
	BLANK A	<20	<20	<20
	BLANK B	<20	<20	<20

## LEACHING/MIGRATION TESTS

Analytical Results - GC/MS Analysis

First Stagnation 72 h

Sample Code	Results		
	Scan No.	Compound	Concn. ug/l
TEST A	0038	2-Ethoxy-2-methyl propane	8
	0166	Unknown m/z 59, 57, 29, 93	1
	0186	Unknown m/z 41, 56, 68, 31, 112	3
	0192	2-Methyl pentan-2-ol	2
	0260	Tetrahydro 2,2,5,5-tetramethyl furan	7
	0302	Unknown m/z 87, 43, 55, 101	2
	0388	2-Methyl-2-Hexanol	1
	0440	1-Methoxyethoxy octane	5
	0464	Alkane	1
	0518	Unknown m/z 43, 57, 59, 99, 115 2,5-dimethyl-2-hexanol?	9
	0571	Unknown m/z 43, 58, 69, 75	1
	0638	Unknown m/z 57, 71, 87 128 1-(1,1-dimethylethoxy)2, 2-dimethyl	49
	0652	2,5 dimethyl-2, 5-hexanediol + Unknown	137
	0677	Tetrahydro-2,2,4,4,tetramethyl 3-furanol	2
	0712	Unknown m/z 57, 41, 103, 159	26
	0724	2,5-Dihydroxytetrahydro Furan + Unknown	171
	0750	Unknown m/z 43, 73, 55, 41, 69, 129	7
	0756	Unknown m/z 57, 43, 41, 55, 70, 83, 98	12
	0766	Unknown m/z 57, 59, 41, 43, 71, 102	3
	0792	Unknown m/z 57, 73, 69, 41, 43	13
	0800	Unknown m/z 43, 59, 70, 95, 113 2,5-dimethyl-2,5-hexanediol	6
	0830	2,5-dimethoxy-2,5-dimethyl hexane	18
	0900	Unknown m/z 57, 58, 43, 72	17
	0975	d8 Naphthalene	15
	1000	Unknown m/z 43, 58, 113 Ketone?	17
	1018	Unknown m/z 43, 57, 71, 87 Alkane?	3
	1047	Dodecanone + Unknown	17
	1106	Unknown m/z 43, 71, 59, 111, 129 Alcohol?	2
	1123	Unknown m/z 43, 59, 83, 113, 173	2
	1150	Alkane	2
	1202	Methyl-tolyl-Carbonic acid ester + Unknown	72



Form No: 005/3b Cont'd  
Product: Rubber sheet

LEACHING/MIGRATION TESTS

Analytical Results - GC/MS Analysis

Sample Code	Results		
	Scan No.	Compound	Concn. ug/l
TEST A	1228	Tetrahydro-2, 5-dimethyl-2H Pyranmethanol + Unknown	25
	1236	Unknown m/z 43, 57, 113, 129	25
	1250	Unknown m/z 43, 71, 57, 58, 59 Propanioc acid ester?	4
	1257	Unknown m/z 57, 71, 41, 43, 99, 147, 189	6
	1281	Unknown m/z 43, 113, 45 methyl cyclohexane isomer?	7
	1290	Unknown m/z 43, 113, 45 methyl cyclohexane isomer?	6
	1350	Unknown m/z 57, 45, 69, 89	6
	1383	Unknown m/z 43, 59, 69, 111, 113	2
	1398	Unknown m/z 43, 71, 101, 189	1
	1461	Unknown m/z 60, 42, 122, 196	1
	1563	Unknown m/z 60, 45, 92, 61, 59, 64 Thiolane cpd?	3
	1569	Unknown m/z 60, 45, 92, 61, 210 Thiolane cpd isomer?	2
	1865	Unknown m/z 45, 60, 59, 43, 254	3
	1871	Unknown m/z 149, 45, 91, 254	5
	2046	Unknown m/z 57, 45, 103, 59	25
	2213	Unknown m/z 60, 62, 45, 241, 300	6

## LEACHING/MIGRATION TESTS

Analytical Results - GC/MS Analysis

Third Stagnation 72 h

Sample Code	Results		
	Scan No.	Compound	Concn. ug/l
TEST A	0050	1,2-Dichloroethylene	8
	0087	Methylethylketone	5
	0136	Ethylacetate	4
	0142	Chloroform	7
	0165	Trimethyloxirane	3
	0195	Unknown 59, 73, 55, 43	2
	0304	2-Methoxy-2-methylbutane	7
	0310	Unknown 57, 29, 75, 72	1
	0350	C8H18 isomer	3
	0369	C7H16 isomer	7

Form No: 005/3d  
Product: Rubber sheet

# LEACHING/MIGRATION TESTS

## Analytical Results - GC/MS Analysis

First Stagnation 72 h

Sample Code	Results		
	Scan No.	Compound	Concn. ug/l
TEST B	0042	Propane-2-ethoxy-2-methyl	14
	0169	Propane-2-methyl-2-(1,methylethoxy)	1
	0190	3-Butan-1-ol-3-methyl	4
	0195	2-Pentanol-2-methyl	4
	0252	Peroxide, bis(1,1-dimethylethyl)	1
	0263	Furan tetrahydro 2, 2, 5, 5, tetramethyl	9
	0392	Unknown m/z 59, 101, 45, C8 Alcohol?	2
	0446	Unknown m/z 59, 43, 57, 58	6
	0468	C9 Alkene	1
	0521	Propane 2, 2-methylene bis(oxy)bis-2-methyl?	8
	0574	Unknown m/z 43, 69, 58, 75	3
	0643	Unknown m/z, 57, 71, 87,...129	73
	0656	Unknown m/z 113, 95, 70, 55, 41	151
	0685	Unknown m/z 71, 69, 86, 111	2
	0716	Unknown m/z 57, 41, 103, 159 Ethanol, 2-11 dimethylethyl? And Unknown	29
	0728	Unknown m/z 56, 69, 89, 145	190
	0795	Unknown m/z 73, 57, 41, 69, 117	11
	0812	Unknown m/z 43, 59, 70, 95, 113 2,5 Hexanediol 2,5 dimethyl	7
	0835	Unknown m/z 73, 43, 59, 95, 113 Hexane 2,5 dimethoxy 2,5 dimethyl + Unknown	21
	0905	Unknown m/z 57, 59, 41, 43, 72	25
	1000	Unknown m/z 43, 58, 99, 113	27
	1021	Unknown m/z 43, 57, 71 Alkane?	5
	1053	Unknown m/z 58, 59, 43, 69, 111 Decanol?	117
	1110	Unknown m/z 43, 71, 58, 111, 129	3
	1128	Unknown m/z 43, 59, 83, 113, 173	4
	1153	Alkane	2
	1204	Unknown m/z 91, 122, 162, Carbonic acid methyl M-Tolyl	43
	1239	Unknown m/z 43, 57, 113, 129	19
	1253	Unknown m/z 43, 57, 59, 71, 56, 89 Proprionic acid ester?	4
	1260	Unknown m/z 57, 71, 41, 99	5
	1284	Unknown m/z 43, 113, 45, methyl cyclohexanol cpd?	8
	1294	Unknown m/z 43, 113, 45, methyl cyclohexanol cpd?	9

Form No: 005/3d Cont'd  
Product: Rubber sheet

LEACHING/MIGRATION TESTS

Analytical Results - GC/MS Analysis

First Stagnation 72 h

Sample Code	Results		
	Scan No.	Compound	Concn. ug/l
TEST B	1352	Unknown m/z 45, 57, 69, 89	4
	1566	Unknown m/z 60, 45, 92, 210 Thiolane cpd isomer?	4
	1572	Unknown m/z 60, 45, 92, 61, 210 Thiolane cpd isomer?	7
	1637	Unknown m/z 71, 43, 99, 101, 161	3
	2050	Unknown m/z 57, 45, 103, 59, 29, 101	16

Form No: 005/3e  
Product: Rubber Sheet

# LEACHING/MIGRATION TESTS

## Analytical Results - GC/MS Analysis

Third Stagnation 72 h

Sample Code	Results		
	Scan No.	Compound	Concn. ug/l
TEST B	0773	Unknown (C8H18O isomer ??)	8
	0785	C8H16O isomer ??	1
	0800	Unknown 59, 43, 95, 55 (M + 110)	11
	0840	Unknown 43, 58, 69, 41 ??	2
	0890	Unknown 43, 57, 71, 87, 41 ??	35
	0901	2,2,4,4-Tetramethylethrahydrofuran -3-ol	2
	0915	Unknown 43, 113, 59, 55 (Alcohol??)	120
	0966	C7H16O2 ? Alkoxy compound	30
	0983	Unknown 57, 59, 43, 41 ??	150
	1012	Unknown 57, 43, 41 29 (Alcohol ?)	30
	1027	Unknown 57, 43, 59, 70	3
	1041	2, 5-Dimethyl-2,5-hexanediol ?	16
	1056	Unknown 73, 43, 59, 55 (Alcohol ?)	15
	1097	Unknown 73, 43, 59, 55 (Alcohol ?)	20
	1154	Unknown 43, 57, 59, 55 ??	5
	1179	Unknown Ions 57, 59, 43, 41 (Alcohol ?)	12
	1230-1280	Unknown ions 43, 58, 59, 73	100
	1315	2-Phenoxyethanol	20
	1400	Unknown ions 43, 71, 59, 111 (Branched chain ketone ?)	2
	1531	Unknown ions 43, 57, 113, 41 ??	10
	1540	Sulphur compound 7 Unknown ions 60, 92, 166, 45	22
	1570	Unknown ions 57, 43, 71, 99	2
	1594	Unknown ions 43, 45, 70, 71	5
	1656	Unknown ions 43, 59, 179, 161	5
	1662	Unknown ions 43, 113, 69, 57	6
	1673	Unknown ions 43, 163, 121, 145	3
	1825	Unknown ions 92, 122, 60, 71 ?, 196	1
	1836	Unknown ions 57, 43, 99, 71	2
	1875	Unknown ions 57, 59, 43, 69	1
	1890	Unknown ions 159, 71, 83, 59	1
	1905	Unknown ions 43, 111, 59, 69	1
	1938	Unknown ions 60, 45, 92, 73, 180, 210 - M+	5
	1974	Unknown ions 71, 43, 99, 70	2
	2122	Dibutylphthalate isomer	3
	2255	Unknown ions 45, 60, 61, 92	1
	2255	Unknown ions 45, 60, 61, 92	1
	2390	Unknown ions 57, 45, 103, 59	3
	2737	Unknown ions 60, 45, 61, 73 (Acid ?) 256 M+?	2

LEACHING/MIGRATION TESTS

Migration Values

DETERMINAND Sample code	Migration values $\mu\text{gdm}^{-2} \text{ day}^{-1}$		
	First Stagnation 72h	Second Stagnation 72h	Third Stagnation 72h
<u>TOC</u>			
TEST A	465	343	200
TEST B	637	410	291
BLANK A	<20	<20	<20
BLANK B	<20	<20	<20
<u>PHENOLS</u>			
TEST A	<5	<5	<5
TEST B	<5	<5	<5
BLANK A	<5	<5	<5
BLANK B	<5	<5	<5
<u>PRIMARY AROMATIC AMINES</u>			
TEST A	<2	<2	<2
TEST B	<2	<2	<2
BLANK A	<2	<2	<2
BLANK B	<2	<2	<2

Form No: 005/5  
Product: Rubber sheet

**LEACHING/MIGRATION TESTS**

**Analytical Results - Control Determinands**

DETERMINAND (concentration added µg/l)	Concentration found µg/l		
	First Stagnation T1	Second Stagnation T2	Third Stagnation T3
Primary aromatic amines (500µg/l)	537	532	485