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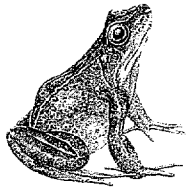
DEVELOPMENT OF A GCMS PROCEDURE FOR IDENTIFICATION OF ORGANIC SUBSTANCES IN MATERIALS TESTING LEACHATES

Final Report to the Drinking Water Inspectorate



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DEVELOPMENT OF A GCMS PROCEDURE FOR IDENTIFICATION OF ORGANIC SUBSTANCES IN MATERIALS TESTING LEACHATES

SUMMARY

Two protocols have been drafted, in BSi format, for the conduct of leaching tests to produce leachates from materials intended for use in contact with drinking water, and for the analysis of these leachates for unspecified organic substances using GCMS. This latter protocol was based on the use of a suite of various types of deuterium-labelled compounds to ensure that the methodology utilised was appropriate to allow the requirements of the protocol to be met.

A within-laboratory performance test of these protocols was undertaken using three materials agreed with the Department. These materials were polyethylene pipe, glass-reinforced polyester (GRP) pipe and bitumen-lined ductile iron pipe. This test demonstrated that, with minimal amendment, the required performance (in terms of limit of detection and relative standard deviations for detected compounds) could be met. The only amendment required was dechlorination of the leachate samples prior to analysis, as it became clear that antioxidants leached from materials could be oxidised by residual free chlorine in the leachates obtained using chlorinated water.

Four laboratories were involved in the subsequent interlaboratory performance testing of the protocols. These were WRc, The Water Quality Centre (Thames Water), KIWA (The Netherlands) and Centre de Recherche et de Controle des Eaux, Paris (CRECEP) (France). This testing showed that comparable results to those from the initial within laboratory testing could be obtained for the internal standards, although difficulties were encountered by two laboratories in detecting the most volatile internal standard. In one case, the chromatographic conditions were such that this internal standard should have been detected, and it therefore appears that the technique used for concentrating the extract prior to GCMS analysis was inappropriate. In the other case, the laboratory concerned did not comply fully with the GCMS protocol. Various criteria, such as the recoveries of the internal standards, the quality of the procedural blanks, the consistency of the ratios of the peak areas of the compounds detected and the internal standards, and the consistency of the mass spectra obtained were used to evaluate the performance of the protocols.

Provided they are strictly adhered to, the protocols are satisfactory, in that they allowed organic compounds added to leachates from materials at known levels to be reliably determined at concentrations in the range 1-10 $\mu g\ l^{-1}$. The limit of detection was about 0.75 $\mu g\ l^{-1}$. However, for compounds leached from the materials being tested the performance was more difficult to assess and there were discrepancies between laboratories, both in terms of compounds detected and in quantifying these compounds. Some of discrepancies may have been due to an increased variation arising from the leaching process itself, and some problems were caused by difficulties in assessing whether compounds detected in both leachates and blanks were classified as originating from the materials tested.

Some recommendations are given regarding possible improvements to the protocols.

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1. INTRODUCTION

New materials (e.g. pipes or pipe-lining materials) designed to come into contact with drinking water, must be approved by the Secretary of State under Regulation 25(1)(a) of the Water Supply (Water Quality) Regulations 1989. Tests on these materials are requested by the Committee which advises the Secretary of State (CCM) in order to provide both qualitative and quantitative information on compounds which may leach from them. If some constituents of the materials are of particular concern, it may be necessary to apply specific analytical methods for their quantification in leachates produced. The CCM may also request analysis using a basic gas chromatography-mass spectrometry (GCMS) method, intended to detect, identify and quantify unsuspected contaminants in materials leachates.

The current recommended method is described in Annex F of the Drinking Water Inspectorate's (DWI's) Guidance Note on the Approval of Substances and Products used in the Provision of Public Water Supplies. This method was developed several years ago by WRc, and outlines the general procedures to be used, but there may be considerable variation in the way in which different laboratories undertaking the analysis apply these procedures. Harmonised European standards for the testing of products for use in contact with potable water are under development in CEN. When agreed, these standards must be published as national standards within the EU and EFTA countries. In order to promote the adoption of the concept that general survey GCMS analysis should be a requirement for this type of materials testing, standardised procedures of known performance are required. The Drinking Water Inspectorate (DWI) therefore issued an invitation to tender for a consultancy contract on the development of protocols for leaching tests and an analytical method involving GCMS for the identification of organic substances in leachates produced from materials intended for use in contact with drinking water. WRc were chosen to undertake the work.

The objectives of the work were as follows:

a) to draft methods for the conduct of the leaching test and analysis by GCMS, based on the procedures described in Annex A of the tender documents (Annex F of the DWI Guidance Note on the Approval of Substances and Products used in the Provision of Public Water Supplies);

b) to ensure that the test procedures are described precisely and unambiguously in accordance with the requirements set out in BS 0, Part 3, 1991 (A Standard for Standards);

c) to establish the limits of deviation and detection for specific substances in three test materials to be specified by the Department's Nominated Officer;

d) conditional upon the demonstration of satisfactory performance characteristics following initial testing, to arrange collaborative testing of the three test materials in four different laboratories, one of which would be the contractor's laboratory and

two of which would be located outside of the United Kingdom but within a member state of the European Union;

e) to evaluate and report upon the performance of the test methods within the participating laboratories; and

f) conditional upon the completion of the collaborative trial confirming the achievement of acceptable performance characteristics by the participating laboratories, to attend two briefing meetings with BSi Committees to present the draft BS procedures, and to respond to any comments made by the Committees.

As the within-laboratory and interlaboratory performance testing generated significant quantities of data from a total of 80 GCMS runs, this report generally contains summaries of these data in Appendices C (within-laboratory performance testing) and D (interlaboratory performance testing). The data tables from the interlaboratory performance testing are given in Appendix E, which forms a separate volume of this report. The complete data reported by each laboratory, including mass spectra for all of the compounds detected in each GCMS run will be held, for reference, at WRc.

2. WORK PROGRAMME

2.1 Protocols

The initial requirement was to draft two protocols, one for the leaching test and the other for the GCMS analysis of the leachate produced.

Laboratory leaching tests on materials in contact with drinking water usually consist of sequential stagnation periods, during which a test sample (of specified surface area) of the product under test is in contact with a specified volume of test water. Contaminants leaching from the material during that period are then determined in the test water. The test protocol described in Annex F of the Guidance Note on the Approval of Substances and Products used in the Provision of Public Water Supplies specifies three sequential stagnation periods of 24, 48 and 72 hours. The test protocol currently under development through CEN is based on three 72 hour periods.

It was considered inappropriate to draft a leaching test protocol for all types of materials which would require testing for CCM purposes. The main purpose of the protocol, in this instance, was to ensure as far as possible that identical leachates were produced by each laboratory undertaking GCMS analysis of the leachates. The protocol is therefore limited to non-metallic (non-cementitious) factory-produced products. A single leaching period of 72 hours was chosen, as slight variations (e.g. ± 1-2 hours) would be unlikely to be significant.

The protocol for the GCMS analysis covers the internal standards to be used, the extraction technique, the GCMS operating parameters, the production of the required GCMS outputs and the basis for quantification. It was considered desirable to draft this protocol to allow various manufacturers equipment to be used. Therefore the required performance of the method is generally based on the internal standards rather than the equipment, although some minimal equipment specifications are included.

These protocols are presented in Appendix A (leaching tests) and Appendix B (GCMS general survey analysis) of this report.

2.2 Materials used for performance testing of the protocols

Following consultation with the Nominated Officer, three materials were chosen to check the performance of the protocols. These were as follows:

25 mm (nominal) ID polyethylene pipe, obtained from and manufactured by BP;

50 mm (nominal) ID glass-reinforced polyester pipe (GRP), obtained from and manufactured by Deutsche Fibercast GmbH; and

100 mm (nominal) ID bitumen-lined ductile iron pipe, obtained from Thames Water and manufactured by Stanton and Staveley.

Sufficient lengths of each of these pipes were obtained to allow all of the experimental work (within-laboratory and interlaboratory performance testing) to be undertaken with the same batch of each of the materials. The polyethylene pipe was donated by BP.

2.3 Laboratories undertaking performance testing

An initial assessment of the performance of the two protocols was undertaken at WRc. The subsequent interlaboratory performance testing was undertaken by WRc and the following laboratories, chosen on the basis of their considerable experience of the use of GCMS for the analysis of leachates from materials:

The Water Quality Centre (Thames Water) (UK);

Centre de Recherche et de Controle des Eaux (CRECEP) (Paris, France); and

KIWA N.V. (Nieuwegein, The Netherlands).

3. RESULTS

3.1 Protocols

3.1.1 Leaching tests

This protocol is presented in full in Appendix A.

The principle is as follows:

"Following a pre-washing period, the pipe test sample is filled with test water with which it is kept in contact for 72 hours at 25°C. The resulting leachate, and an appropriate corresponding procedural blank, are then analysed in accordance with the protocol 'GCMS general survey procedure for the analysis of leachates produced from leaching tests conducted on materials for use in contact with drinking water'. For each product leachates with chlorinated and unchlorinated test water are prepared."

The protocol provides specifications for the test water and chlorinated test water, detailed instructions for the leaching tests, and the defined requirements for a test report on the leaching tests.

No amendments to this protocol were deemed necessary following the initial within laboratory testing undertaken by WRc.

The protocol was supplied to the laboratories carrying out the inter-laboratory testing several weeks prior to the commencement of these tests, to allow time for familiarisation with the contents and resolution of any uncertainties regarding its application.

3.1.2 GCMS protocol

This protocol is given in full in Appendix B.

The principle is as follows:

"Leachate samples and procedural blanks obtained using chlorinated test waters should be dechlorinated immediately following the completion of the leaching test, to prevent any change in the concentration of compounds leached due to the continued presence of residual free chlorine.

The leachate sample(s) and appropriate procedural blanks are spiked with the mixture of isotopically labelled internal standard compounds, and solvent extracted with dichloromethane. The extract is concentrated and analysed by GCMS. The mass spectrometer is used in a repetitive full scan mode (mass range 20-700 amu) and the mass spectra produced recorded by, and stored on, the GCMS data system.

A range of internal standards is added to the samples at known levels in order to (a) allow compounds detected to be quantified, and (b) to provide quality control. This latter aspect is particularly important in this type of analysis, where the compounds to be analysed are not known until the analysis has been undertaken. The internal standards are chosen to represent various types of compounds which may typically be present in water samples, and isotopically labelled compounds are used to ensure that the internal standards are only present in the samples due to deliberate addition. Provided that the standards are carefully selected they may be distinguished mass spectrometrically from any naturally occurring analogues which may be present.

Whenever possible each compound detected is identified. The methods used to identify organic compounds from their mass spectra do not form part of this protocol, but further information on this subject is provided in the Appendix. Each compound detected may be quantified by reference to the isotopically labelled internal standards."

Several of the internal standards selected were those listed in Annex F of the DWI Guidance document. However two additional new internal standards were included, as experience of analysing leachates from various materials suggested that it would be useful to have an internal standard which was analogous to an anti-oxidant, and a late-eluting (i.e. high boiling point) internal standard, as many compounds present in leachates eluted a considerable time after the least volatile of the standards listed in Annex F (d_{10}-phenanathrene) in a general survey GCMS run. The two additional standards were d_{20}-BHT (d_{20}-2,6-di-t-butyl-4-methyl-phenol) and d_{62}-squalane (d_{62}-2,6,10,15,19,23-hexamethyltetracosane). The total list of internal standards was as follows:

d_6-benzene
d_{20}-BHT
d_5-chlorobenzene
d_{34}-hexadecane
d_8-naphthalene
d_{10}-phenanthrene
d_5-phenol
d_{62}-squalane
d_{10}-p-xylene

Two different internal standard solutions are used in the protocol. Both of these contain all of the compounds listed above, but the concentrations of the compounds differ. One solution (in which the concentrations of the internal standards are in the range 1-16 ng μl^{-1} is used to ensure that the chosen GCMS conditions meet the specification of the protocol and that the GCMS system is performing satisfactorily. The other internal standard solution (in which the concentrations of the internal standards are in the range 5-80 ng μl^{-1}) is used as a spiking solution to add the internal standards to the samples at the correct concentration.

The concentration of the internal standards in the spiking solution, the volume of this solution added to each sample, and the sample volume are such that three of the internal standards (d_{34}-hexadecane, d_8-naphthalene, and d_{10}-p-xylene) are present in the sample at 0.5 $\mu g\ l^{-1}$, three (d_6-benzene, d_5-chlorobenzene and d_{10}-phenanthrene) are present at 2 $\mu g\ l^{-1}$, and the remaining three (d_{20}-BHT, d_5-phenol and d_{62}-squalane) are present at 8 $\mu g\ l^{-1}$. These levels were chosen to allow the protocol to be performance tested over the range 0-10 $\mu g\ l^{-1}$, and to establish the limit of detection.

A check on the effectiveness of the extraction and concentration procedures during the production of the concentrated extract for GCMS from the leachate sample is provided by the requirement that the recovery of three of the internal standards (d_8-naphthalene, present at 0.5 µg l^{-1}; d_{10}-phenanthrene, present at 2.0 µg l^{-1}; d_{62}-squalane, present at 8.0 µg l^{-1}) must be in excess of 50%.

Operating conditions for the GCMS system are generally based on the internal standards used e.g. the most volatile internal standard (d_6-benzene) must be detected and separated from the solvent, and the least volatile internal standard (d_{62}-squalane) must elute after 35 minutes. This requirement places constraints on the initial temperature of the GC column, and it is now specified (following the inter-laboratory testing) that the temperature programming rate cannot be greater than 8 °C min^{-1}, so that inappropriate conditions cannot be used. Certain parameters are specified in terms of instrumental performance e.g. the mass spectrometric resolution must be >700. This specification is based on the knowledge that organic compounds with molecular weights up to 700 amu can be detected in leachates from materials using GCMS. Unless the mass spectrometric resolution is >700, the mass spectra obtained for such compounds are likely to be incorrect (the high mass ions may be incorrectly mass measured).

The performance of the GCMS system is monitored by ensuring that the response obtained from a solution of the internal standards (the GC column check standard) does not vary significantly (<30%) during the course of the analyses conducted, and that the asymmetry of the peaks obtained for two of the internal standards (d_8-naphthalene and d_5-phenol) is acceptable. The GC column check standard must be analysed prior to the analysis of extracts from leachates, to demonstrate that the GCMS system performs satisfactorily, and after every six extracts to ensure that the performance does not become unacceptable during the course of a series of analyses.

It is considered that the specifications in the protocol should be met without difficulty by most laboratories undertaking GCMS analysis for materials testing purposes, although it is recognised that some may need to gain additional experience of appropriate concentration techniques to ensure adequate recovery of all of the internal standards.

The information which should be presented in the test report is specified in the protocol. Again, meeting these requirements should not present problems to competent laboratories.

It was recognised that there could be a need for modifications to the protocol after the within-laboratory and interlaboratory performance testing.

3.2 Within-laboratory performance testing of protocols

Following the protocol (3.1.1; Appendix A), leachates (one using chlorinated water, one using unchlorinated water) were produced from each of the three chosen materials on two separate occasions. This gave twelve leachates. Blanks were also produced on each occasion for each water type, a total of four. Thus sixteen leachate samples were submitted for GCMS analysis.

Concentrated extracts (in dichloromethane) were produced from the leachate samples, following the procedures in the GCMS protocol (3.1.2; Appendix B). These extracts were then examined by GCMS, using conditions which complied with the requirements of the protocol.

As noted above (3.1.2), internal standards were added to all of the samples at known levels to allow the protocols to be performance tested. Compounds leached from the materials tested could not be used for this purpose as their true concentration in the leachates was not known. The range of interest for the performance testing had been defined as 0-10 μg^{-1}, and it was necessary to demonstrate that a detection limit of $<1\ \mu g\ l^{-1}$ could be achieved. The internal standards added to the samples at 2 $\mu g\ l^{-1}$ (d_6-benzene, d_5-chlorobenzene and d_{10}-phenanthrene) represent the lower end of the range of interest, those added at 8 $\mu g\ l^{-1}$ (d_{20}-BHT, d_5-phenol and d_{62}-squalane) represent the higher end of the range of interest, and those added at 0.5 $\mu g\ l^{-1}$ (d_{34}-hexadecane, d_8-naphthalene and d_{10}-p-xylene) were used to establish the limit of detection.

The data for the internal standards obtained from the GCMS runs are presented in Appendix C, and is summarised below. The data tables for each GCMS run, which include details of all of the compounds detected, are also included in Appendix C.

The requirement to demonstrate that satisfactory recoveries (>50% for three specified internal standards) had been achieved during the extraction/concentration steps of the analysis was satisfied. The average recoveries were as follows:

- d_8-naphthalene - 55%
- d_{10}-phenanthrene - 69%
- d_{62}-squalane - 77%

The relative standard deviation (RSD) of the peak areas for the internal standards ranged from 21% (for d_{10}-phenanthrene, present at 2.0 $\mu g\ l^{-1}$) to 49% (for d_{20}-BHT, present at 8.0 $\mu g\ l^{-1}$). For the internal standards present at 0.5 $\mu g\ l^{-1}$ (d_{10}-p-xylene, d_8-naphthalene and d_{34}-hexadecane) the RSD was between 28% and 32%. Based on these RSD values, the limit of detection (LOD; calculated on the basis of 4.65 × standard deviation) for these three internal standards is about 0.75 $\mu g\ l^{-1}$, which is below the desired LOD (1 $\mu g\ l^{-1}$). Ignoring the high RSD for d_{20}-BHT (which is addressed below), the RSD values for the remaining internal standards present at either 2.0 or 8.0 $\mu g\ l^{-1}$ were between 21% and 42%, which is considered acceptable for this type of complex analysis.

In terms of quantification of other compounds detected in GCMS runs on extracts from leachate samples, the likely RSD has been calculated by ratioing the responses of the internal standards to the response obtained for d_{62}-squalane, and determining the RSD for each of these ratios. This suggests that RSD values in the range 25-46% are obtained when using a response from an internal standard to quantify another detected compound.

The high RSD found for d_{20}-BHT was initially considered surprising, but on closer examination it was apparent that on average the response was considerably lower (by about 25%) for the leachates produced using chlorinated water, compared to the leachates produced using unchlorinated water. Indeed, for one chlorinated water leachate, no d_{20}-BHT was detected. Also the RSD for d_{20}-BHT (64%) was higher in the chlorinated water leachates than in the unchlorinated water leachates (35%). It was considered that this problem with d_{20}-BHT was caused by residual free chlorine in the chlorinated water leachates, which was oxidising this internal standard. The time delay between spiking the leachate samples and the extraction step was always less than thirty minutes, so this reaction is obviously rapid. This finding suggests that unless dechlorination is included in the procedure, extremely variable results will be obtained for any antioxidants present in leachates produced using chlorinated water. Therefore, prior to the interlaboratory performance testing, the GCMS protocol was amended to include a dechlorination step, so that any residual free chlorine was removed before addition of the internal standards.

On the basis of these results, it was recommended that the interlaboratory performance testing of the protocols should proceed, provided a dechlorination step was incorporated into the GCMS protocol. This recommendation was agreed by DWI.

3.3 Interlaboratory performance testing of protocols

The laboratories involved in the interlaboratory performance testing of the protocols were sent copies of the two protocols (Appendix A; Appendix B) several weeks prior to the commencement of the exercise, so that staff involved with this work had an opportunity to study them and query any ambiguities or points that were unclear. Examples of the output reports that were specified in the protocols were included.

Some telephone discussions took place regarding potential difficulties relating to the identification of compounds detected by GCMS in the concentrated extracts from the materials leachates. It was agreed that since the primary aim of the exercise was to establish the performance of the protocols, the identity of the compounds detected was of secondary importance. The GCMS protocol includes a requirement to supply a mass spectrum of each compound detected which is considered to have leached from the test material, so it would be possible to carry out comparisons of the data produced by each laboratory (simply by checking the mass spectra to ensure that the same compounds had been detected) even if some of the detected compounds were not identified.

Following the agreement of the Department to the interlaboratory exercise, sufficient of each of the three materials to carry out the leaching tests was sent to each laboratory. Additionally, two solutions of the mixtures of internal standards were sent to the participants. One solution contained the internal standards at the correct concentration for use in checking that the GCMS system operating conditions conformed to the specification in the GCMS protocol, and the other solution contained the internal standards at the correct concentration for spiking the leachate samples prior to applying the extraction/concentration procedures described in the GCMS protocol.

Following the completion of the performance testing exercise, the results were sent to WRc for collation and assessment in terms of:

- recoveries of the internal standards;
- limits of detection for the internal standards;
- quality of the procedural blanks;
- consistency of the retention times of the internal standards and peaks detected;
- consistency of the ratios of peak areas of the compounds detected and the internal standards;
- consistency of the mass spectra;
- consistency of identification of the peaks detected.

3.3.1 Results produced by WRc

The protocol for the production of leachates (Appendix A) was used, as for 3.2, resulting in sixteen samples for GCMS analysis. These were then analysed using the amended GCMS protocol, with addition of ascorbic acid solution to dechlorinate the samples as soon as the leaching period was over.

The data for the internal standards obtained from the GCMS runs are presented in Appendix D, while the data tables for each GCMS run are presented in Appendix E (a separate volume of this report).

Generally, compared to the initial within-laboratory performance testing, there was an improvement in the data produced. The RSD of the peak areas for the internal standards ranged from 15% (for d_5-chlorobenzene and d_{10}-phenanthrene in the first batch of samples analysed) to 52% (for d_{34}-hexadecane in the first batch). Obviously the inclusion of the dechlorination step resulted in the marked change noted for d_{20}-BHT (RSD reduced from 49% to 16% (batch 1)-23%(batch 2)). The recoveries of the specified internal standards were also improved.

The quality of the procedural blanks was considered acceptable, although a series of hydrocarbons was detected at low levels. It is known that there was a laboratory problem at the time relating to the cleanliness of the glass-wool used to remove any ice frozen out of the solvent extracts prior to their concentration. There was a marked improvement in the chlorinated blanks, as no chlorinated artefacts (produced by reaction of residual free chlorine with a stabiliser (amylene) in the dichloromethane used for the solvent extraction) were detected.

The retention times of all the internal standards were consistent to within a few seconds. When the variation was expressed as a percentage, the coefficients of variation (%RSD)

were in the range 0.02-0.20%. The consistency of quantification of detected compounds was generally as good as expected, with RSDs of about 50%, but there was some inconsistencies in the detection of compounds present at concentrations below 1 µg l^{-1}. Similarly, for compounds apparently present at less than 1 µg l^{-1}, there were some inconsistency in the mass spectra obtained. For compounds present at higher levels this was not a problem, although it was noted that for compounds present at levels >100 µg l^{-1} the mass spectra were beginning to saturate.

3.3.2 Results produced by The Water Quality Centre

The data obtained for the internal standards from the GCMS runs are presented in Appendix D, while the data tables listing the compounds detected in each GCMS run are in Appendix E.

Peak areas were provided for all of the internal standards in most of the GCMS runs on the extracts. The only exception was d_8-naphthalene which could not be quantified in the extracts from the leachates from the GRP pipe. This was due to the fact that this standard co-eluted with benzoic acid, which was present in these leachates at very high levels. Generally, the %RSD of the peak areas for the internal standards were within the range 15-51%, although the majority were within the range 15-40%. The %RSD for the internal standards present at 0.5 µg l^{-1} are consistent with a detection limit of <1 µg l^{-1}. The retention times of the internal standards were consistent, with %RSD being between 0.07% and 1.06%.

There were some inconsistencies in the identification of compounds detected in the leachates, particularly when the levels were <1 µg l^{-1}. This is assumed to be partly due to the fact that one analyst undertook the mass spectral data interpretation for the "Batch 1" samples, while another analyst independently interpreted the "Batch 2" data.

3.3.3 Results produced by CRECEP

The data obtained for the internal standards from the GCMS runs are presented in Appendix D; the data tables listing the compounds detected in each GCMS run are in Appendix E.

Unfortunately this laboratory did not fully comply with the GCMS protocol, either for the conditions to be used for the GCMS analysis, or with respect to the reporting requirements. In the former case, the GC column used for the analysis was only 25 m long, rather than at least 50 m long, as specified in the protocol and although the requirement that the retention time for the least volatile internal standard (d_{62}-squalane) should be at least 35 minutes was met, the means of achieving this were inappropriate. As a result, the most volatile internal standards (d_6-benzene) could not be detected, even in standard solutions.

The reporting requirements of the protocol demand that the mass spectra of all of the compounds detected in the extracts of the various leachates, which are considered to have

arisen from the material tested, should be included in the report detailing the results of the test. Also, where compounds could not be identified, the tabulated results should have reported the four most intense peaks in the mass spectrum, in decreasing order of intensity. CRECEP only provided some examples of the mass spectra obtained, and did not list the most intense peaks in the mass spectra of the unknowns. This made it impossible to satisfactorily compare their data with data from other participating laboratories.

From the data submitted the variation in the recoveries of the internal standards, expressed as %RSD, ranged from 10% to 96% and as already noted d_6-enzene was not detected. The reproducibility of the GC retention times of the internal standards was affected by the fact that different GC columns were used for some of the work. However for the GCMS runs for the extracts from the "Batch 2" samples, the %RSD for the internal standards detected was in the range 0.06-1.01%.

3.3.4 Results produced by KIWA

The data obtained for the internal standards from the GCMS runs are presented in Appendix D and the data tables listing the compounds detected in each GCMS run are in Appendix E.

KIWA did not fully comply with the requirements of the protocol, as the most volatile internal standard (d_6-benzene) was not detected in the extracts from the leachates. As this standard could be detected by them in GC standard solutions, it is assumed that this was due to its loss during the concentration procedure employed. KIWA also provided results as means of results from the two batches of leachates, so it is not possible to assess between batch variability. Additionally, as Kovats indices were used rather than retention times, it has not been possible to assess the variability of the absolute retention times. However, the variability of the responses for the internal standards present at 0.5 $\mu g\ l^{-1}$ was good (within the range 8-15%), which indicates that the limit of detection of <1 $\mu g\ l^{-1}$ was achieved.

4. DISCUSSION

The objective of this work was to establish whether the written protocols, when used by laboratories with experience in materials testing, would ensure that consistent results could be achieved for organic compounds leached from materials under test. The results produced have been assessed to gauge how far this aim has been achieved.

For the internal standards, which were the only compounds present at known levels, the performance of the protocols in terms of limits of detection and performance in the range 1-10 $\mu g\ l^{-1}$ have been assessed. Attempts have also been made to assess the performance of the protocols in relation to compounds leached from the materials under test, the criteria used being the peaks detected in the extracts from the leachates and their identities, their reported concentrations and the quality of the mass spectra.

On the basis of the results for the internal standards, three of which were present at 0.5 $\mu g\ l^{-1}$ three at 2 $\mu g\ l^{-1}$ and three at 8 $\mu g\ l^{-1}$, the limit of detection (based on 4.65 × the standard deviation of the internal standards present at the lowest concentration) found by three of the laboratories was less than 1 $\mu g\ l^{-1}$. For the remaining internal standards, which covered the range of interest (1-10 $\mu g\ l^{-1}$), the relative standard deviations found by these three laboratories were in the range 8-52%, the majority being in the range 15-30%. However, one of these laboratories did not detect the most volatile internal standard (d_6-benzene) and therefore did not comply fully with the protocol. This laboratory also reported the reported the results as means of the two batches, and therefore the %RSD values are not strictly comparable to those reported by the other laboratories. The results from the fourth laboratory were not satisfactory, either with respect to the limit of detection or performance in the range 1-10 $\mu g\ l^{-1}$, but as the GCMS protocol was not followed (e.g. the GC column used was only 25 m long, rather than at least 50 m; the GC temperature programme was inappropriate) this is perhaps not surprising and does serve to illustrate the fact that following the protocol does lead to better results. The results for the internal standards are summarised in Table 4.1, and are given in full in Appendix D.

The data have also been assessed with respect to the compounds detected in the extracts from the leachates. However this assessment is not as straightforward as that of the performance for the internal standards, as it is not known with certainty which of the detected compounds are derived from the materials under test, and their true concentrations are not known. The variability of the leaching process will also affect these data.

For simplicity, and because the limit of detection required by the general survey GCMS protocol was 1 $\mu g\ l^{-1}$, only compounds with reported concentrations $\geq 1\ \mu g\ l^{-1}$ were considered. Lower reported concentrations were considered where the same compound was also reported in another leachate from the same product at a concentration above 1 $\mu g\ l^{-1}$. Tables 4.2 to 4.4 summarise the number of peaks detected whose concentrations were above 1, 5, 10, and 100 $\mu g\ l^{-1}$ in each leachate extract, and considered by the testing laboratory to originate from the test samples. Tables 4.5 to 4.10 show the compounds

Table 4.1 Summary of variation (%RSD) for internal standards in interlaboratory performance testing

Laboratory	Batch number	%RSD for peak areas of internal standards								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
WQc	1	16	15	37	24	18	23	52	15	26
	2	25	19	27	51	42	16	21	19	21
WRc	1	30	19	26	24	21	11	19	14	19
	2	15	20	19	35	19	14	32	15	30
CRECEP	1		15	68	32	27	36	24	10	33
	2		35	27	63	44	53	84	36	96
KIWA	1/2		10	8	12	12	14	15	9	47

Table 4.2 Summary of GC-MS peaks detected in leachates from the polyethylene pipe

		Number of peaks detected at concentration					
Product	Leachate	>1 µg l^{-1}	>5 µg l^{-1}	>10 µg l^{-1}	>100 µg l^{-1}	Named	Unknown
PE-U	WRcI-a	3	0	0	0	1	2
	-b	8	0	0	0	0	8
	II-a	3	0	0	0	0	3
	-b	2	0	0	0	0	2
	WQC-a	9	1	0	0	6	3
	-b	4	0	0	0	3	1
	CRECEP-a	5	2	0	0	3	2
	-b	2	0	0	0	2	0
	KIWA-a/b	5	0	0	0	2	3
	MIN	2	0	0	0		
	MAX	9	2	0	0		
	MEAN	4.6	0.3	0	0		
PE-Cl	WRcI-a	9	3	0	0	5	4
	II-a	2	0	0	0	0	2
	-b	1	0	0	0	1	0
	WQC-a		0	0	0	2	2
	-b	15	2	0	0	10	5
	CRECEP-a	2	1	0	0	2	0
	-b	2	1	1?	0	1	1
	KIWA-a/b	1	0	0	0	1	0
	MIN	1	0	0	0		
	MAX	15	3	1?	0		
	MEAN	4.5	0.9	0.1	0		

Table 4.3 Summary of GC-MS peaks detected in leachates from the GRP pipe

		Number of peaks detected at concentration					
Product	Leachate	>1 µg l^{-1}	>5 µg l^{-1}	>10 µg l^{-1}	>100 µg l^{-1}	Named	Unknown
GRP-U	WRcI-a	9	3	3	0	5	4
	-b	10	8	6	0	7	3
	II-a	9	2	2	0	5	4
	-b	8	2	2	0	4	4
	WQC-a	12	6	2	1	8	4
	-b	19	6	4	0	14	5
	CRECEP-a	2	2	2	1	2	0
	-b	4	3	1	1	4	0
	KIWA-a/b	4	3	0	0	4	0
	MIN	2	2	0	0		
	MAX	19	8	6	1		
	MEAN	8.6	3.9	2.4	0.3		
GRP-Cl	WRcI-a	11	6	5	1	7	4
	-b	13	8	7	1	9	4
	II-a	8	4	2	1	5	3
	-b	7	2	2	1	4	3
	WQC-a	19	10	6	1	12	7
	-b	21	8	4	1	15	6
	CRECEP-a	6	4	2	1	6	0
	-b	4	4	1	1	4	0
	KIWA-a/b	11	4	1	0	7	4
	MIN	4	2	1	0		
	MAX	21	10	7	1		
	MEAN	11.1	5.6	3.3	0.9		

Table 4.4 Summary of GC-MS peaks detected in leachates from the bitumen-lined ductile iron pipe

Product	Leachate Unknown	>1 µg l^{-1}	>5 µg l^{-1}	>10 µg l^{-1}	>100 µg l^{-1}	Named	Unknown
Bitumen-U	WRcI-a	0	0	0	0	0	0
	-b	0	0	0	0	0	0
	II-a	1	0	0	0	0	1
	-b	0	0	0	0	0	0
	WQC-a	5	2	1	0	5	0
	-b	10	1	0	0	6	4
	CRECEP-a	0	0	0	0	0	0
	-b	0	0	0	0	0	0
	KIWA-a/b	4	1	0	0	3	1
	MIN	0	0	0	0		
	MAX	10	2	1	0		
	MEAN	2.2	0.4	0.1	0		
Bitumen-Cl	WRcI-a	2	0	0	0	1	1
	I-b	3	1	0	0	2	1
	II-a	0	0	0	0	0	0
	-b	1	0	0	0	0	1
	WQC-a	13	2	0	0	10	3
	-b	13	0	0	0	11	2
	CRECEP-a	0	0	0	0	0	0
	-b	0	0	0	0	0	0
	KIWA-a/b	5	0	0	0	3	2
	MIN	0	0	0	0		
	MAX	13	3	0	0		
	MEAN	4.1	0.3	0	0		

Table 4.5 Compounds leached from polyethylene - chlorinated leachate

Compound detected	WRc I-1	WRc II-1	WRc II-2	WQC-1	WQC-2	CRECEP-1	CRECEP-2	KIWA-1+2
2-Chloroethanol-(U)	NR	NR	NR	NR	1.2	NR	NR	NR
1,2 Dichloropropane-(U)	NR	NR	NR	2.3	NR	NR	NR	NR
Chloro-t-butyl ether	NR	NR	NR	NR	3.2	NR	NR	NR
Dimethylester-butandioic acid-(U)	5.2	ND	ND	2.1	1.4	1.9	1.3	0.6
2,6-Di-t-butyl-p-benzoquinone-(U)	1.1	NR	1.5	0.5	1	NR	0.6	1.2
2,4-Bis(1,1-dimethylethyl)phenol-(U)	NR	NR	NR	NR	NR	6.4	NR	NR
Dodecanoic acid	NR	NR	NR	NR	1.4	NR	NR	NR
Diethyl pthalate ester-(U)	NR	NR	NR	1.1	3.3	NR	NR	NR
4-Hydroxy-3,5-di-tert-butylbenzaldehyde-(U)	NR	NR	NR	NR	NR	0.3	0.3	NR
Tetradecanoic acid	NR	NR	NR	NR	2.1	NR	NR	NR
Bis(dimethylethyl)methyl phenol	NR	NR	NR	NR	NR	0.7	0.8	NR
Methyl-3-(3,5-ditertbutyl-4-hydroxyphenyl)propionate-(U)	NR	NR	NR	NR	NR	0.7	0.6	NR
Hexadecanoic acid	NR	NR	NR	NR	5.3	NR	NR	NR
Octadecanoic acid	NR	NR	NR	NR	1.5	NR	NR	NR
Palmitic acid	1.3	NR(BK)	NR(BK)	NR	NR	NR	NR	NR
2,2-Methylenebis(1,1-dimethylethyl)-4-methylphenol-(U)	NR	NR	NR	0.4	2.7	NR	NR	NR

NR - not reported

(U) - compound also detected in the unchlorinated extracts

NR(BK) - detected in the extract but judged to be a contamination

WRc I -1/2 - batches 1 and 2 - within laboratory tests

WRc II -1/2 - batches 1 and 2 - interlaboratory tests

WQC/CRECEP-1 or -2 - batches 1 or 2 - interlaboratory tests

KIWA 1 + 2 - results reported as means

Table 4.6 Compounds leached from polyethylene - unchlorinated leachate

Compound detected	WRc I-1	WRc I-2	WRc II-1	WRc II-2	WQC-1	WQC-2	CRECEP-1	CRECEP-2	KIWA-1+2
2-Chloroethanol-(Cl)	NR	NR	NR	NR	NR	2.5	NR	NR	NR
1,2 Dichloropropane-(Cl)	NR	NR	NR	NR	1.6	NR	NR	NR	NR
Dimethylester-butandioic acid-(Cl)	1.1	{1.2}	{1.3}	{1.2}	2.5	0.3	8.6	1.5	0.5
1-H-isoindole-1,3-(2H)-dione	NR	NR	NR	NR	0.4	1.2	NR	NR	0.3
2,6-Bis(1,1-dimethylethyl)-2,5-cyclohexadienone-(Cl)	NR	NR	NR	NR	0.3	0.7	NR	0.8	{0.7}?
2,4-Di-tert-butyl phenol	NR	NR	NR	NR	NR	1.3	3.6	{1.9}Blk	4.4
Diethyl pthalate ester-(Cl)	NR	NR	NR	NR	0.7	2.7	NR	NR	NR
4-Hydroxy-3,5-di-tert-butylbenzaldehyde-(Cl)	NR	NR	NR	NR	{0.9}	1.8	0.5	0.5	NR
Dibutylethyl phenol	0.6	{1.1}	{0.4}	{0.6}	NR	NR	NR	NR	{1.0}
Dibutylpropyl phenol	0.9	{1.4}	{0.3}	{0.5}	NR	{2.2}	NR	NR	{1.1}
Methyl-3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate-(Cl)	NR	NR	NR	NR	2.8	3.3	NR	1.1	NR
Hexadecanoic acid dioctyl ester	NR	NR	NR	NR	NR	NR	1.3	NR	NR
2,2-Methylenebis(1,1-dimethylethyl)-4-methyl phenol-(Cl)	NR	NR	NR	NR	NR	2.7	NR	NR	NR

NR - not reported

(Cl) - also detected in chlorinated extracts

Blk - detected in the extract but judged to be a contaminant

{x.y} - reported as an unknown with a comparable GC retention

WRc I -1/2 - batches 1 and 2 - within laboratory tests

WRc II -1/2 - batches 1 and 2 - interlaboratory tests

WQC/CRECEP-1 or 2 - batches 1 or 2 - interlaboratory tests

KIWA 1+2 - results reported as means

Table 4.7 Compounds leached from GRP - chlorinated leachate

Compound detected	WRc I-1	WRc I-2	WRc II-1	WRc II-2	WQC-1	WQC-2	CRECEP-1	CRECEP-2	KIWA-1+2
Toluene (Methyl benzene)-(U)	41.5	39.1	2.7	2.0	NR	3.9	NR	NR	NR
1,2-Dichloropropane	NR	NR	NR	NR	1.7	NR	NR	NR	NR
m-Xylene-(U)	1.0	NR	NR	NR	NR	NR	NR	NR	NR
3,3,5-Trimethylcyclohexanone-(U)	NR	NR	NR	NR	6.4	4.6	1.8	6.9	NR
Benzaldehyde-(U)	22.8	24.2	4.7	7.2	15.6	16.5	5.9	9.8	6.8
Isophorone-(U)	5.8	5.7	0.8	1.9	NR	NR	NR	NR	NR
Benzoic acid (+Benzothiazole)-(U)	238.5	98.7	124.8	212.8	315.1	572.9	142.0	151.0	NR
Alkylcyclohexanol-(U)	NR	NR	NR	NR	NR	NR	NR	NR	5.6
Dimethylethyl cyclohexanol ?-(U)	NR	NR	NR	NR	NR	NR	2.5	7.3	NR
Decanoic acid	NR	NR	NR	NR	1.0	0.9	NR	NR	NR
Dimethyl phthalate ester-(U)	1.2	NR	NR	NR	0.7	1.5	4.7	NR	NR
Dodecyl oxirane 57,41,56,83-(U)	9.9	4.5	NR	NR	NR	NR	NR	NR	NR
Dodecanoic acid	NR	NR	NR	NR	2.5	2.9	NR	NR	NR
Diethyl phthalate ester-(U)	NR	NR	NR	NR	1.9	1.2	3.8	NR	NR
Benzoic acid phenyl ester	NR	NR	NR	NR	1.0	0.6	NR	NR	NR
Tetradecanoic acid	NR	NR	NR	NR	2.6	0.8	NR	NR	NR
Bis(methylpropyl)phthalate ester	NR	NR	NR	NR	{0.5}Blk	1.3	NR	NR	NR
Dibutyl phthalate isomer-(U)	24.5	12.8	15.4	14.2	{19.9}Blk	32.8	13.7	NR	8.5
Phthalate-(U)	NR	NR	NR	NR	NR	NR	NR	NR	1.3
Hexadecanoic acid	NR	NR	NR	NR	5.6	5.2	NR	NR	NR
Octadecanoic acid	NR	NR	NR	NR	17.0	1.4	NR	NR	NR
Phthalate-(U)	2.1	{1.0}	NR	NR	3.7	4.1	NR	NR	NR
Carboxylic acid (Rt 35.47)	NR	NR	NR	NR	1.5	NR	NR	NR	NR
N-ethyl pentamide	NR	NR	NR	NR	NR	2.2	NR	NR	NR
N,N-dimethyl stearamide	NR	NR	NR	NR	NR	5.2	NR	NR	NR
Bis(2-ethylhexyl)phthalate	[Blk]	{Blk}	NR	NR	[0.2]Blk	[0.3]Blk	9.7	[2.6]Blk	11.9

NR - not reported

U - also detected in unchlorinated extracts

? - probably the same compound as above

Blk - detected in the extract but judged to be a contaminant

{x.y} - reported as an unknown with a comparable GC retention time

WRc I - Batches 1 and 2 - within laboratory test

WRc II - batches 1 and 2 - interlaboratory tests

WQC/CRECEP-1 or -2 - batches 1 and 2 - interlaboraory tests

KIWA 1 + 2 - results reported as means

Table 4.8 Compounds leached from GRP - unchlorinated leachate

Compound detected	WRc I-1	WRc I-2	WRc II-1	WRc II-2	WQC-1	WQC-2	CRECEP-1	CRECEP-2	KIWA-1+2
Toluene-(Cl)	36.7	53.9	4.7	4.2	NR	NR	NR	NR	NR
m-Xylene-(Cl)	0.5	0.7	NR	NR	NR	NR	NR	NR	NR
3,3,5-trimethylcyclohexanone-(Cl)	NR	NR	NR	NR	6.8	5.8	2.0	2.2	3.6
t-Butylcyclohexanone	NR	1.5	NR	NR	NR	NR	NR	NR	NR
Benzaldehyde-(Cl)	NR	NR	NR	0.8	NR	NR	12.7	7.8	5.5
Isophorone-(Cl)	2.5	9.8	1.3	1.3	NR	NR	NR	NR	NR
Benzoic acid (+Benzothiazole)-(Cl)	NR	84.5	53.7	79.0	108.9	63.6	128.2	178	NR
2-(1,1-Dimethylethyl)-cyclohexanol	NR	NR	NR	NR	3.4	3.4	NR	NR	NR
4-(1,1-Dimethylethyl)-cyclohexanol	NR	NR	NR	NR	11.6	11.0	NR	NR	NR
Alkylcyclohexanol ?-(Cl)	NR	NR	NR	NR	NR	NR	NR	NR	6.5
Dimethylethyl cyclohexanol-(Cl)	NR	NR	NR	NR	NR	NR	6.6	6.0	NR
2-Hydroxy-1-phenyl-ethanone	NR	NR	NR	NR	{0.9}	1.5	NR	NR	NR
Dimethyl phthalate ester	0.3	NR	0.2	0.6	0.5	1.5	NR	{0.9}Blk	NR
Dodecyl oxirane? 57,41,56,83-(Cl)	2.0	17.0	2.2	2.2	NR	NR	NR	NR	NR
Diethyl phthalate-(Cl)	NR	NR	NR	NR	{1.6}Blk	1.9	NR	NR	NR
N-butylbenzene sulfonamide	NR	NR	NR	NR	1.2	NR	NR	NR	NR
Dibutyl phthalate isomer-(Cl)	16.0	29.6	16.5	20.4	{18.8}Blk	{22.8}Blk	NR	{17.5}Blk	5.1
Phthalate-(Cl)	1.0	3.0	NR	0.6	7.7	11.1	{10.6}Blk	{3.6}Blk	0.9
Carboxylic acid (Rt 34.35)	NR	NR	NR	NR	1.4	NR	NR	NR	NR
Ethylhexyl(methoxyphenyl)propanoic acid	NR	NR	NR	NR	NR	NR	1.2	NR	NR
4,4'(1-methylethyldiene)bis phenol (Rt 34.36)	NR	NR	NR	NR	NR	1.4	NR	NR	NR
Phthalate-(Cl)	NR	NR	NR	NR	2.8	0.9	NR	NR	NR
Bis(2-ethylhexyl) phthalate-(Cl)	NR	NR	NR	NR	0.2	3.4	NR	{1.4}Blk	NR

NR - not reported

(Cl) - also reported in chlorinated extracts

? - probably the same compound as above

Blk- detected in the extract but judged to be a contaminant

{x.y} - reported as an unknown with a comparable GC retention time

WRc I - 1/2 batches 1 and 2 - within laboratory tests

WRc II - batches 1 and 2 - interlaboratory tests

WQC/CRECEP - 1 or -2 - batches 1 or 2 - interlaboratory tests

KIWA 1 + 2 - results reported as means

Table 4.9 Compounds leached from bitumen - chlorinated leachate

Compound detected	WRc I-1	WRc I-2	WRc II-1	WRc II-2	WQC-1	WQC-2	CRECEP-1	CRECEP-2	KIWA-1+2
Ethyltetrahydrothiophene-(U)	NR	NR	NR	NR	NR	4.8	NR	NR	NR
Hexanal	1.7	0.9	NR	NR	NR	NR	NR	NR	NR
Hexanoic acid-(U)	NR	NR	NR	NR	1.8	NR	NR	NR	NR
Naphthalene	1.0	NR	NR	NR	NR	NR	NR	NR	NR
Octanal	NR	NR	NR	NR	1.8	NR	NR	NR	NR
Heptanoic acid-(U)	NR	NR	NR	NR	1.3	NR	NR	NR	NR
Octanoic acid	NR	NR	NR	NR	1.1	NR	NR	NR	NR
Nonanol	NR	NR	NR	NR	2.0	NR	NR	NR	NR
Nonanoic acid-(U)	NR	NR	NR	NR	5.9	4.3	NR	NR	NR
Alkyl ester carboxylic acid	NR	NR	NR	NR	1.0	2.8	NR	NR	NR
Alkylpropyl ester propanoic acid	NR	NR	NR	NR	NR	1.5	NR	NR	NR
Alkylpentyl ester propionic acid	NR	NR	NR	NR	NR	1.1	NR	NR	NR
Dodecanoic acid	NR	NR	NR	NR	1.1	1.8	NR	NR	4.3
Diethylphthalate-(U)	NR	NR	NR	NR	NR	0.9	NR	NR	NR
Cyclohexadecane-(U)	NR	NR	NR	NR	NR	0.4	NR	NR	NR
Dibutylphthalate isomer	NR	NR	NR	NR	NR	1.8	NR	NR	1.2
Dibutylphthalate isomer	NR	NR	NR	NR	NR	NR	NR	NR	1.8
Hexadecanoic acid-(U)	NR	NR	NR	NR	NR	2.7	NR	NR	NR
1-hexadecane	NR	NR	NR	NR	NR	1.5	NR	NR	NR
Carboxylic acid-(U)	NR	NR	NR	NR	9.0	NR	NR	NR	NR
Carboxylic acid-(U)	NR	NR	NR	NR	2.2	NR	NR	NR	NR
Hydrocarbon	NR	NR	NR	NR	NR	2.1	NR	NR	NR
2-(2-butoxyethoxy) ethyl acetate	NR	NR	NR	NR	NR	NR	NR	NR	1.8
Di-iso-octyl phthalate	NR	NR	NR	NR	NR	1.6	NR	NR	NR

NR- not reported
(U) - also detected in unchlorinated leachates

WRc I-1/2- batches 1 and 2 - within boratory test
WRc II-1/2- batches 1 and 2 - interlaboratory test
WQC/CRECEPI-1 or 2- batches 1 or 2 - interlaboratory test
KIWA 1+2 - results reported as means

Table 4.10 Compounds leached from bitumen - unchlorinated leachate

Compound detected	WRc I-1	WRc I-2	WRc II-1	WRc II-2	WQC-1	WQC-2	CRECEP-1	CRECEP-2	KIWA-1+2
Ethyltetrahydro thiophene-(Cl)	NR	NR	NR	NR	NR	**6.2**	NR	NR	NR
Hexanoic acid-(Cl)	NR	NR	NR	NR	**3.7**	NR	NR	NR	NR
Heptanoic acid-(Cl)	NR	NR	NR	NR	**1.0**	**1.8**	NR	NR	NR
Nonanoic acid-(Cl)	NR	NR	NR	NR	**5.3**	NR	NR	NR	NR
Diethylphthalate-(Cl)	NR	NR	NR	NR	NR	**1.1**	NR	NR	NR
Cyclohexadecane-(Cl)	NR	NR	NR	NR	NR	**1.9**	NR	NR	NR
Hexadecanoic acid-(Cl)	NR	NR	NR	NR	NR	**1.0**	NR	NR	NR
Carboxylic acid-(Cl)	NR	NR	NR	NR	**10.9**	NR	NR	NR	NR
Carboxylic acid-(Cl)	NR	NR	NR	NR	**2.9**	NR	NR	NR	NR
N-stearamide	NR	NR	NR	NR	NR	**1.7**	NR	NR	NR
Cyclohexane isothiocyanate	NR	NR	NR	NR	NR	NR	NR	NR	**1.5**
Urea	NR	NR	NR	NR	NR	NR	NR	NR	**1.3**
Di-iso-octyl phthalate	NR	NR	NR	NR	NR	NR	NR	NR	**7.4**

NR- not reported

(Cl) - also detected in chlorinated extracts

WRc I-1/2 - batches 1 and 2 - within laboratory tests

WRc I I-1/2 - batches 1 and 2 - interlaboratory tests

WQC/CRECEP-1 or -2 - batches 1 or 2 - interlaboratory tests

KIWA 1+2 - results reported as means

identified, or partially identified, by each laboratory in the extracts from the leachates of the three pipes, tested with and without chlorine.

The results show that a reasonable degree of consistency has been achieved particularly in the following respects:

- Most of the compounds detected in the leachates of both the polyethylene (PE) and bitumen coated ductile iron pipes were present at concentrations below 5 $\mu g\ l^{-1}$.

- Most laboratories detected peaks above 10 $\mu g\ l^{-1}$ in the unchlorinated leachates of the glass reinforced polyester (GRP) pipe and generally increased leaching in the chlorinated leachates.

- With some exceptions, the major contaminants were detected and identified by all laboratories, e.g. benzaldehyde, benzoic acid, dibutyl phthalate (GRP), dimethylsuccinate (PE).

Overall, however, the consistency of the results has not been as good as was expected, for the following reasons:

- The total number of compounds considered to originate from the test sample, and reported at concentrations of $\geq 1\ \mu g\ l^{-1}$ in the leachates of the same product varied widely, sometimes within the same laboratory but more often between laboratories (Tables 4.2 to 4.4). The within-laboratory variation may well be due to the fact that, in some instances, different analysts undertook the data interpretation for the two batches of extracts from the leaching tests.

- Compounds with comparable retentions and concentrations were judged by some laboratories to originate from the test sample in certain leachates, but as procedural contaminants in other leachates from the same product (e.g. phthalates from GRP).

- Benzoic acid, detected in 15 extracts from GRP leachates at concentrations ranging from 50 to 570 $\mu g\ l^{-1}$, was not reported in one leachate from one laboratory (although on checking the data it was found to be present) and not at all by another laboratory.

- Toluene, which is volatile and hence elutes soon after the solvent on most GC column (although it elutes later than d_6-benzene, the most volatile internal standard, and should therefore have been detected (if present in the extracts from the leachates) by all laboratories), was detected in all 6 PE leachates by one laboratory but reported by only one other laboratory in one extract.

Some of the differences could well be due to within- and between-laboratory variations in the actual concentrations in the leachates. Additionally, not all details of the details of the GCMS protocol were strictly followed, e.g.

- under the GC conditions used, d_6-benzene, the most volatile internal standard, could not be determined by two laboratories; in one case this was due to inappropriate conditions and in another appears to have been due to its total loss during the concentration stage of the extraction procedure. The GCMS protocol does emphasise the requirement to be able to detect this internal standard. One laboratory, in order to meet the requirement that d_{62}-squalane should elute after 35 minutes, chose to use a temperature programme involving a rapid initial increase in temperature (from 35 °C to 65 °C at 10 °C min^{-1}, immediately following the injection of the extract) followed by a reduction in the programming rate (4 °C min^{-1}).

- results were not adequately reported e.g. in one instance, many detected peaks were reported without any MS information (the protocol specified that the four most intense peaks in the mass spectrum of each detected compound considered to originate from the material under test should be listed) and without making it clear whether the reported peaks originated from the test sample.

- there was inadequate control of the GC column performance over a large batch of GCMS analyses, where a possible increase in column adsorption at the end of the run would not be detected and, consequently, polar compounds such as benzoic acid might not give detectable peaks; the inclusion of a more polar internal standard (e.g. a deuterated carboxylic acid such as d_{31}-hexadecanoic acid) could be used to check for such losses.

In all laboratories it seems to be impossible, under routine conditions, to ensure that the levels of procedural/solvent contaminants is below 1 µg l^{-1}. Therefore the results for compounds known to occur as contaminants, and which are detected at concentrations of up to 5 µg l^{-1} in one batch of leachates, are likely to be unreliable since it is difficult to judge whether the increased concentration of such contaminants in the extracts from the leachates is genuinely due to leaching from the sample or due to a variation in the procedural contamination. Compounds such as aromatic hydrocarbons and chlorinated solvents (often present in laboratory atmosphere) and phthalates are of concern in this respect as they are frequent contaminants in procedural blanks (sometimes detected at concentrations above 10 µg l^{-1}, or even 100 µg l^{-1}) and are known to leach from plastic materials. For example, toluene is known to leach from PE and phthalates from GRP materials. The large concentrations of contaminants found in some instances should, however, be minimised by better quality control.

It is quite likely that a number of the unknowns compounds reported by one laboratory are the same compounds reported as unknowns, or identified compounds by another laboratory. Where, from the tables of results provided, it was highly probable that an unknown is the same as an identified compound, they have been included in Tables 4.4 to 4.9 in brackets. However, between laboratory comparisons were difficult because:

- GC peak retentions were expressed in different ways (absolute and relative retention times, scan numbers, Kovats Indices);

- GC columns of different polarity, run under different operating procedures were used (the GCMS protocol does not specify the type of column to be used or the exact operating conditions, but does provide specifications based on the GC behaviour of the internal standards);

- the four major ions reported for the unknowns varied for the same compound both within and between laboratories, although the latter variation was more marked; while it should have been possible to resolve uncertainties by referring to GC retention times, as different GC columns and temperature programming rates were used by different laboratories, comparisons can sometimes be difficult and may require input from experts;

- detailed comparisons of the mass spectra were very time consuming, and again required input from experts; also not all of the relevant mass spectra were provided (as required by the protocol) by all laboratories.

APPENDIX A
LEACHING TESTS FOR FACTORY MADE OR FACTORY APPLIED ORGANIC PRODUCTS FOR USE IN CONTACT WITH DRINKING WATER

Leaching tests for factory made or factory applied organic products for use in contact with drinking water.

Procedure for the preparation of leachates for the validation of general survey gas chromatography - mass spectrometry (GCMS) method

1 Scope

This protocol describes the procedure for the preparation of leachates from factory-made or factory-applied organic products. The leachates are to be used for the identification, by general survey gas chromatography- mass spectrometry (GCMS) analysis, of unsuspected contaminants leaching from the material into water.

It has been prepared specifically for three products, i.e.

- 2.5-cm internal diameter (i.d.) polyethylene pipe (PE)
- 5 cm i.d. glass reinforced polyester pipe (GRP)
- 10-cm i.d. bitumen-coated ductile iron pipe (Bitumen)

as leachates from these products are to be used for the validation of the general survey GCMS method. However, it may be applied to other pipes (except cementitious pipes) with i.d. up to about 10 cm.

2 Definitions

For the purposes of this procedure, the following definitions apply:

2.1 Test piece (TP). A part of the product to be tested that has been received by the testing laboratory and has been prepared in the same way as the product and has had no additional treatment.

2.2 Test sample (TS). One or more test pieces used in a single leaching test, which provide the required S/V ratio and sufficient volume of the leachate for analysis

2.3 Test water (TW). Specified water for the leaching test

2.4 Chlorinated test water (TW+Cl). Specified water for the leaching test containing free chlorine.

2.5 Leachate . The aqueous solution resulting from keeping the test sample in contact with the test water under the specified test conditions.

2.6 Procedural blank (PB). For laboratory tests, a test water sample, known to contain negligible levels of contamination which would interfere with the analysis. PB is subjected to the same test conditions (test water, test temperature, leaching periods, stoppers, etc.) as the TS, but does not come into contact with the TS.

2.7 General survey GCMS. The acquisition of a series of mass spectra (up to several thousand) during the entire course of a gas chromatographic run, by operating the mass spectrometer in a continuous cyclic scanning mode over a wide mass range. The purpose of operating in this mode is to attempt to detect as wide a range of compounds as possible. Typical operating conditions would include a mass range of 20-700 amu, with total cycle times for this mass range of approximately one second.

2.8 Surface to volume ratio (S/V). A ratio of the surface area of the test sample exposed to the test water to the volume of the test water. The units are expressed as cm^2 /ml. For cylindrical pipes S/V = 4/(internal diameter in cm).

3 Principle

Following a prewashing period the pipe test sample is filled with test water with which it is kept in contact for 72 hours at 25°C.

The resulting leachate, and an appropriate corresponding procedural blank, are then extracted and analysed in accordance with the protocol "GCMS general survey procedure for the analysis of leachates produced from leaching tests conducted on materials for use in contact with drinking water".

For each product leachates with chlorinated and unchlorinated test water are prepared.

4 Reagents

For the purpose of this procedure, the following apply:

NOTE. COSHH assessment (or, outside the UK, alternative risk assessments) should be consulted for all reagents to ensure that they are handled in the recommended manner.

4.1 Tap water. With a free chlorine content less than 0.2 ± 0.05 mg l^{-1}.

4.2 Test water. The requirement for the test water is that it shall be chlorine free,with a total organic carbon (TOC) concentration of less than 0.2 mg l^{-1} and shall not contain organic contaminants which may interfere with the GCMS analysis of the leachates. Any new supply of test water shall be analysed before performing any leaching tests to demonstrate that the level of any impurities present do not interfere with the detection of contaminants leaching from the TS or the internal standards used in the GCMS analysis.

NOTE.Glass distilled or deionised water passed through granular activated carbon should meet these requirements. For a batch of TSs a single batch of test water should be used for the preparation of all leachates and procedural blanks and the volume should also be sufficient to perform the specified prewashing. Thus a single batch of test water should be prepared and stored in a glass storage vessel.

4.3 Chlorinated test water. TW according to 4.2 but having a free chlorine content of 1 ± 0.2 mg l^{-1} , prepared by adding a sufficient quantity of a sodium hypochlorite solution (see 4.4). Determine the free chlorine concentration accurately in accordance with a recognised method (Standing Committee of Analysts "Chemical Disinfecting Agents in Waters and Effluents and Chlorine Demand" (1980) published by HMSO, ISBN 0117514934, ISO 7393-2, or a national equivalent).

4.4 Sodium hypochlorite solution. Prepared from a commercial solution of 1N sodium hypochlorite (NaOCl) and having a known concentration of about 0.1% by mass of free chlorine.

NOTE. The sodium hypochlorite solution is not stable and should be prepared on the day of use.

4.5 Non-ionic detergent solution. Add 600 ml ± 10 ml of non-ionic detergent concentrate to 15 ± 0.1 l of mains tap water and mix well. Replace the solution after two weeks, or when it becomes opaque, whichever is the sooner.

4.6 Hydrochloric acid. Concentrated (30%m/v) analytical reagent grade

4.7 Approx. 10% v/v Hydrochloric acid solution. Slowly add 1.5 ± 0.1 l of concentrated hydrochloric acid to 13.5 ± 0.1 l of mains tap water while constantly stirring, and mix well. Replace the solution on a monthly basis.

5 Apparatus

5.1 General. Contamination may arise from various sources e.g. plastic or rubber materials. Therefore, as far as possible, it is necessary to ensure that the TS, TW, and the leachate only come into contact with inert materials such as cleaned glassware, teflon, and stainless steel.The use of procedural blanks assists in detecting contamination.

Before use, clean all glassware, as well as other materials that will come into contact with the test water, using a non-ionic

detergent (4.5), rinse with hydrochloric acid (4.7) (glassware only), with tap water (4.1) and finally with test water (4.2).

5.2 1 litre glass bottles. Fitted with ground glass stoppers or screw-top caps with PTFE liners.

5.3 Temperature-controlled environment. Suitable apparatus, such as water baths, incubators or temperature-controlled laboratories, to maintain the test temperature at 25 ± 2°C . The apparatus shall be operated in a way that assures that the environment does not contaminate the leachate.

The choice depends on the scale of work to be undertaken.

5.4 Sealing devices. The pipe test pieces, which are to be tested by filling with water, must be sealed with devices that will neither release substances which would be detected when using the GCMS general survey method, nor adsorb such substances which may be released from the test material.

The first choice of seal would be glass stoppers, provided that they are effective . Alternatives are glass stoppers with a PTFE seal or PTFE stoppers. Test pieces of the 10 cm i.d. ductile iron pipes can be sealed using a stainless steel plate at each end of the test piece, held together by four threaded connecting rods and nuts. A small amount of a silicone sealant may be used on the outside, between the TP and the bottom stainless steel plate to stop minor leaks. The top plate has to include two openings for filling and emptying, which are stoppered during the test (see Figure 1).

Note. The silicone sealant should be aquarium grade.

6 Sample preparation

6.1 Sample storage. If the test pieces have to be stored, this should be done in the absence of light, at ambient temperature, and in an environment free of contamination e.g.metal boxes or containers, or wrapped in tissue paper or other material that does not affect the leaching test.

6.2 Preparation of test samples. Seal one end of each pipe test piece with an appropriate stopper (5.4) and check with a small amount of tap water that no leaks occur.

6.3 Sample prewashing. Fill each test piece with unchlorinated TW, seal the top end of the pipe and leave for 24 ± 2 h at 25 ± 2°C.

Empty the test pieces and flush them with tap water (4.1) for 60 ± 10 min at a constant flow of 5 ± 2 m min^{-1} and subsequently rinse with the appropriate TW (4.2 or 4.3) for 2 ± 1 min.

7 Test procedure

7.1 Test samples. The protocol for the GCMS analysis requires that at least 1 litre of the leachate is available. The test samples (2.2) will therefore consist of the following:

Pipe	No of TP	Length (cm)	I.d. (cm)	Volume (l)
PE	2	100	2.5	1.0
GRP	1	60	5.4	1.4
Bitumen	1	25	11.0	2.3

Note. All measurements are approximate

7.2 Procedural blanks. Include with each batch of samples a blank sample (2.5); where only glass stoppers or stainless steel plates are used to seal the tests pieces, a glass container (5.2) is suitable. Where other stoppers or sealants are used (e.g. the silicone sealant), the PB shall also include the sealing device.

7.3 Test design. Each product shall be tested on two separate occasions, with chlorinated and unchlorinated TW(4.2 and 4.3). When the three products specified in

this protocol are tested simultaneously, the test design shall be as follows:

Batch 1	PE - TW(1)
	PE - TW+Cl(1)
	GRP - TW(1)
	GRP - TW+Cl(1)
	Bitumen - TW(1)
	Bitumen - TW+Cl(1)
	PB -TW(1)
	PB -TW+Cl(1)
Batch 2:	PE - TW(2)
	PE - TW+Cl(2)
	GRP - TW(2)
	GRP - TW+Cl(2)
	Bitumen - TW(2)
	Bitumen - TW+Cl(2)
	PB -TW(2)
	PB -TW+Cl(2)

NOTE. When products are tested separately, additional procedural blanks shall be prepared as appropriate.

7.4 Leaching. Immediately after pre-washing, fill the test pieces with the appropriate test water. Insure that the test piece is completely filled, i.e. there is no free head space in the test piece, seal the top end and leave for 72 ± 3 h at 25 ± 2°C.

7.5 Leachate collection. After the leaching period, determine the residual free chlorine in the leachate, and collect a sample for analysis as described in the "GCMS general survey procedure for the analysis of leachates produced from leaching tests conducted on materials for use in contacte with drinking water". Where the test sample consist of more than one test piece, combine the leachates in one sampling bottle.

8 Test report

The test report shall include the following information:

(a) a reference to this document;

(b) the name and address of the laboratory undertaking the testing; the date of the report and a unique laboratory number;

(c) the date of reception of the test pieces;

(d) the identity of each test sample;

(e) details of storage conditions;

(f) the date of the start of each test;

(g) details of the test water used;

(h) description of the method used to seal the test pieces ends;

(i) volume of the test water and S/V during the test ;

(j) free chlorine concentrations at the beginning and the end of the 72-hours leaching period for tests with chlorinated TW; and

(k) any deviation from the test procedure specified in this document.

NOTE. An example Test Report is provided in the Appendix.

APPENDIX

Example test report for leaching test

TEST LABORATORY: WRc Medmenham

Form No: 1.

Product type: Bitumen lined ductile iron pipe

Laboratory's Product Ref No: 8782/Bitumen

LEACHING/MIGRATION TESTS - PRODUCT TEST PIECES RECORD

Test pieces received from: WRc Medmenham

Date received in the Laboratory: ?

No of test pieces received: 2.

Dimensions:
Length: 25.0cm
OD: 11.72cm
ID: 10.74cm

Appearance of test pieces on receipt:
Uncovered.
Dusty appearance.
Undamaged.

Storage conditions of test pieces before tests:
At room temperature in original packaging.

Test procedure document:
Procedure for the Preparation of Leachates according to protocol supplied.

Tests to be carried out:
Leaching study (72 hour duration) in non-chlorinated and chlorinated test water

Determinands to be measured:
Organic leachates by General Survey GC-MS according to protocol supplied.

TEST LABORATORY: WRc Medmenham
Form No: 2a.

Product type: Bitumen lined ductile iron pipe

Laboratory's Product Ref No: 8782/Bitumen/Batch 1

LEACHING/MIGRATION TESTS - TEST SAMPLE RECORD

Sample identification Number/code: Bitumen-TW(1)

Sample preparation

No of test pieces for test: one

Surface area for test: ~843.5cm^2

Method(s) of sealing pipe ends (bottom/top):
Stainless steel plates positioned top and bottom of pipe section and held in position with lengths of studding and nuts. Between the bottom plate and pipe was a PTFE plate.

Prewashing procedure:
Tap water (free chlorine <0.2mg/l
Flow: Actual: 22l/min Theorectical: 27l/min for 0.5cm/s

Test Conditions

Test Water: Deionised and carbon filtered tap water

- **Origin:** Medmenham raw water passed through a mixed-bed ion-exchange resin

- **Conductivity:** <12Ω/cm

- **Method for determination of chlorine:** Chemical Disinfecting Agents in Water and Effluents, and Chlorine Demand 1980, HMSO. Section C, Titrametric DPD Method.

- **Chlorination;** Not applicable

- **Volume of test water used:** 2250cm^3

- **Surface to volume ratio:** 0.37cm^{2}cm^{-3}

- **Temperature °C:** 25

Deviations from test procedure

TEST LABORATORY: -

Form No: 2b.

Product type: Bitumen lined ductile iron pipe

Laboratory's Product Ref No: 8782/Bitumen/Batch 1

LEACHING/MIGRATION TESTS - TEST SAMPLE RECORD

Sample identification Number/code: Bitumen-TW+Cl(1)

Sample preparation

No of test pieces for test: one

Surface area for test: ~843.5cm^2

Method(s) of sealing pipe ends (bottom/top):
Stainless steel plates positioned top and bottom of pipe section and held in position wit
lengths of studding and nuts. Between the bottom plate and pipe was a PTFE plate.

Prewashing procedure:
Tap water (free chlorine <0.2mg/l
Flow: Actual: 22l/min Theorectical: 27l/min for 0.5cm/s

Test Conditions

Test Water: Deionised and carbon filtered tap water

- **Origin:** Medmenham raw water passed through a mixed-bed ion-exchange resin

- **Conductivity:** <12Ω/cm

- **Method for determination of chlorine:** Chemical Disinfecting Agents in Water and Effluents, and Chlorine Demand 1980, HMSO. Section C, Titrametric DPD Method.

- **Chlorination;**	Free chlorine (mg/l)	Initial value	Final value
		1.2	0

- **Volume of test water used:** 2250cm^3

- **Surface to volume ratio:** 0.37cm^{2}cm^{-3}

- **Temperature °C:** 25

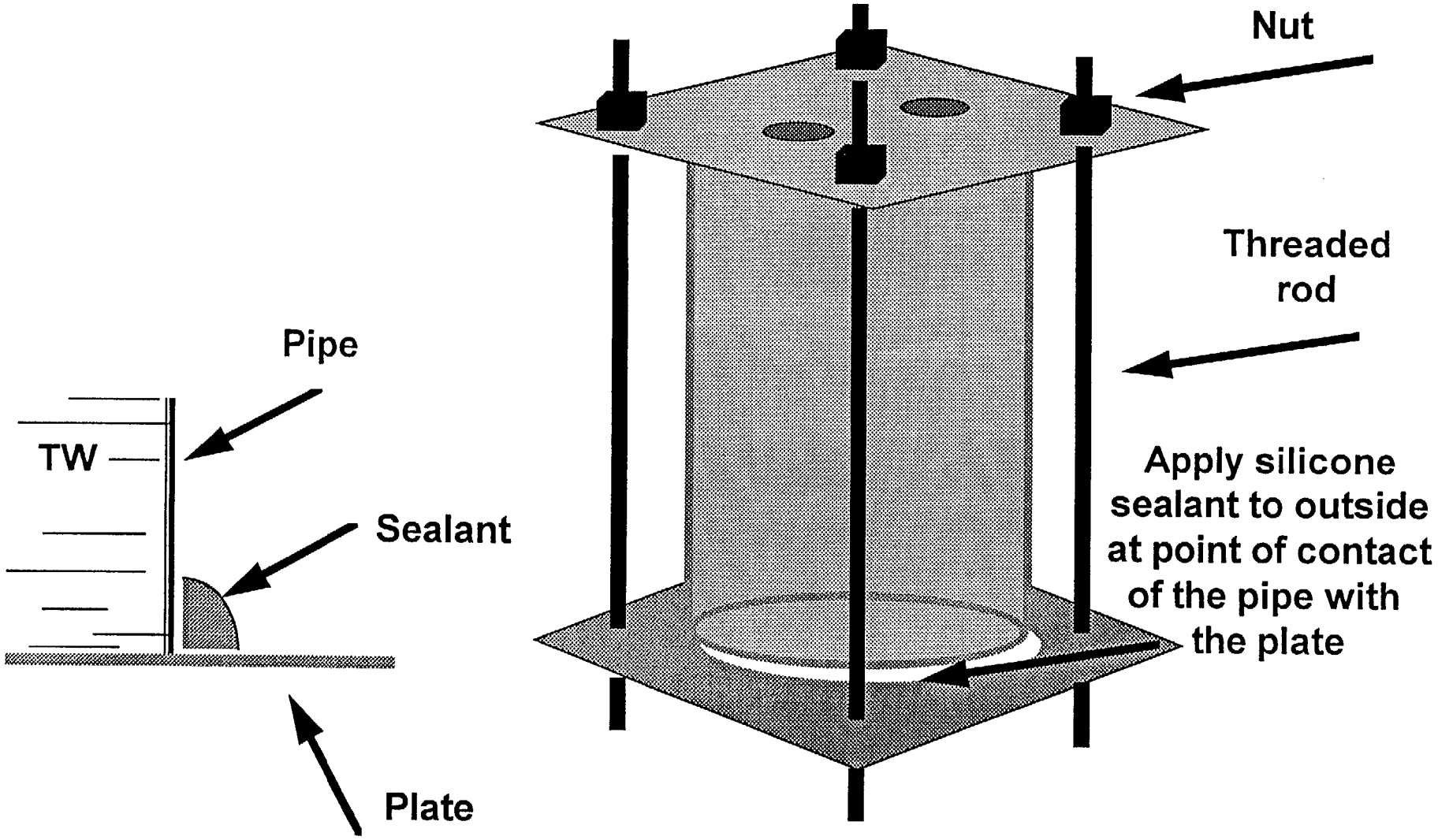
Deviations from test procedure

TEST LABORATORY WRc MEDMENHAM.
BATCH No 1.
SAMPLES PE, GRP, Bitumen

LEACHING/MIGRATION TESTS - TEST EVENTS RECORD

Table of Test Events for a batch of test samples including prewashing, leaching of samples, preparation of procedural blanks, and collection of analytical samples

EVENT	START		FINISH		Elapsed time (hrs)
	Date	Time	Date	Time	
Sample prewashing	16th Jan 95	10.55	17th Jan'95	9-00	22.1
Flush	17th Jan'95	9-15	17th Jan'95	10-20	1.1
Rinse	17th Jan 95	10-25	17th Jan 95	10-45	0.3
Leaching	17th Jan 95	11-00	20th Jan'95	11-35	72.6
Sample collection	20th Jan 95	11-35	20th Jan 95	11-55	0.3


Nut
Threaded rod
Pipe
TW
Sealant
Apply silicone sealant to outside at point of contact of the pipe with the plate
Plate

APPENDIX B

GCMS GENERAL SURVEY PROCEDURE FOR THE ANALYSIS OF LEACHATES PRODUCED FROM LEACHING TESTS CONDUCTED ON MATERIALS FOR USE IN CONTACT WITH DRINKING WATER

GCMS general survey procedure for the analysis of leachates produced from leaching tests conducted on materials for use in contact with drinking water.

1 Scope

This protocol describes the procedure for the determination of organic compounds in leachates produced during field and laboratory leaching tests. It involves extraction of the leachates to produce solvent extracts, and analysis using general survey gas chromatography-mass spectrometry (GCMS) of these solvent extracts.

Prior to analysis, the substances to be determined are unknown. The method is therefore primarily qualitative, but estimates of the levels of the compounds detected can be obtained by relating their response to the response of isotopically labelled internal standards which are added to the sample. However in arriving at these quantitative estimates, various assumptions have to be made, and generally they are of uncertain accuracy. If accurate quantitative information is required, the compounds to be determined must be specified prior to analysis and the GC-MS analysis performed using a different protocol.

This procedure can only be applied to those organic compounds which are amenable to the solvent extraction procedure, elute from the gas chromatographic column used after the extraction solvent and within the temperature range employed, and which produce ions within the mass range 20-700 amu when electron impact ionisation is utilised.

2 Definitions

For the purposes of this procedure, the following definitions apply:

2.1 Leachate. The aqueous solution resulting from leaving water in contact with the test sample under the specified test conditions.

2.2 Solvent extract. A solution containing compounds partitioned from the sample into the extracting solvent (in this case dichloromethane).

2.3 Procedural blank. For laboratory tests, a test water sample, known to contain negligible levels of contamination, which is treated in the same way as the leachate, but which does not come into contact with the material tested, and which is then analysed in the same way as the leachate. For field tests, a sample of the water to be used for the leaching test, sometimes referred to as the inlet water, may be used as a procedural blank. Procedural blanks are used to check for potential contamination of either leachates or solvent extracts during the whole of the procedure, from sampling (which may be undertaken outside the laboratory, and involve sample transport) through to analysis.

2.4 Laboratory blank. A water sample, known to contain negligible levels of contamination, to which internal standards have been added, and which is then analysed in the same way as the leachate. Laboratory blanks are used to check for potential contamination of either leachates or solvent extracts which has occurred within the laboratory undertaking the analysis. The maximum allowable concentration of an individual contaminant in blanks is 2 $\mu g\ l^{-1}$; if a laboratory cannot meet this requirement, sufficient replicate blanks must be analysed to establish a realistic limit of detection for leachates, for compounds (e.g. phthalates) commonly present in blanks.

2.5 Internal standard. A compound added to a sample to be analysed as soon as the sample is taken. Ideally an internal standard should be present in the sample only as a result of deliberate addition.

2.6 General survey GCMS. The acquisition of a series of mass spectra (up to several thousand) during the entire course of a gas chromatographic run, by operating the mass spectrometer in a continuous cyclic scanning mode over a wide mass range. The purpose of operating in this mode is to attempt to detect as wide a range of compounds as possible. Typical operating conditions would include operation in electron impact mode, a mass range of 20-700 amu, with total cycle times for this mass range of approximately one second.

2.7 Total ion current (TIC). The sum of all the separate ion currents carried by the individual ions contributing to a mass spectrum.

2.8 TIC chromatogram. A graphical representation of the TIC versus time. The trace is similar to a GC chromatogram.

2.9 Electron impact ionisation. Ionisation by a beam of electrons.

2.10 Mass spectrometric resolution. A measure of the capability of the mass spectrometer to correctly detect two mass spectral peaks, having similar m/z values, as separate peaks. Generally denoted by M/dM, where M is the higher m/z value and dM is the difference between the two masses e.g. a mass spectrometer set up so that the resolution is 700 will satisfactorily resolve and assign the correct masses to mass spectral peaks at m/z 699 and m/z 700.

2.11 m/z. The mass-to-charge ratio of an ion. As most ions produced by electron impact ionisation are singly charged, this ratio usually corresponds to the mass of an ion; however, exceptionally, ions may possess multiple charges.

2.12 Asymmetry factor (A_S). A measure of the adsorption of a compound during gas chromatographic analysis; it may be derived from the equation

$$As = (a + b)/2b$$

where a is the distance from the leading edge of the peak to the point on the baseline at which a perpendicular dropped from the peak maximum crosses it, and b is corresponding distance from the trailing edge of the peak.

3 Principle

Leachate samples and procedural blanks obtained using chlorinated test waters should be dechlorinated immediately following the completion of the leaching test, to prevent any change in the concentration of compounds leached, due to the continued presence of residual free chlorine.

The leachate sample(s) and appropriate procedural blanks are spiked with the mixture of isotopically labelled internal standard compounds, and solvent extracted with dichloromethane.

The extract is concentrated, and analysed by GCMS. The mass spectrometer is used in a repetitive full scan mode (mass range 20-700 amu) and the mass spectra produced recorded by, and stored on, the GCMS data system.

A range of internal standards is added to the samples at known levels in order to (a) allow compounds detected to be quantified, and (b) to provide quality control. This latter aspect is particularly important in this type of analysis, where the compounds to be analysed are not known until the analysis has been undertaken.

The internal standards are chosen to represent various types of compounds which may typically be present in water samples, and isotopically labelled compounds are used to ensure that the internal standards are only present in the samples due to deliberate addition. Provided that the standards are carefully selected they may be distinguished, mass spectrometrically, from any naturally occurring analogues which may be present.

Whenever possible each compound detected is identified. The methods used to identify organic compounds from their mass spectra

do not form part of this protocol, but further information on this subject is provided in the Appendix.

Each compound detected may be quantified by reference to the isotopically labelled internal standards.

4 Safety

All reagents, solvents and standards used are classified as hazardous and COSHH assessments should be consulted for all materials used, to ensure that they are handled in the recommended manner. Caution must be exercised when preparing stock and working standard solutions; skin contact, ingestion and inhalation must be avoided.

GCMS systems typically operate from a nominal mains voltage (e.g. 220-240V; exceptionally, some MS systems require a "3-phase" supply at 415V) However, certain parts or components of the mass spectrometer may be at a very high electrical potential (up to 8 kV), relative to earth. Due care must be exercised during the operation of GCMS systems.

5 Reagents

5.1 General. All chemicals and reagents must be of sufficient purity so that they do not give rise to significant interferences during the GCMS analysis. Contamination may arise from various sources e.g. plastic or rubber materials. The use of procedural blanks and laboratory blanks assists in detecting and identifying the source of any contamination.

5.1.1 *Blank Water.* A supply of water which contains negligible levels of organic compounds detectable by GCMS. Distilled water may be suitable for this purpose, but should be checked prior to use.

5.1.2 *Dichloromethane.* Glass distilled grade is recommended. Other grades may be suitable, but it is necessary to demonstrate that the level of any impurities present does not interfere with the detection of determinands of interest or the internal standards, or introduce unacceptable contamination.

5.1.3 *Sulphuric acid solution (0.5M).* Slowly add 14.0 ml +/- 0.5 ml of concentrated sulphuric acid (specific gravity 1.84) to 300 ml +/- 1 ml of double distilled water, and make up to 500 ml +/- 1 ml.

5.1.4 *Sodium Hydroxide (0.5M).* Dissolve 2.00 g +/- 0.01 g sodium hydroxide pellets in double distilled water and make up to 500 ml +/- 1 ml.

5.1.5 *Acetone.* Glass distilled grade.

5.1.6 *Non-ionic detergent.* Add 600 ml +/- 10 ml of non-ionic detergent concentrate to 15 l +/- 0.1 l of mains tap water and mix well. The solution should be replaced after two weeks, or when it becomes opaque, whichever is the sooner.

5.1.7 *Approx. 10% v/v Hydrochloric Acid solution.* Slowly add 1.5 +/- 0.1 l of concentrated hydrochloric acid to 13.5 +/- 0.1 l of mains tap water while constantly stirring, and mix well. The solution should be replaced on a monthly basis.

5.1.8 *Ascorbic acid solution.* Dissolve 4g ascorbic acid in 1.0 l blank water (5.1.1). This solution should be freshly prepared for each batch of samples, and prior to use should be extracted with dichloromethane (2 x 200 ml) to remove any potential interferences.

5.2 Standards.

5.2.1 *Isotopically labelled internal standards.*

The following compounds are used as internal standards:

d_6-benzene
d_{20}-2,6-di-t-butyl-4-methylphenol
d_5-chlorobenzene
d_{34}-hexadecane
d_8-naphthalene
d_{31}-palmitic acid *
d_{10}-phenanthrene
d_5-phenol
d_{62}-squalane
d_{10}-p-xylene

* - d_{31}-palmitic acid was not used as an internal standard during the performance testing of this protocol; it was added following the performance testing because it was considered necessary to ensure that laboratories using this protocol should be able to detect acidic compounds present in leachates.

5.2.2 *Internal standards, stock solutions.* Make up the following individual stock solutions in acetone:

d_6-benzene+	2 mg/ml
d_{20}-BHT*	8 mg/ml
d_5-chlorobenzene	2 mg/ml
d_{34}-hexadecane	0.5 mg/ml
d_8-naphthalene	0.5 mg/ml
d_{31}-palmitic acid	8 mg/ml
d_{10}-phenanthrene	2 mg/ml
d_5-phenol	8 mg/ml
d_{10}-p-xylene	0.5 mg/ml

+ due to its volatility, it is difficult to make standard solutions of d_6-benzene by weighing; it is recommended that suitable volumes of d_6-benzene, based on its density (1.4986 @ 20°C), are used.
* BHT = 2,6,-di-t-butyl-4-methylphenol

Make up the following stock solution in dichloromethane:

d_{62}-squalane	8 mg/ml

5.2.3 *Internal standards, intermediate solution.* Add 2.5 ml +/- 0.025 ml of the d_{62}-squalane stock solution to a 25 ml volumetric flask. Remove as much as possible of the dichloromethane using nitrogen blow down. Then put 2.5 ml +/- 0.025 ml of each of the remaining individual internal standard stock solutions (5.2.2) into the volumetric flask and make up to 25 ml +/- 0.025 ml with acetone.

5.2.4 *Internal standards, GC column test solution.* Add 200 µl +/- 2 ul of the internal standards intermediate solution (5.2.3) to a 10 ml volumetric flask and make up to 10 ml +/- 0.01 ml with dichloromethane.

5.2.5 *Internal standard, spiking mixture.* Add 1 ml +/- 0.01 ml of the internal standards intermediate solution (5.2.3) to a 10 ml volumetric flask and make up to 10 ml +/- 0.1 ml with acetone.

Standard solutions 5.2.2 and 5.2.3 are stable for at least six months, provided they are stored in the dark at -18° C, or below. Standard solutions 5.2.4 and 5.2.5 should be renewed every three months or sooner if, during their use, an indication is obtained that the concentrations of any of the internal standards have changed.

6 Apparatus

6.1 Glassware. All glassware should be cleaned using an aqueous solution of a proprietary detergent specially designed for the removal of organic materials (5.1.6).

6.1.1 *Sample bottles.* 1 litre glass bottles with ground glass stoppers or screw tops fitted with PTFE liners.

6.1.2 *Separating funnels.* 2 L capacity.

6.1.3 *Measuring cylinders.* 1L and 100 ml.

6.1.4 *Volumetric Flasks.* Various e.g. 10 ml, 25 ml , and 50 ml .

6.1.5 *Pipettes and syringes.* Various e.g. 1 ml, 2.5 ml pipettes, 100 µl and 500 µl syringes.

6.1.6 *Analytical balance,* capable of weighing 0.1000 g +/- 0.0001 g, for

preparation of internal standard stock solutions.

6.1.7 *Analytical balance,* capable of weighing 2.00 g +/- 0.01 g, for preparation of reagents.

6.1.8 *Flasks,* various.

6.1.9 *Concentration apparatus,* required to reduce the volume of the solvent extract from 200 ml to 500 µl. During this operation, which may proceed in several steps, losses of volatile compounds must be minimised. The response obtained for the most volatile internal standard (d_6-benzene) should be checked to ensure that losses of this compound in the concentration step do not exceed 50%. One method of checking for losses is given in the Appendix.

6.2 Instrumental

6.2.1 *Capillary gas chromatograph* with temperature gradient facility, equipped with an on-column injector (optional), with autosampler (optional), and interfaced to a mass spectrometer (6.2.7) via a GCMS interface(6.2.6).

6.2.2 *GC capillary column,* **length at least 50m,** coated with a bonded phase. May optionally be used in conjunction with 6.2.3.

6.2.3 *Deactivated silica pre-column,* optional (requires 6.2.4 if used); length 1 m, appropriate internal diameter.

6.2.4 *Press-fit capillary connector,* optional, to connect *6.2.3,* when used, to *6.2.2.*

6.2.5 *Carrier gas,* (for the GCMS system) helium (99.999% purity) connected to the gas chromatograph via in-line traps for the removal of hydrocarbons, moisture and oxygen. Some GCMS operators prefer to use hydrogen as carrier gas; this is acceptable provided that the performance achieved is satisfactory. **(N.B. Additional safety precautions may be required when hydrogen is used as carrier gas.)**

6.2.6 *GCMS interface* The GCMS interface should be operated so that no degradation of the GC resolution is observed, and the least volatile internal standard (d_{62}-squalane) is efficiently transferred to the mass spectrometer.

6.2.7 *Mass spectrometer.* capable of operating as follows:

(a) over the required mass range (20-700 amu);

(b) at a resolution >700;

(c) with a scan cycle time for the required mass range of 1 second or less;

(d) in the electron impact ionisation mode.

6.2.8 *Mass spectrometry data system.* capable of operating as follows:

(a) acquiring data from the mass spectrometer under the conditions noted in 6.2.7;

(b) producing a TIC chromatogram;

(c) measuring peak areas on the TIC chromatogram;

(d) producing background-subtracted averaged mass spectra;

(e) producing hard-copy outputs of TIC chromatograms and mass spectra.

6.2.10 *Mass spectral library,* installed on the mass spectrometry data system, or available as a hard copy document, e.g. Environmental Protection Agency - National Institute of Health Mass Spectral Data Base, or The Eight Peak Index.

7 Sample collection

7.1 Bottles. Leachate samples should be collected in glass bottles (6.1.1). Before use, the bottles should be washed using a non-ionic detergent (5.1.7), rinsed with hydrochloric acid solution (5.1.8) and finally with blank water (5.1.1).

7.2 Sample collection. When taking a leachate sample, the bottle should be rinsed with the sample prior to taking the sample for analysis. For the leachate samples produced using chlorinated test water, ascorbic acid solution (2 ml; 5.1.8) should be added to the sampling bottle before collecting the sample to be analysed. For leachate samples produced using test water (with no chlorine), there is no requirement to add ascorbic acid. Sample bottles should be completely filled with the sample so that there is no head-space above the sample. This is to prevent the loss of volatile components from the sample.

7.3 Sample storage. The analysis of samples should commence as soon as possible following sample collection. However, if there is an unavoidable delay between sampling and analysis, samples are preserved by storage (in the dark) at 4°C +\- 2°C in a refrigerator. Samples should not be stored for longer than 48 hours prior to extraction.

8 Analytical procedure

The procedure is divided into five stages as follows:

(a) Extraction
(b) GCMS analysis
(c) Identification of individual compounds.
(d) Quantification of each compound.
(e) Quality control.

8.1 Extraction procedure

8.1.1 Transfer 1 L +/- 10 ml of the sample to a 2 l separating funnel.

8.1.2 Add 100 µl of the internal standard spiking mixture (5.2.5), into the sample, using a syringe (250 ul capacity), ensuring that the tip of the syringe needle is below the surface of the sample. The levels of the internal standards in the sample will then be as follows:

d_6-benzene	2.0 µg/l
d_{20}-BHT*	8.0 µg/l
d_5-chlorobenzene	2.0 µg/l
d_{34}-hexadecane	0.5 µg/l
d_8-naphthalene	0.5 µg/l
d_{31}-palmitic acid	8.0 µg/l
d_{10}-phenanthrene	2.0 µg/l
d_5-phenol	8.0 µg/l
d_{62}-squalane	8.0 µg/l
d_{10}-p-xylene	0.5 µg/l

* BHT = 2,6-di-t-butyl-4-methylphenol

8.1.3 Check the pH of the sample and adjust to pH 2.0 +/- 0.2, if necessary, by dropwise addition of either sulphuric acid solution (5.1.3) or sodium hydroxide solution (5.1.4) as appropriate.

8.1.4 Add dichloromethane (100 ml +\-5 ml) to the spiked, pH adjusted sample in the separating funnel and extract by shaking for 3 minutes (+\- 10 seconds). Remove the dichloromethane (lower layer) into a 250 ml round-bottomed flask (6.1.7). Repeat the extraction with a further aliquot (100 +\- 5 ml) of dichloromethane, and add the dichloromethane to the flask in which the initial extract is stored, so that the two extracts are combined.

8.1.5 Dry the solvent extract by freezing out any water present by storing in a freezer (-18°C) overnight.

8.1.6 Carefully decant the solvent extract from the flask into the apparatus to be used for concentrating the extract (6.1.8). Any ice particles present may be removed by the extract through a quartz-wool plug.

8.1.7 The solvent extract should then be reduced in volume to 500 +/- 50 µl. As noted earlier (6.1.9), it is essential to ensure

that losses of the most volatile standard (d6-benzene) do not exceed 50% during this concentration step.

8.1.8 Store the concentrated extract in a freezer at -18°C or below, until required for GCMS analysis.

8.2 GCMS analysis

8.2.1 *Mass spectrometer operating parameters.* The mass spectrometric operating conditions should be set, following the manufacturers instructions, as follows:

Ionisation Technique:	Electron Impact(EI)
Electron Energy	70 eV
Mass Range	20 - 700u
Scan speed	>1 scan per second
Scan mode	repetitive

8.2.2 *Setting up the mass spectrometry data system.* The manufacturers instructions relating to tuning, calibration, data acquisition and processing should be followed.

8.2.3 *Initial tuning and calibration of the mass spectrometer.* Tune the mass spectrometer to ensure satisfactory sensitivity and mass spectrometric resolution using appropriate peaks produced by a calibrant such as perfluorokerosene (PFK) or heptacosafluorotributylamine (Heptacosa) introduced from a heated septum inlet, at the beginning of each day. As the means of carrying this out is dependent on the type and model of mass spectrometer used, the manufacturer's instructions should be followed.

All the major (>5% of the intensity of the base peak of the calibrant used) reference peaks in the mass range 20-700 amu in the calibration table held on the MS data system should be found in the scan used for calibration purposes.

8.2.4 *Setting up the GCMS system.* The GC column should be installed according to the manufacturers instructions, and its performance (e.g. in terms of separation number and adsorption) checked against the column performance data supplied by the manufacturer. Proprietary standard solutions are available for this purpose (see Appendix). Provided this measure of the performance of the column is satisfactory, the GC column test standard solution (5.2.4) should be used to establish the initial performance of the column for this particular mixture. The temperature programme used for this purpose should be the same as that used for the GCMS analysis of the concentrated solvent extracts, and **the programming rate must not, at any stage of the GCMS run, exceed 8°C per minute.** d_6-Benzene **must be** detected and separated from the solvent peak. The recommended initial temperature of the GC column is 30°C. The retention time of d_{62}-squalane **must be** between 35 and 45 minutes. All of the internal standards **must be** detected. Particular attention should be paid to the asymmetry of the peaks for d_8-naphthalene and d_5-phenol. The individual asymmetry factors (A_S) for these two peaks should be measured, and provided $0.67 < A_S < 2.0$, the GC column is satisfactory.

If the quality of the chromatography does not meet these requirements, the cause should be investigated and corrected before continuing with the analysis. If necessary, a new GC column should be installed.

The sensitivity of the mass spectrometer should be adjusted so that the mass spectra obtained from the internal standards present at the highest level (d_5-phenol, d_{20}-BHT and d_{62}-squalane, each present at 16 ng/ul in the GC column test standard solution (5.2.4)) are not saturated.

The mass spectra obtained from the system performance test should be inspected by the analyst to ensure that they match mass spectra previously acquired on the same GCMS system under identical operating conditions. The m/z value of base peak should be consistent, and the intensities of all peaks having an intensity >10% of the base peak should not vary by more than 30% of their intensity, compared to previously

acquired spectra. If the GC column test standard solution (5.2.4) has not previously been analysed, it should be run once a day on the GCMS system on five separate days to obtain typical spectra of all of the internal standards.

Check that the mass spectrometer has stayed within calibration by checking that the high mass ions (> m/z 300) in the mass spectra acquired for d_{62}-squalane are correctly mass measured. If this is not the case, the mass spectrometer must be recalibrated, and this check repeated.

8.2.5 *GCMS operating conditions for analysis of solvent extracts.* All solvent extracts are analysed using identical GCMS operating conditions to those used for the analysis of the system performance test (8.2.1). The performance of the system is checked at the end of every batch of solvent extracts run, or after every sixth solvent extract if batch sizes are greater than six, and the criteria noted above (8.2.4) are checked to ensure that the performance of the system has not deteriorated. If any if the criteria are not met, the analysis must be stopped and the causes must be investigated and corrected before continuing with the analysis.

8.2.6 *Production of required outputs from the mass spectrometry data system.* The following outputs are required for each of the GCMS run carried out on a solvent extract:

(a) a hard copy of the TIC trace, covering the mass range 20-700 amu, for the GCMS run (if a "solvent delay" is included as part of the data acquisition, the TIC trace will not include a peak for the solvent; this is acceptable);

(b) a listing of the retention times (correct to +/- 1 second) of the peak maximum of all of the peaks detected; this should include the retention times of the internal standards;

(c) the areas of all of the detected peaks , including the internal standards;

(d) hard copies of a mass spectrum obtained for each of the peaks detected considered to originate from the test sample; this should be the best spectrum obtainable following manipulation of the data by the data system; normally this would include background subtraction and averaging of several mass spectra obtained for each compound.

Compounds detected which are not considered to arise from the material being tested, or which are not internal standards, are included in the above requirements, except where stated otherwise. However, an indication should be given of which of the detected compounds fall into this category, and their probable origin e.g. contaminants in the solvent used for the solvent extraction.

8.3 Identification of compounds detected

It is beyond the scope of this procedure to give detailed instructions regarding the identification of unknowns from their electron impact mass spectra and the analyst should refer to standard texts on the subject. However, the general approach normally used is outlined in the Appendix.

8.3.1 *Certainty of detection.* Three categories are used to define the confidence level associated with compound identification, as follows:

(a) positive identification - the mass spectrum and GC retention time of the detected compound are the same as those obtained from a pure standard of the compound run under identical GCMS conditions on the GCMS system used to analyse the solvent extract;

(b) tentative identification - a possible identification has been obtained either from computerised

library searching of a mass spectral data base, or manual searching of a printed mass spectral data base, or by interpretation from first principles by the mass spectroscopist, but a pure standard has not been run under identical GCMS conditions on the GCMS system used to analyse the solvent extract, or is not available;

(c) unknown - this covers all compounds detected which do not fall into the above categories; the four most intense peaks in the mass spectrum should be noted, in descending order of intensity, with the base (100%) peak being emphasised by underlining (e.g. 147, 43, 71, 91), together with the retention time or scan number, for each detected peak in this category.

8.4 Quantification of compounds

The concentration of a determinand D in a sample extract ([D]), is calculated from the equation:

$$[D] = \frac{\text{Pk Area(D) x I.S. Conc'n}}{\text{Pk Area(I.S.)}}$$

where

Pk Area(D) is the peak area for determinand D in the solvent extract TIC chromatogram;

Pk Area(I.S.) is the peak area of the chosen internal standard in the solvent extract TIC chromatogram;

I.S. Conc'n is the concentration of the internal standard; if this is expressed as ug/l, [D] will also be ug/l.

Each detected compound is quantified by comparing its response to the nearest (in terms of GC retention time) of the internal standard present at 2 or 8 ug/l, with the exception of d_5-phenol which is not used for quantification. The internal standards present at 0.5 ug/l are not used for quantification.

No attempt should be made to adjust [D] for extraction efficiency.

8.5 Quality control

It is difficult to apply conventionally accepted analytical quality control criteria to this type of analysis, as the determinands are unknown prior to the analysis. The use of isotopically labelled internal standards provides a degree of quality control, in that their detection and the response obtained provide information on the success of the sample preparation and GCMS procedures.

As a minimum, the following QA/QC procedures must be followed:

8.5.1 *Mass calibration of the mass spectrometer.* This should be checked on each day that analysis of solvent extracts is carried out. The calibrant normally used for mass calibration may be used for this purpose; if any of the calibrant masses are incorrectly assigned, the mass spectrometer should be re-calibrated.

8.5.2 *System performance check.* The performance of the GCMS system should be checked on each occasion a batch of solvent extracts is to be run, by analysing the GC column test standard (5.2.4). The response obtained for each of the internal standards should be compared to that obtained when setting up the GCMS system (8.2.1), and provided that the peak areas are within 30% of these, and that the asymmetry factors (A_S) for d_8-naphthalene and d_5-phenol are acceptable ($0.67 < A_S < 2.0$), the performance is satisfactory. The retention times of each of the internal standards should be checked, and should be consistent (rsd $< 2\%$ on an absolute basis).

System stability checks are carried out by analysing the GC column test standard after

every six solvent extracts. The criteria for satisfactory performance are as given above.

8.5.3 *Establishing acceptable performance for the method.* An acceptable GCMS response for the internal standards detected in the solvent extracts from leachates, is based upon the response obtained from solvent extracts from blank water samples which have been spiked with the internal standards at the same levels as used for leachate samples. The analytical procedure (8.1 through 8.2.7) should be followed, and the recoveries of d_8-naphthalene, d_{10}-phenanthrene and d_{62}-squalane calculated. The recoveries for each of these compounds should be > 50%. A procedure for performing this calculation is given in the Appendix.

8.5.4 *Monitoring internal standards detected in solvent extracts.* All of the isotopically labelled standards listed in section 5.2.1, added to each leachate sample to be analysed, must be detected in the GCMS run of the solvent extract. The retention times of the internal standards should be consistent (rsd <2%) in all of the solvent extracts analysed. The absence of any of the internal standards, or a poorer than expected response for any of the standards, indicates that either

(a) the extraction step has not been carried out correctly; or

(b) the concentration of the solvent extract has not been undertaken correctly;

(c) the GCMS system is not functioning correctly.

The cause of the problem should be investigated and addressed.

9 Test report

The test report shall include the following parameters:

(a) a reference to this standard document.

(b) the name and address of the laboratory undertaking the testing; the date of the report and a unique laboratory number;

(c) results from the GCMS examination of each solvent extract reported in a tabular format, together with the TIC chromatogram for each solvent extract and a TIC chromatogram for the internal standards GC column test solution (5.2.4). Data tables should list all of the peaks detected, but those peaks considered not to originate from the material being tested should be indicated and suggestions as to their origin provided. The use of procedural blanks and laboratory blanks are useful in this respect.

The retention time of each compound listed should be stated and the identity of the compound given (see 8.3.1). The estimated concentration of each compound considered to originate from the material under test should be given in ug/l (8.4), and the internal standard used to derive this estimate should be noted.

In cases where compounds detected in procedural blanks or laboratory blanks are also detected in solvent extracts from leachates, if the apparent levels in the procedural or laboratory blanks and extracts are low (< 2 ug/l) and do not differ by more than 25% (of the highest level), the levels and differences are not considered significant, and no level should be indicated in the table of results. In cases where the apparent levels of such compounds are lower in a solvent extract than in the procedural or laboratory blank, no level should be indicated in the table of results. However, when the apparent levels of such compounds are higher in the solvent extracts than in the procedural or laboratory blanks, and >2 ug/l, a "blank subtracted" level should be reported i.e. the apparent level in the procedural or laboratory blank is subtracted from the apparent level in the solvent extract.

(d) print-out (or copy) of a mass spectrum for each of the compounds detected which are considered to originate from the material being tested. This should be the best

spectrum obtainable, and may be background subtracted and produced by averaging several spectra.

(d) any deviation from the test procedure specified in this document should be noted in the Test Report.

(e) an example Test Report is provided in the Appendix.

APPENDIX

1. Outline of general approach for identification of compounds detected.

The data acquired during the GCMS run for each solvent extract is normally stored on the mass spectrometry data system as a discrete data file which may be inspected either while the run is proceeding, or after the run has been completed.

The data is usually initially displayed on a data system visual display unit (VDU) as a total ion current (TIC) chromatogram. Each compound detected should appear as a peak on the TIC trace, and the mass spectra produced by each compound can be displayed on the VDU using the appropriate commands.

Normally, the mass spectrum initially chosen for display will be that produced when the concentration of the compound of interest is at its maximum (i.e. at the top of the peak) However, if it is suspected that the eluting peak is a mixture (i.e. two or more compounds are not satisfactorily separated by the GC column), or if the mass spectrum is saturated (due to the dynamic range of the mass spectrometer being exceeded), other spectra may be chosen for display.

An obvious indication that a mass spectrum is saturated, or overloaded, is provided by the presence of more than one peak in a mass spectrum at an intensity of 100%. Mass spectra from scans obtained before or after the intensity maximises should be inspected to obtain a representative mass spectrum for the compound of interest, although if a single spectrum is chosen it should be ascertained that it is not distorted ("skewed").

Mass spectra may be averaged across a peak (provided it is considered that the peak is due to a single compound) to minimise any distortion of the spectra which can occur if the concentration of a compound entering the mass spectrometer changes significantly during the course of a single mass spectrometer scan. This can occur when a GC peak is very sharp e.g. only 2-3 seconds wide. However, before averaging several spectra through a peak, each spectrum should be checked to ascertain whether any are saturated.

A background subtraction should also be performed, either on a mass spectrum from a single scan or on an averaged spectrum, in order to remove spurious peaks such as those produced by residual air in the mass spectrometer, or from GC column bleed.

The mass spectrum obtained for each peak detected is generally initially inspected visually. Depending on the experience of the mass spectroscopist, it may be possible to identify the compound giving rise to the spectrum without recourse to reference mass spectra held in a libraries (on the data system, or in reference books).

If the mass spectrum is not visually recognised, a library search is usually carried out on the data system. It is recommended that a reverse searching procedure should be used. The closeness of the match between the unknown and the chosen library spectra is usually expressed in terms of three parameters - fit, purity and reverse fit. However, the best match chosen by the data system does not necessarily lead to the identification of the unknown, and the mass spectroscopist has to apply his/her judgement, taking into account such factors as the GC retention time, in order to decide whether the identification suggested by the computerised library search is accepted. If there is any doubt concerning such an identification it is noted as a tentative identification, and if it is necessary to confirm the identification pure standard of the compound in question could be obtained and run on the GCMS system in order to check the mass spectrum obtained and the GC retention time. The same principles apply to potential identifications resulting from manual inspection of mass spectral reference

collections in books such as "The Eight Peak Index".

If it is supected that a TIC peak is a mixture of two or more compounds, mass chromatography may be of use in deciding whether this is the case, and by careful choice of mass spectra it may be possible to produce spectra corresponding to each co-eluting component. However, where two compounds have identical retention times this may not be possible, and further progress is dependent on the experience of the mass spectroscopist.

It is inevitable that a significant proportion of the compounds detected in many general survey GCMS runs will only be tentatively identified, and that some will be unidentified, as the reference collections of mass spectra currently available represent a very small proportion (< 10%) of the known organic compounds that are amenable to GCMS analysis.

2. Checking suitability of apparatus used for concentrating solvent extracts.

It is necessary to be able to reduce the volume of the dichloromethane solvent extracts from about 200 ml to 500 ul without significant losses of volatile components which may have been present in the leachate sample. To check that this can be satisfactorily achieved, it is recommended that a 500 ul portion of the GC column test standard solution (5.2 .4) is diluted to 200 ml with dichloromethane, and the resulting solution concentrated to 500 ul, using appropriate apparatus or equipment. This concentrate should be run on the GCMS system under exactly the same conditions as used when using the GC column test standard solution for checking for satisfactory GC performance, and the TIC trace compared to a TIC trace obtained when the GC column test standard is run. Provided the loss of the most volatile internal standard, d_6-benzene, is not more than 50% the technique used for the concentration of the solvent extracts is considered satisfactory.

3. Procedure for calculation of recoveries of internal standards.

The concentrations of the various internal standard solutions, the volume of the leachate analysed, the final volume of the solvent extract, and the volumes injected onto the GCMS system, as described in this protocol are such that the TIC chromatograms generated for the internal standards GC column test solution (5.2.4) and the concentrated extract (8.1.8) are directly comparable, so that the following equation can be used to calculate % recoveries:

$$\% \text{ Recovery} = \frac{\text{pk. area of } IS_n \text{ in extract}}{\text{pk. area of } IS_n \text{ in standard}} \times 100$$

4. Standard solutions for checking GC column performance.

Several chromatography supply companies produce mixtures specifically designed to evaluate the performance of GC columns, in terms of parameters such as column efficiency and adsorptive or 'active' sites. If the GC column used is from a manufacturer who does not provide a suitable test chromatogram, the column should be evaluated before use with solvent extracts of leachates, using this type of test mixture.

5. Example of Test Report.

This is given on the following pages.

6. Performance testing data for this protocol

A tabulated summary of the data obtained during the within-laboratory and interlaboratory performance testing of this protocol is given following the example of the test report. Competent laboratories intending to use this protocol should be able to produce comparable data.

EXAMPLE OF TEST REPORT

[**NB** - *only single examples of the various outputs (TIC chromatograms, mass spectra etc.) are provided in this example. Test reports produced as a result of using this protocol should contain all of the required outputs, as specified in Section 9 of the protocol*].

Test Report on the GCMS analysis of Leachates samples produced from pipe leaching tests by The General Survey GCMS method (BSI Protocol xxxx)

Report prepared for:

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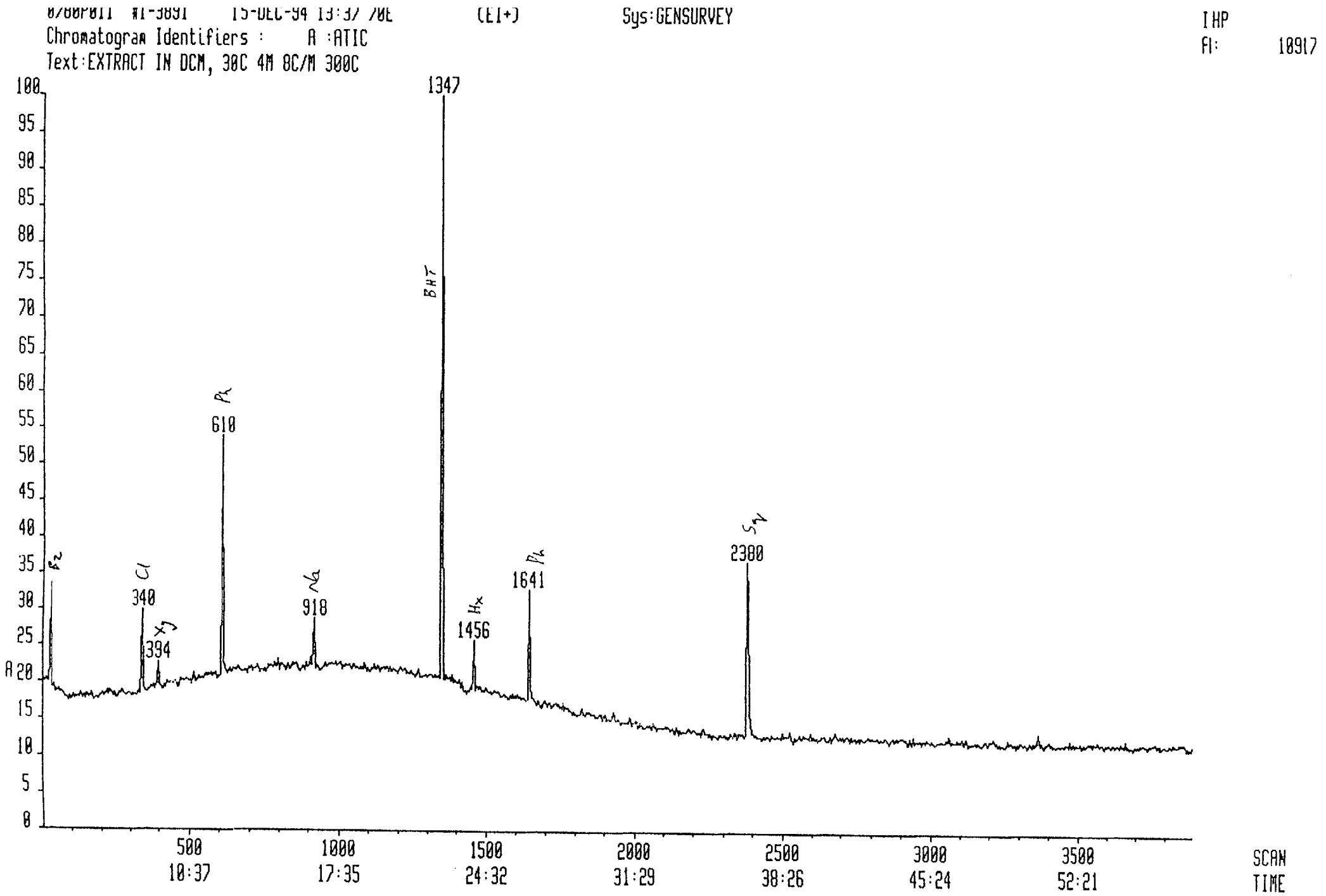
MEDMENHAM

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	xxxx	**Date Sampled:**	09-Nov-94
Analyst:	RK	**Data System Code:**	xxxx	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	xxxx	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	Example results table				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0024	d6-Benzene	694	2.0	Bz	IS
0192	Toluene	250	0.7	Bz	Leachate
0339	d5-Chlorobenzene	1622	2.0	Cl	IS
0393	d10-p-Xylene	832	0.5	Xy	IS
0610	d5-Phenol	2749	8.0	Po	IS
0669	Dimethylester-butandioc acid	2479	1.3	Na	Leachate
0869	Unknown 65,91,120	780	0.4	Na	Leachate
0917	d8-Naphthalene	964	0.5	Na	IS
1306	Naphthalene	955	0.5	Na	Leachate
1340	Unknown 57,138,153,168 Quinone? C10 H16 O2?	1057	0.6	Ph	Leachate
1345	d20-BHT	14719	8.0	BHT	IS
1356	Dibutylphenol	1356	0.8	Ph	Leachate
1454	d34-Hexadecane	950	0.6	Ph	IS
1636	d10-Phenanthrene	3378	2.0	Ph	IS
1809	Dibutylphthlate	782		Ph	Contaminant
1987	Unknown 57,69,83,97	2961	1.8	Ph	Leachate
2364	Hexacosane	3255		Sq	Laboratory contaminant
2378	d62-Squalane	4065	8.0	Sq	IS

Internal standards used: Bz = d6-benzene, Cl = d5-chlorobenzene, Xy = d10-p-xylene, Po = d5-Phenol, Na = d8-Naphthelene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene.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IHP
FI: 10917
Chromatogram Identifiers : A :ATIC
Text:EXTRACT IN DCM, 30C 4M 8C/M 300C
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95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
A 20
15
10
5
0
1347
BHT
Pr
610
Fz
Cl
340
Xy
394
Na
918
Hx
1456
Pl
1641
Sq
2380
500
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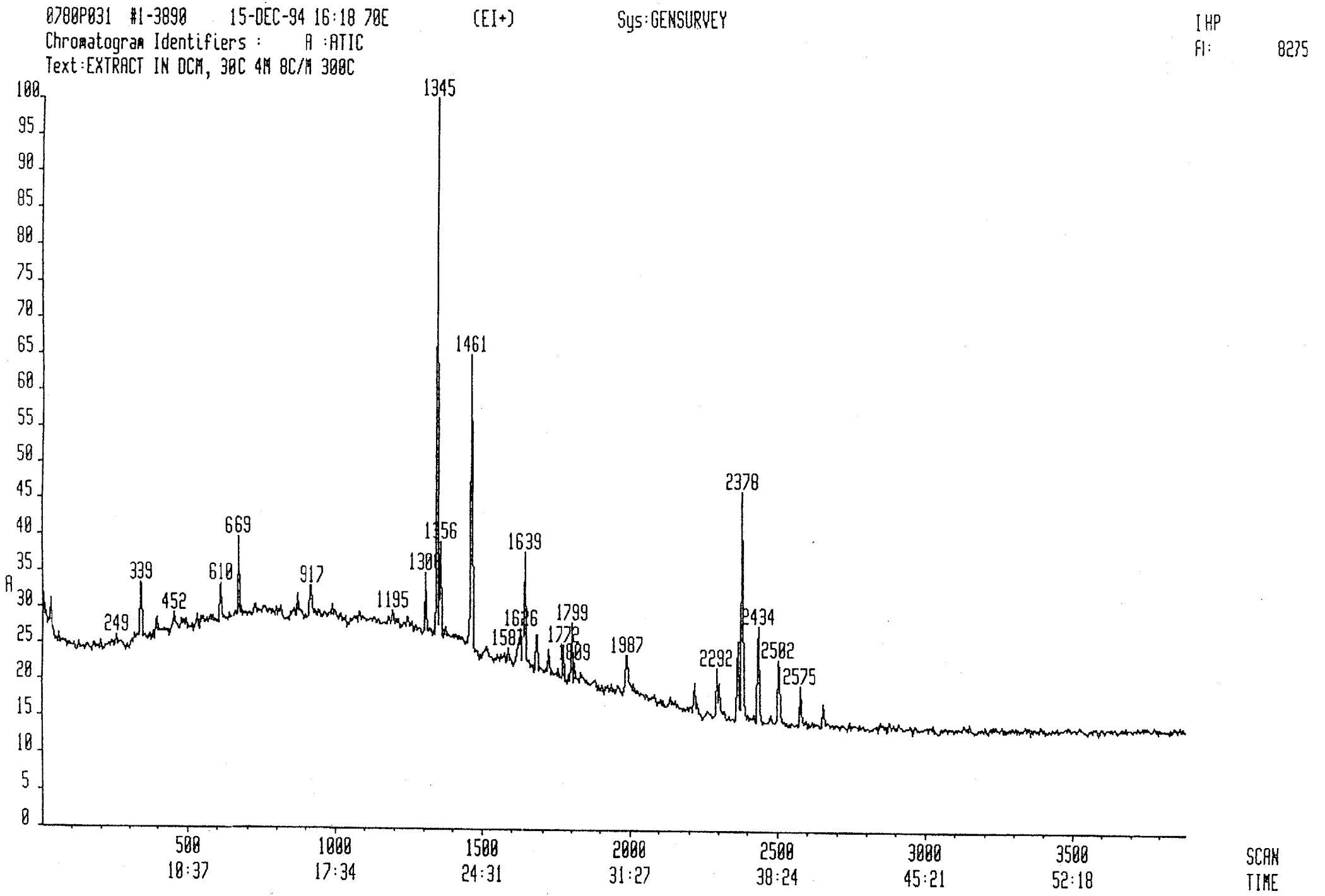
3500
52:21
SCAN
TIME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IHP
FI: 8275
Chromatogram Identifiers : A :ATIC
Text:EXTRACT IN DCM, 30C 4M 8C/M 300C
A
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
1345
1461
2378
669
1356
1639
339
610
917
452
249
1195
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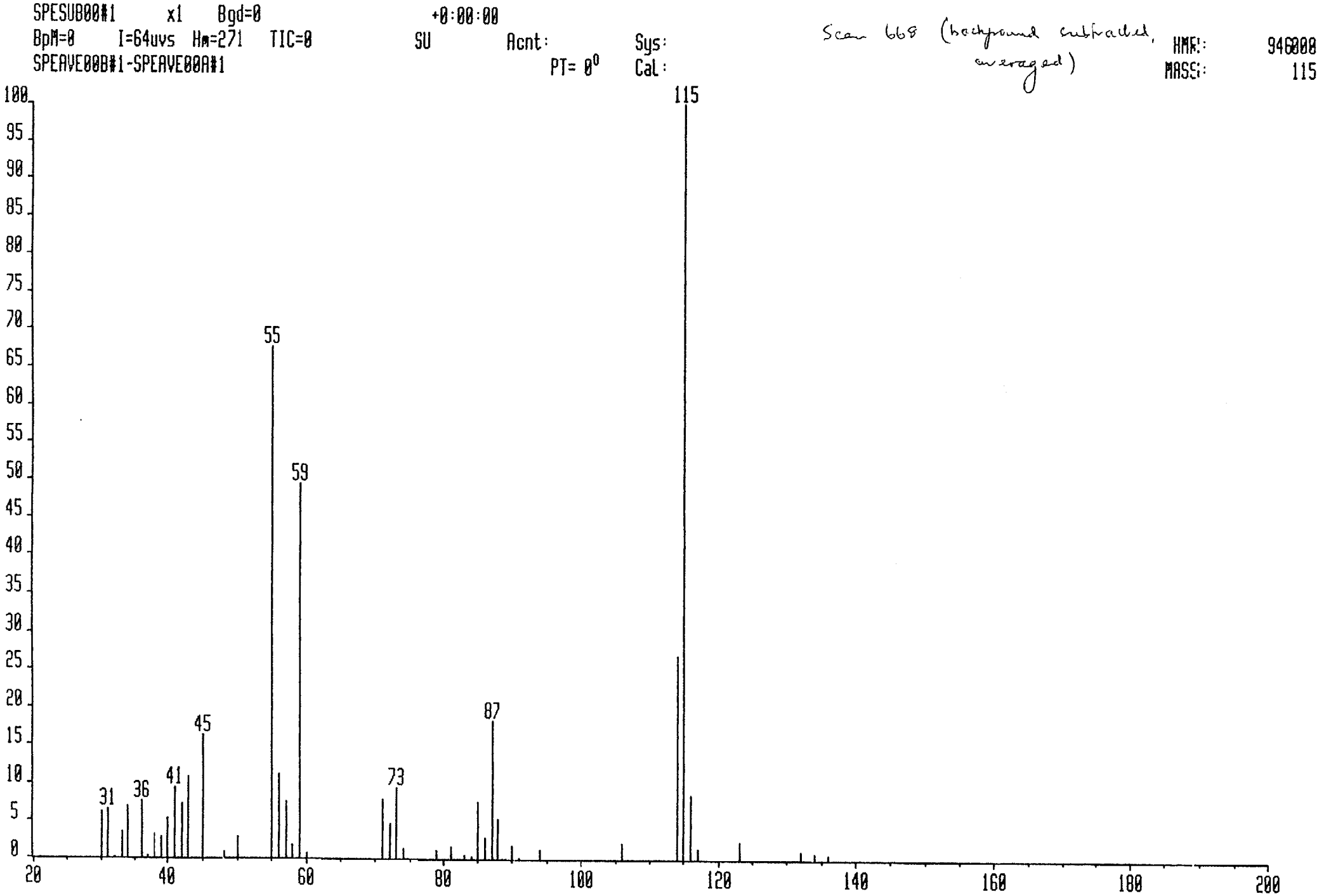
SCAN
TIME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BpM=0 I=64uvs Hm=271 TIC=0 SU Acnt: Sys:
SPEAVE00B#1-SPEAVE00A#1 PT= 0° Cal:
Scan 668 (background subtracted, averaged)
HMF: 946000
MASS: 115
115
55
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87
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20
40
60
80
100
120
140
160
180
200

Summary of variation (%RSD) for internal standards in within-laboratory and interlaboratory performance testing

Laboratory	Batch number	%RSD for peak areas of internal standards								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalen	d20-BHT	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
WRc	*1*	*48*	*24*	*36*	*24*	*33*	*57*	*23*	*11*	*26*
	2	*39*	*31*	*8*	*35*	*23*	*39*	*45*	*32*	*32*
1	1	16	15	37	24	18	23	52	15	26
	2	25	19	27	51	42	16	21	19	21
2	1	30	19	26	24	21	11	19	14	19
	2	15	20	19	35	19	14	32	15	30
3	1/2		10	8	12	12	14	15	9	47

The results from the within-laboratory testing carried out by WRc are presented in the above table in italics. The remaining results are from the interlaboratory testing; four laboratories participated but as one did not comply with the protocol, these data have been omitted. Two of the participating laboratories were within the UK and two were outside the UK, but within the European Union.

APPENDIX C

DATA TABLES (INTERNAL STANDARDS AND OTHER COMPOUNDS DETECTED) FOR GCMS RUNS UNDERTAKEN FOR WITHIN-LABORATORY PERFORMANCE TESTING OF PROTOCOLS

GC-MS General Survey AQC results

Peak areas of internal standards detected in leachates (within laboratory performance testing)

Project No: 8782-0
Form Number: 1307/1313
Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate (Batch 1)

Sample	Data sys Code	Peak Area								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (1)	0780P031	694	1622	832	2749	964	14691	941	3381	12275
PE-TW-CI (1)	0780P111	800	1265	531	3202	848	7726	776	3234	11517
GRP-TW (1)	0780P141	347	1433	416	2638	271	10424	1324	2484	12974
GRP-TW-CI (1)	0780P121	308	1068	257	1537	485	0	768	3533	7974
BIT-TW (1)	0780P101	185	655	480	2523	632	7802	700	3017	5796
BIT-TW-CI (1)	0780P051	836	1401	697	3348	933	10668	882	2902	8974
PB-TW (1)	0780P071	364	1489	392	2329	801	12885	863	2870	10332
PB-TW-CI (1)	0780P041	521	1212	422	1965	723	3494	717	2869	7846
Mean		**507**	**1268**	**503**	**2536**	**707**	**8461**	**871**	**3036**	**9711**
SD		**245**	**302**	**183**	**601**	**236**	**4845**	**201**	**335**	**2485**
%RSD		**48%**	**24%**	**36%**	**24%**	**33%**	**57%**	**23%**	**11%**	**26%**

Column test Results:

CCM Coltest	0780P011	3427	2867	1212	8348	1548	21027	2163	4958	11951
CCM Coltest	0780P081	3440	2921	1025	7166	1418	18890	1662	3732	11602
Mean		**3434**	**2894**	**1119**	**7757**	**1483**	**19959**	**1913**	**4345**	**11777**
SD		**9**	**38**	**132**	**836**	**92**	**1511**	**354**	**867**	**247**
%RSD		**0%**	**1%**	**12%**	**11%**	**6%**	**8%**	**19%**	**20%**	**2%**

GC-MS General Survey AQC results

Peak area ratios (normalised to d62-squalane) (within laboratory testing)

Project No: 8782-0
Form Number: 1307/1313
Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate (Batch 1)

Sample	Data sys Code	Peak area ratios (normalised to d62-Squalane)								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (1)	0780P031	0.0565	0.1321	0.0678	0.2240	0.0785	1.1968	0.0767	0.2754	1.0000
PE-TW-Cl (1)	0780P111	0.0695	0.1098	0.0461	0.2780	0.0736	0.6708	0.0674	0.2808	1.0000
GRP-TW (1)	0780P141	0.0267	0.1105	0.0321	0.2033	0.0209	0.8035	0.1021	0.1915	1.0000
GRP-TW-Cl (1)	0780P121	0.0386	0.1339	0.0322	0.1928	0.0608	0.0000	0.0963	0.4431	1.0000
BIT-TW (1)	0780P101	0.0319	0.1130	0.0828	0.4353	0.1090	1.3461	0.1208	0.5205	1.0000
BIT-TW-Cl (1)	0780P051	0.0932	0.1561	0.0777	0.3731	0.1040	1.1888	0.0983	0.3234	1.0000
PB-TW (1)	0780P071	0.0352	0.1441	0.0379	0.2254	0.0775	1.2471	0.0835	0.2778	1.0000
PB-TW-Cl (1)	0780P041	0.0664	0.1545	0.0538	0.2504	0.0921	0.4453	0.0914	0.3657	1.0000
Mean		**0.0523**	**0.1318**	**0.0538**	**0.2728**	**0.0771**	**0.8623**	**0.0921**	**0.3348**	**1.0000**
SD		**0.0231**	**0.0191**	**0.0202**	**0.0869**	**0.0278**	**0.4718**	**0.0165**	**0.1052**	**0.0000**
%RSD		**44%**	**14%**	**38%**	**32%**	**36%**	**55%**	**18%**	**31%**	**0%**

Column test Results:

CCM Coltest	0780P011	0.2868	0.2399	0.1014	0.6985	0.1295	1.7594	0.1810	0.4149	1.0000
CCM Coltest	0780P081	0.2965	0.2518	0.0883	0.6177	0.1222	1.6282	0.1433	0.3217	1.0000
Mean		**0.2916**	**0.2458**	**0.0949**	**0.6581**	**0.1259**	**1.6938**	**0.1621**	**0.3683**	**1.0000**
SD		**0.0069**	**0.0084**	**0.0092**	**0.0572**	**0.0052**	**0.0928**	**0.0267**	**0.0659**	**0.0000**
%RSD		**2%**	**3%**	**10%**	**9%**	**4%**	**5%**	**16%**	**18%**	**0%**

GC-MS General Survey AQC results

Retention times of internal standards (within laboratory performance testing)

Project No: 8782-0
Form Number: 1307/1313
Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate (Batch 1)

Sample	Data sys	Retention time (minutes)								
	Code	d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (1)	0780P031	4.00	8.38	9.13	12.15	16.41	22.36	23.88	26.45	36.72
PE-TW-CI (1)	0780P111	4.01	8.39	9.13	12.13	16.41	22.36	23.88	26.45	36.71
GRP-TW (1)	0780P141	3.99	8.36	9.10	12.15	16.39	22.33	23.85	26.42	36.67
GRP-TW-CI (1)	0780P121	4.00	8.36	9.10	12.12	16.39		23.85	26.42	36.68
BIT-TW (1)	0780P101	3.99	8.36	9.12	12.15	16.39	22.35	23.86	26.43	36.69
BIT-TW-CI (1)	0780P051	4.00	8.39	9.13	12.16	16.41	22.36	23.88	26.46	36.72
PB-TW (1)	0780P041	4.00	8.38	9.12	12.15	16.40	22.36	23.88	26.45	36.73
PB-TW-CI (1)	0780P071	4.01	8.39	9.14	12.13	16.41	22.36	23.88	26.45	36.72
Mean		**4.00**	**8.38**	**9.12**	**12.14**	**16.40**	**22.36**	**23.87**	**26.44**	**36.71**
SD		0.011	0.013	0.015	0.013	0.014	0.011	0.013	0.015	0.024
%RSD		0.26%	0.15%	0.16%	0.10%	0.08%	0.05%	0.05%	0.06%	0.07%

Column test Results:

CCM Coltest	0780P011	4.00	8.36	9.10	12.15	16.40	22.33	23.88	26.41	36.64
CCM Coltest	0780P081	4.00	8.35	9.12	12.15	16.39	22.32	23.85	26.41	36.64
Mean		**4.00**	**8.36**	**9.11**	**12.15**	**16.39**	**22.33**	**23.86**	**26.41**	**36.64**
SD		0.000	0.010	0.010	0.000	0.010	0.010	0.020	0.000	0.000
%RSD		0.00%	0.12%	0.11%	0.00%	0.06%	0.04%	0.08%	0.00%	0.00%

GC-MS General Survey AQC results

Relative retention times of internal standards (within laboratory testing)

Project No: 8782-0
Form Number: 1307/1313
Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate (Batch 1)

Sample	Data sys Code	Relative retention times (normalised to d62-squalane)								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (1)	0780P031	0.1089	0.2282	0.2486	0.3308	0.4470	0.6090	0.6502	0.7203	1.0000
PE-TW-CI (1)	0780P111	0.1094	0.2286	0.2487	0.3305	0.4471	0.6092	0.6505	0.7205	1.0000
GRP-TW (1)	0780P141	0.1087	0.2281	0.2482	0.3313	0.4469	0.6091	0.6505	0.7206	1.0000
GRP-TW-CI (1)	0780P121	0.1091	0.2281	0.2481	0.3304	0.4467		0.6502	0.7203	1.0000
BIT-TW (1)	0780P101	0.1086	0.2280	0.2484	0.3310	0.4465	0.6091	0.6504	0.7204	1.0000
BIT-TW-CI (1)	0780P051	0.1089	0.2286	0.2486	0.3311	0.4470	0.6090	0.6502	0.7206	1.0000
PB-TW (1)	0780P071	0.1093	0.2286	0.2490	0.3304	0.4470	0.6090	0.6502	0.7203	1.0000
PB-TW-CI (1)	0780P041	0.1089	0.2281	0.2481	0.3306	0.4464	0.6087	0.6500	0.7200	1.0000
Mean		**0.1090**	**0.2283**	**0.2485**	**0.3308**	**0.4468**	**0.6090**	**0.6503**	**0.7204**	**1.0000**
SD		**0.0003**	**0.0003**	**0.0003**	**0.0003**	**0.0002**	**0.0001**	**0.0002**	**0.0002**	**0.0000**
%RSD		**0.23%**	**0.12%**	**0.12%**	**0.10%**	**0.05%**	**0.02%**	**0.02%**	**0.03%**	**0.00%**

Column test Results:

CCM Coltest	0780P011	0.1092	0.2283	0.2484	0.3315	0.4476	0.6096	0.6517	0.7208	1.0000
CCM Coltest	0780P081	0.1092	0.2279	0.2488	0.3315	0.4472	0.6092	0.6510	0.7208	1.0000
Mean		**0.1092**	**0.2281**	**0.2486**	**0.3315**	**0.4474**	**0.6094**	**0.6513**	**0.7208**	**1.0000**
SD		**0.0000**	**0.0003**	**0.0003**	**0.0000**	**0.0003**	**0.0003**	**0.0005**	**0.0000**	**0.0000**
%RSD		**0.00%**	**0.12%**	**0.11%**	**0.00%**	**0.06%**	**0.04%**	**0.08%**	**0.00%**	**0.00%**

GC-MS General Survey AQC results

Peak areas of internal standards detected in leachates (within laboratory performance testing)

Project No: 8782-0
Form Number: 1307/1313
Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate (Batch 2)

Sample	Data sys	Peak Area								
	Code	d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (2)	0780P191	260	710	416	690	492	4320	211	1733	3656
GRP-TW (2)	0780P211	270	1212	426	1773	914	7934	465	2008	8924
GRP-TW-Cl (2)	0780P201	303	1027	418	1417	522	14511	1028	3025	11766
BIT-TW (2)	0780P171	316	929	403	895	622	7421	628	2463	4899
BIT-TW-Cl (2)	0780P181	637	1736	480	1202	529	11673	459	4035	5752
PB-TW (2)	0780P131	479	1500	366	1679	674	11848	620	2124	9142
PB-TW-Cl (2)	0780P061	582	943	423	1997	750	6356	911	2679	9093
Mean		**407**	**1151**	**419**	**1379**	**643**	**9152**	**617**	**2402**	**9118**
SD		**157**	**358**	**34**	**478**	**151**	**3605**	**279**	**774**	**2886**
%RSD		**39%**	**31%**	**8%**	**35%**	**23%**	**39%**	**45%**	**32%**	**32%**

Column test Results:

CCM Coltest	0780P221	3333	3101	1277	3417	1068	19723	1390	3492	12961
CCM Coltest	0780P151	3895	3400	986	7903	1086	18756	1695	4506	11325
Mean		**3614**	**3251**	**1132**	**5660**	**1077**	**19240**	**1543**	**3999**	**12143**
SD		**397**	**211**	**206**	**3172**	**13**	**684**	**216**	**717**	**1157**
%RSD		**11%**	**7%**	**18%**	**56%**	**1%**	**4%**	**14%**	**18%**	**10%**

GC-MS General Survey AQC results

Peak area ratios (normalised to d62-squalane) (within laboratory testing)

Project No: 8782-0
Form Number: 1307/1313
Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate (Batch 2)

Sample	Data sys Code	Peak Area ratios								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (2)	0780P191	0.0711	0.1942	0.1138	0.1887	0.1346	1.1816	0.0577	0.4740	1.0000
GRP-TW (2)	0780P211	0.0303	0.1358	0.0477	0.1987	0.1024	0.8891	0.0521	0.2250	1.0000
GRP-TW-CI (2)	0780P201	0.0258	0.0873	0.0355	0.1204	0.0444	1.2333	0.0874	0.2571	1.0000
BIT-TW (2)	0780P171	0.0645	0.1896	0.0823	0.1827	0.1270	1.5148	0.1282	0.5028	1.0000
BIT-TW-CI (2)	0780P181	0.1107	0.3018	0.0834	0.2090	0.0920	2.0294	0.0798	0.7015	1.0000
PB-TW (2)	0780P131	0.0524	0.1641	0.0400	0.1837	0.0737	1.2960	0.0678	0.2323	1.0000
BIT-TW (2)	0780P171	0.0640	0.1037	0.0465	0.2196	0.0825	0.6990	0.1002	0.2946	1.0000
Mean		**0.0598**	**0.1681**	**0.0642**	**0.1861**	**0.0938**	**1.2633**	**0.0819**	**0.3839**	**1.0000**
SD		**0.0284**	**0.0716**	**0.0293**	**0.0320**	**0.0311**	**0.4315**	**0.0264**	**0.1805**	**0.0000**
%RSD		**48%**	**43%**	**46%**	**17%**	**33%**	**34%**	**32%**	**47%**	**0%**

Column test Results:

CCM Coltest	0780P221	0.2572	0.2393	0.0985	0.2636	0.0824	1.5217	0.1072	0.2694	1.0000
CCM Coltest	0780P151	0.3439	0.3002	0.0871	0.6978	0.0959	1.6562	0.1497	0.3979	1.0000
Mean		**0.3005**	**0.2697**	**0.0928**	**0.4807**	**0.0891**	**1.5889**	**0.1285**	**0.3337**	**1.0000**
SD		**0.0614**	**0.0431**	**0.0081**	**0.3070**	**0.0095**	**0.0951**	**0.0300**	**0.0908**	**0.0000**
%RSD		**20%**	**16%**	**9%**	**64%**	**11%**	**6%**	**23%**	**27%**	**0%**

GC-MS General Survey AQC results

Retention times of internal standards

Project No: 8782-0
Form Number: 1307/1313
Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate (Batch 2)

Sample	Data sys Code	Retention time (minutes)								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (2)	0780P191	4.00	8.35	9.12	12.15	16.39	22.32	23.85	26.41	36.64
GRP-TW (2)	0780P211	4.01	8.38	9.12	12.13	16.39	22.35	23.85	26.45	36.67
GRP-TW-CI (2)	0780P201	3.96	8.35	9.12	12.12	16.39	22.39	23.84	26.42	36.65
BIT-TW (2)	0780P171	4.00	8.36	9.12	12.15	16.39	22.35	23.86	26.43	36.65
BIT-TW-CI (2)	0780P181	4.00	8.36	9.10	12.15	16.40	22.33	23.88	26.41	36.64
PB-TW (2)	0780P131	3.99	8.35	9.10	12.12	16.40	22.33	23.84	26.43	36.67
PB-TW-CI (2)	0780P061	4.00	8.39	9.12	12.13	16.41	22.36	23.88	26.45	36.72
Mean		**3.99**	**8.36**	**9.11**	**12.14**	**16.40**	**22.35**	**23.86**	**26.43**	**36.66**
SD		**0.016**	**0.017**	**0.009**	**0.014**	**0.009**	**0.024**	**0.016**	**0.017**	**0.028**
%RSD		**0.41%**	**0.20%**	**0.10%**	**0.11%**	**0.05%**	**0.11%**	**0.07%**	**0.06%**	**0.08%**

Column test Results:

CCM Coltest	0780P221	3.96	8.35	9.12	12.12	16.39	22.39	23.84	26.42	36.65
CCM Coltest	0780P151	4.01	8.38	9.12	12.13	16.39	22.35	23.85	26.45	36.67
Mean		**3.99**	**8.36**	**9.12**	**12.12**	**16.39**	**22.37**	**23.84**	**26.43**	**36.66**
SD		**0.039**	**0.020**	**0.000**	**0.010**	**0.000**	**0.029**	**0.010**	**0.020**	**0.010**
%RSD		**0.99%**	**0.24%**	**0.00%**	**0.08%**	**0.00%**	**0.13%**	**0.04%**	**0.07%**	**0.03%**

GC-MS General Survey AQC results

Relative retention times of internal standards (within laboratory testing)

Project No: 8782-0
Form Number: 1307/1313
Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate (Batch 2)

Sample	Data sys	Relative Retention times								
	Code	d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (2)	0780P191	0.1092	0.2279	0.2489	0.3316	0.4473	0.6092	0.6509	0.7208	1.0000
GRP-TW (2)	0780P211	0.1094	0.2285	0.2487	0.3308	0.4470	0.6095	0.6504	0.7213	1.0000
GRP-TW-Cl (20	0780P201	0.1080	0.2278	0.2488	0.3307	0.4472	0.6109	0.6505	0.7209	1.0000
BIT-TW (20	0780P171	0.1091	0.2281	0.2488	0.3315	0.4472	0.6098	0.6510	0.7211	1.0000
BIT-TW-Cl (2)	0780P181	0.1092	0.2282	0.2484	0.3316	0.4476	0.6094	0.6517	0.7208	1.0000
PB-TW (2)	0780P131	0.1088	0.2277	0.2482	0.3305	0.4472	0.6089	0.6501	0.7208	1.0000
PB-TW-Cl (2)	0780P061	0.1089	0.2286	0.2482	0.3304	0.4470	0.6090	0.6502	0.7203	1.0000
Mean		**0.1089**	**0.2281**	**0.2486**	**0.3310**	**0.4472**	**0.6095**	**0.6507**	**0.7209**	**1.0000**
SD		**0.0004**	**0.0003**	**0.0003**	**0.0005**	**0.0002**	**0.0007**	**0.0006**	**0.0003**	**0.0000**
%RSD		**0.40%**	**0.15%**	**0.13%**	**0.16%**	**0.05%**	**0.11%**	**0.09%**	**0.04%**	**0.00%**

Column test Results:

CCM Coltest	0780P221	0.1080	0.2278	0.2487	0.3306	0.4471	0.6109	0.6503	0.7209	1.0000
CCM Coltest	0780P151	0.1095	0.2285	0.2486	0.3309	0.4469	0.6095	0.6505	0.7214	1.0000
Mean		**0.1087**	**0.2282**	**0.2487**	**0.3308**	**0.4470**	**0.6102**	**0.6504**	**0.7211**	**1.0000**
SD		**0.0010**	**0.0005**	**0.0001**	**0.0002**	**0.0001**	**0.0010**	**0.0001**	**0.0003**	**0.0000**
%RSD		**0.96%**	**0.21%**	**0.03%**	**0.05%**	**0.03%**	**0.16%**	**0.01%**	**0.05%**	**0.00%**

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P131	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	Blank	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**			
Sample Type:	BLANK Unchlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0023	d6-Benzene	479	2.0		IS
0337	d5-Chlorobenzene	1500	2.0		IS
0391	d10-p-Xylene	366	0.5		IS
0610	d5-Phenol	1679	8.0		IS
0916	d8-Naphthalene	674	0.5		IS
1343	d20-BHT	11848	8.0		IS
1452	d34-Hexadecane	620	0.6		IS
1458	Texanol isobutyrate		NQ		GC artefact
1632	d10-Phenanthrene	2124	2.0		IS
1717	Dibutylphthalate isomer	1371	NQ		Contaminant
1805	Dibutylphthalate isomer	1291	NQ		Contaminant
2295	Dioctylphthalate	870	NQ		Contaminant
2373	d62-Squalane	9142	8.0		IS

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Po=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P071	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	Blank	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**			
Sample Type:	BLANK Unchlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0025	d6-Benzene	364	2.0		IS
0340	d5-Chlorobenzene	1489	2.0		IS
0393	d10-p-Xylene	392	0.5		IS
0610	d5-Phenol	2329	8.0		IS
0917	d8-Naphthalene	801	0.5		IS
1195	Unknown 43,58,41,71	342	NQ		Contaminant
1345	d20-BHT	12885	8.0		IS
1427	Diethylphthalate	303	NQ		Contaminant
1454	d34-Hexadecane	863	0.5		IS
1461	Texanol isobutyrate		NQ		GC artefact
1639	d10-Phenanthrene	2870	2.0		IS
1720	Dibutylphthalate isomer	1167	NQ		Contaminant
1808	Dibutylphthalate isomer	2674	NQ		Contaminant
1828	Palmitic acid	905	NQ		
1986	Unknown 55,41,43,69	2152	NQ		Contaminant
2007	Unknown 55,43,41,73 Stearic acid	1321	NQ		
2195	Dioctyl adipate	1146	NQ		Contaminant
2217	Unknown 57,71,43,85	618	NQ		Contaminant
2291	Unknown 57,43,71,85	1402	NQ		Contaminant
2300	Dioctylphthalate isomer	6364	NQ		Contaminant
2364	Unknown 57,43,71,85	2699	NQ		Contaminant
2378	d62-Squalane	10332	8.0		IS
2433	Unknown 57,43,71,85	3207	NQ		Contaminant
2502	Unknown 57,43,71,85	2814	NQ		Contaminant

Internal standards used: Bz = d6-benzene, Cl = d5-chlorobenzene, Xy = d10-p-xylene, Po = d5-Phenol, Na = d8-Naphthelene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P071	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	Blank	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**			
Sample Type:	BLANK Unchlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
2574	Unknown 57,43,71,85	1662	NQ		Contaminant
2654	Unknown 57,43,71,85	1036	NQ		Contaminant

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Ph=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene.

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P041	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	Blank	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**			
Sample Type:	BLANK Chlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0024	d6-Benzene	521	2.0		IS
0030	Chloroacetone		NQ		Chlorination artefact
0072	Chloromethylbutene		NQ		Chlorination artefact
0260	Unknown 59,107,109		NQ		Chlorination artefact
0299	Unknown 77,41,79,69		NQ		Chlorination artefact
0323	Unknown 73,93,55,43		NQ		Chlorination artefact
0329	d5-Chlorobenzene	1212	2.0		IS
0392	d10-p-Xylene	422	0.5		IS
0601	Unknown 43,58	1162	NQ		Contaminant
0610	d5-Phenol	1965	8.0		IS
0652	Unknown 43,45,59	554	NQ		Contaminant
0916	d8-Naphthalene	723	0.5		IS
1345	d20-BHT	3494	8.0		IS
1427	Diethyl phthalate	523	NQ		Contaminant
1454	d34-Hexadecane	717	0.5		IS
1461	Texanol isobutyrate		NQ		GC artefact
1639	d10-Phenanthrene	2869	2.0		IS
1720	Dibutylphthalate isomer	1136	NQ		Contaminant
1808	Dibutylphthalate isomer	1491	NQ		Contaminant
2300	Dioctylphthalate isomer	971	NQ		Contaminant
2378	d62-Squalane	7846	8.0		IS

Internal standards used: Bz = d6-benzene, Cl = d5-chlorobenzene, Xy = d10-p-xylene, Po = d5-Phenol, Na = d8-Naphthelene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P061	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	Blank	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**			
Sample Type:	BLANK Chlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0024	d6-Benzene	582	2.0		IS
0072	Chloromethylbutene		NQ		Chlorination artefact
0258	Unknown 59,107,109		NQ		Chlorination artefact
0299	Unknown 77,41,79,69		NQ		Chlorination artefact
0240	d5-Chlorobenzene	943	2.0		IS
0392	d10-p-Xylene	423	0.5		IS
0610	d5-Phenol	1997	8.0		IS
0917	d8-Naphthalene	750	0.5		IS
1345	d20-BHT	6356	8.0		IS
1454	d34-Hexadecane	911	0.5		IS
1460	Texanol isobutyrate		NQ		GC artefact
1639	d10-Phenanthrene	2679	2.0		IS
1720	Dibutylphthalate isomer	608	NQ		Contaminant
1808	Dibutylphthalate isomer	870	NQ		Contaminant
1827	Palmitic acid	432	NQ		
1985	Unknown 55,41,43,69	1496	NQ		Contaminant
2291	Unknown 57,43,71,85	889	NQ		Contaminant
2299	Dioctylphthalate isomer	3974	NQ		Contaminant
2363	Unknown 57,43,71,85	1052	NQ		Contaminant
2378	d62-Squalane	9023	8.0		IS
2433	Unknown 57,43,71,85	1741	NQ		Contaminant
2501	Unknown 57,43,71,85	1148	NQ		Contaminant
2574	Unknown 57,43,71,85	1106	NQ		Contaminant

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Po=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P101	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	BIT	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	Bitumen no chlorine				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0023	d6-Benzene	185	2.0		IS
0338	d5-Chlorobenzene	655	2.0		IS
0391	d10-p-Xylene	480	0.5		IS
0610	d5-Phenol	2523	8.0		IS
0915	d8-Naphthalene	632	0.5		IS
1344	d20-BHT	7802	8.0		IS
1426	Diethyl phthalate	1098	NQ		In blank
1452	d34-Hexadecane	700	0.1		IS
1460	Texanol-iso-butyrate		NQ		GC artefact
1638	d10-Phenanthrene	3017	2.0		IS
1718	Dibutylphthalate isomer	1492	NQ		In blank
1807	Dibutylphthalate isomer	1916	NQ		In blank
2299	Phthalate	1658	NQ		In blank
2363	Unknown 57,43,71,	384	NQ		In blank
2376	d62-Squalane	5796	8.0		IS
2431	Unknown 57,43,71	417	NQ		In blank

Internal standards used: Bz = d6-benzene, Cl = d5-chlorobenzene, Xy = d10-p-xylene, Po = d5-Phenol, Na = d8-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62 Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P171	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	BIT	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	Bitumen no chlorine				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0024	d6-Benzene	316	2.0		IS
0038	Cyclohexane	570	3.6	Bz	Test material ?
0339	d5-Chlorobenzene	929	2.0		IS
0392	d10-p-Xylene	403	0.5		IS
0610	d5-Phenol	895	8.0		IS
0917	d8-Naphthalene	622	0.5		IS
1344	d20-BHT	7421	8.0		IS
1425	Diethylphthalate isomer	863	NQ		In blank
1452	d34-Hexadecane	628	0.5		IS
1459	Texanol isobutyrate		NQ		GC artefact
1638	d10-Phenanthrene	2463	2.0		IS
1718	Dibutylphthalate isomer	772	NQ		In blank
1806	Dibutylphthalate isomer	915	NQ		In blank
2374	d62-Squalane	4899	8.0		IS

Internel standards used: Bz = d6-benzene, Cl = d5-chlorobenzene, Xy = d10-p-xylene, Po = d5-Phenol, Na = d8-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P051	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	BIT	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	Bitumen chlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0024	d6-Benzene	836	2.0		IS
0248	Unknown 56,41,44,43 (Hexanal ?)	1203	1.7	Cl	Test material ?
0340	d5-Chlorobenzene	1401	2.0		IS
0384	m-Xylene	342	0.3	Xy	Test material
0393	d10-p-Xylene	679	0.5		IS
0401	p-Xylene & Ethyl benzene	669	0.5	Xy	Test material
0443	o-Xylene	216	0.2	Xy	Test material
0451	Unknown 70,41,43,55	449	0.3	Xy	Test material
0539	Benzaldehyde	442	0.2	Na	Test material
0611	d5-Phenol	3348	8.0		IS
0723	Unknown 105,77,51,120	555	0.3	Na	Test material
0804	Unknown 41,43,56,55	1185	0.6	Na	Test material
0918	d8-Naphthalene	933	0.5		IS
0921	Naphthalene	1265	0.7	Na	Test material
1058	Unknown 36,43,60,73	2446	1.3	Na	Test material
1346	d20-BHT	10668	8.0		IS
1427	Diethyl phthalate isomer	873	NQ		In blank
1454	d34-Hexadecane	882	0.5		IS
1461	Texanol isobutyrate		NQ		GC artefact
1640	d10-Phenanthrene	2902	2.0		IS
1721	Dibutylphthalate isomer	1175	NQ		In blank
1809	Dibutylphthalate isomer	1605	NQ		In blank
1987	Unknown 41,55,67,81	539	NQ		In blank
2292	Unknown 57,71,43,41	859	NQ		In blank

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Po=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P051	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	BIT	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	Bitumen chlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
2300	Dioctylphthalate isomer	1224	NQ		In blank
2364	Unknown 57,43,71,41	1620	NQ		In blank
2378	d62-Squalane	8974	8.0		IS
2454	Unknown 57,43,71,85	1637	NQ		In blank
2582	Unknown 57,43,71,85	1149	NQ		In blank
2574	Unknown 57,43,71,41	1167	NQ		In blank

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Ph=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene.

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P181	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	BIT	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	Bitumen chlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0024	d6-Benzene	637	2.0		IS
0247	Unknown 56,44,41,43 (Hexanal ?)	778	0.9	Cl	Test material ?
0255	Unknown 59,43,107,109		NQ		Chlorination artefact
0338	d5-Chlorobenzene	1736	2.0		IS
0383	m-Xylene	296	0.3	Xy	Test material
0391	d10-p-Xylene	480	0.5		IS
0400	p-xylene/ethylbenzene	250	0.3	Xy	Test material
0492	o-xylene	145	0.2	Xy	Test material
0449	Unknown 70,42,41,55 (Heptanal ?)	612	0.6	Xy	Test material ?
0610	d5-Phenol	1202	8.0		IS
0803	Unknown 41,57,43,29	956	0.9	Na	Test material
0915	d8-Naphthalene	529	0.5		IS
0919	Naphthalene	1036	1.0	Na	Test material
1064	Unknown 87,41,43,55	8243	7.8	Na	Test material
1308	Unknown 220, 172,238,188 (Breakdown product of d20-BHT)	1267	NQ		Artefact
1343	d20-BHT	11673	8.0		IS
1425	Diethyl phthalate	946	NQ		In blank
1452	d34-Hexadecane	459	0.5		IS
1459	Texanol isobutyrate		NQ		GC artefact
1637	d10-Phenanthrene	4035	2.0		IS
1717	Dibutylphthalate	1313	NQ		In blank
1806	Dibutylphthalate isomer	1168	NQ		In blank
1977	Unknown 36,67,81,55	1853	0.9	Ph	Test material ?
1984	Unknown 36,55,41,38	5178	NQ		In blank

Internal standards used: Bz = d6-benzene, Cl = d5-chlorobenzene, Xy = d10-p-xylene, Po = d5-Phenol, Na = d8-Naphthelene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P181	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	BIT	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	Bitumen chlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
2212	Unknown 57,43,71,41	353	NQ		In blank
2287	Unknown 57,43,71,85	1287	NQ		In blank
2295	Dioctylphthalate isomer	1211	NQ		In blank
2359	Unknown 43,57,71,85	2123	NQ		In blank
2373	d62-squalane	5752	2.0		IS
2420	Unknown 57,43,71,85	2721	NQ		In blank
2496	Unknown 57,43,71,85	2277	NQ		In blank
2567	Unknown 57,43,71,85	1983	NQ		In blank
2645	Unknown 57,43,71,55	1031	0.4	Sq	Test material ?

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Ph=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene.

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P031	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	PE	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	PE Unchlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0024	d6-Benzene	694	2.0		IS
0339	d5-Chlorobenzene	1622	2.0		IS
0384	Xylene isomer	203	0.1	Xy	Test material
0393	d10-p-Xylene	832	0.5		IS
0401	Ethylbenzene	93	0.1	Xy	Test material
0443	o-Xylene	240	0.1	Xy	Test material
0529	Unknown 76,71,36,43	258	0.2	Xy	Test material
0610	d5-Phenol	2749	8.0		IS
0669	Dimethylester-butandioc acid	2097	1.1	Na	Test material
0869	Unknown 65,120,91,92 (Phenyl oxirane ?)	646	0.3	Na	Test material
0917	d8-Naphthalene	964	0.5		IS
0921	Naphthalene	296	0.2	Na	Test material
1306	Unknown 177,41,220,67	1223	0.7	Ph	Test material
1340	Unknown 57,41,153,168	507	0.3	Ph	Test material
1345	d20-BHT	14691	8.0		IS
1356	Unknown 191,57,41,206	2253	1.3	Ph	Test material
1454	d34-Hexadecane	941	0.5		IS
1461	Texanol isobutyrate		NQ		GC artefact
1618	N-butylbenzenesulphonamide	458	0.3	Ph	Test material
1626	Dibutylethylphenol	983	0.6	Ph	Test material
1639	d10-Phenanthrene	3381	2.0		IS
1679	Dibutylpropylphenol	1447	0.9	Ph	Test material
1720	Dibutylphthalate isomer	738	NQ		In blank
1772	Unknown 57,41,43,205	1088	0.6	Ph	Test material

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Po=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P031	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	PE	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	PE Unchlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
1799	Unknown 277,57,41,147	1900	1.1	Ph	Test material
1809	Dibutylphthalate isomer	793	NQ		In blank
1987	Unknown 36,38,55,41	2795	NQ		In blank
2217	Unknown 57,43,71,85	879	NQ		In blank
2292	Unknown 57,43,71,85	2069	NQ		In blank
2300	Unknown 41,149,57,43	1503	NQ		In blank
2364	Unknown 53,43,71,85	3178	NQ		In blank
2378	d62-Squalane	12275	8.0		IS
2434	Unknown 57,43,71,85	4305	NQ		In blank
2502	Unknown 53,43,71,85	2896	NQ		In blank
2575	Unknown 57,43,71,85	2193	NQ		In blank
2654	Unknown 57,71,43,85	807	NQ		In blank

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Ph=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene.

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P111	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	PE	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	PE Chlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0024	d6-Benzene	80	2.0		IS
0031	Chloroacetone 43,92,49,94		NQ	Bx	Chlorination artefact
0064	Unknown 44,29,58,41 (3-methylbutanal ?)	368	0.6	Cl	
0072	Chloromethylbutene 69,41,53,89		NQ	Cl	Chlorination artefact
0089	Unknown 57,43,41,29		NQ	Cl	Chlorination artefact
0110	Unknown 59,93,29,31		NQ	Cl	Chlorination artefact
0170	Unknown 69,41,104,53		NQ	Cl	Chlorination artefact
0261	Unknown 59,43,31,107		NQ	Cl	Chlorination artefact
0299	Unknown 77,41,79,78		NQ	Cl	Chlorination artefact
0323	Unknown 73,93,29,55		NQ	Cl	Chlorination artefact
0340	d5-Chlorobenzene	1265	2.0		IS
0392	d10-p-Xylene	551	0.5		IS
0417	Unknown 59,43,31,41	17123	27.1	Cl	
0608	d5-Phenol	3202	8.0		IS
0669	Butandioic acid dimethyl ester 115,55,59,87	3265	5.2	Cl	Test material
0869	Unknown 65,120,91,92 (Phenyl oxirane ?)	581	0.3	Na	Test material
0917	d8-Naphthalene	848	0.5		IS
0934	Unknown 149,121,77,93	738	0.4	Na	Test material
1156	Unknown 135,183,91,41	790	0.5	Na	Test material
1301	Unknown 46,66,174,222	795	0.5	Ph	Test material
1306	2,6-Di-t-butyl-p-benzoquinone 177,41,67,220	1853	1.1	Ph	Test material
1310	Unknown 172,46,44,238	757	0.5	Ph	Test material
1340	Unknown 57,41,153,168	1049	0.6	Ph	Test material
1345	d20-BHT	7726	8.0		IS

Internal standards used: Bz = d6-benzene, Cl = d5-chlorobenzene, Xy = d10-p-xylene, Po = d5-Phenol, Na = d8-Naphthelene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P111	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	PE	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	PE Chlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
1417	Unknown 36,41,57,55	2004	1.2	Ph	Test material
1426	Diethyl phthalate	800	NQ		In blank
1454	d34-Hexadecane	776	0.5		IS
1461	Texanol isobutyrate		NQ		GC artefact
1528	Di-pentyl-p-benzoquinone isomer 177,191,41,163	2751	1.7	Ph	Test material
1554	Unknown 57,41,185,29	8569	5.3	Ph	Test material
1580	Unknown 41,57,39,91	1372	0.8	Ph	Test material
1633	Unknown 36,43,41,60 (Tetradecanoic acid ?)	1776	1.1	Ph	Test material ?
1639	d10-Phenanthrene	3234	2.0		IS
1720	Dibutylphthalate isomer	2302	NQ		In blank
1772	Unknown 57,41,43,205	4273			Test material
1808	Dibutylphthalate isomer	2860	NQ		In blank
1828	Palmitic acid	2166	1.3	Ph	Test material
2138	Unknown 57,43,71,85	424	0.3	Sq	Test material
2216	Unknown 57,43,71,85	1512	NQ		In blank
2250	Unknown 41,36,43,151	12543	8.7	Sq	Test material
2291	Unknown 57,43,71,85	2948	NQ		In blank
2299	Dioctyl phthalate isomer	1079	NQ		In blank
2321	Unknown 36,41,91,119	2358	1.6	Sq	Test material
2363	Unknown 57,43,71,85	5043	NQ		
2377	d62-Squalane	11517	8.0		IS
2432	Unknown 57,43,71,85	6378	NQ		In blank
2501	Unknown 57,43,71,85	4976	NQ		In blank
2573	Unknown 57,43,71,85	3421	NQ		In blank

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Ph=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene.

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P111	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	PE	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	PE Chlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
2652	Unknown 57,43,71,85	1404	NQ		In blank
2740	Unknown 57,43,71,85	408	NQ		In blank

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Ph=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P191	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	PE	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	PE Unchlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0023	d6-Benzene	260	2.0		IS
0337	d5-Chlorobenzene	710	2.0		IS
0391	d10-p-Xylene	416	0.5		IS
0610	d5-Phenol	690	8.0		IS
0668	Unknown 115,57,43,55	962	1.2	Xy	Test Material
0673	Unknown 70,44,99,51	418	0.5	Xy	Test Material
0868	Methyl oxirane	390	0.6	Na	Test Material
0915	d8-Naphthalene	335	0.5		IS
1303	Unknown 177,41,67,39	670	1.0	Na	Test Material
1337	Unknown 57,41,153,168	700	1.0	Na	Test Material
1342	d20-BHT	4320	8.0		IS
1353	Unknown 191,57,91,206	2157	2.5	Ph	Test Material
1452	d34-Hexadecane	211	0.5		IS
1458	Texanol butyrate		NQ		GC artefact
1623	Unknown 219,57,41,191	933	1.1	Ph	Test Material
1636	d10-Phenanthrene	1733	2.0		IS
1676	Unknown 233,43,248,205	1225	1.4	Ph	Test Material
1717	Dibutylphthalate isomer	310	NQ		In blank
1768	Unknown 41,57,205,55	2256	2.6	Ph	Test Material
1795	Unknown 277,57,147,41	1788	2.1	Ph	Test Material
1804	Dibutylphthalate	483	NQ		In blank
2295	Unknown 149,57,41,55	383	NQ		In blank
2372	d62-Squalane	3656	8.0		IS
2427	Unknown 57,71,43,85	446	NQ		In blank

Internal standards used: Bz = d6-benzene, Cl = d5-chlorobenzene, Xy = d10-p-xylene, Po = d5-Phenol, Na = d8-Naphthelene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P191	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	PE	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	PE Unchlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
2496	Unknown 57,43,71,85	323	NQ		In blank

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Ph=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene.

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P211	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	GRP	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	GRP Unchlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0021	d6-Benzene	270	2.0		IS
0194	Toluene	4951	36.7	Bz	Test material
0337	d5-Chlorobenzene	1212	2.0		IS
0381	m-Xylene	432	0.5	Xy	Test material
0390	d10-p-Xylene	426	0.5		IS
0608	d5-Phenol	1773	8.0		IS
0686	Isophorone	4548	2.5	Na	Test material
0918	d8-Naphthalene	914	0.5		IS
0990	Unknown 57,41,81,67	19008	10.4	Na	Test material
1193	Unknown 57,97,41,115	2272	1.2	Na	Test material
1258	Dimethyl phthalate	498	0.3	Na	Test material
1283	Unknown 57,41,56,83 (Dodecyl oxirane ?)	3643	2.0	Na	Test material
1307	Unknown 220,172,84,238 (Breakdown product from d20-BHT)	870	0.5	Na	
1318	Unknown 57,41,56,83	2049	1.1	Na	Test material
1323	Unknown 90,91,78,164	1787	1.0	Na	Test material
1342	d20-BHT	7934	8.0		IS
1452	d34-Hexadecane	465	0.5		IS
1458	Texanol isobutyrate		NQ		GC artefact
1637	d10-Phenanthrene	2008	2.0		IS
1717	Dibutylphthalate isomer	1781	NQ		In blank
1805	Dibutylphthalate isomer	16113	16.0	Ph	Test material
2014	Phthalate	992	1.0	Ph	Test material
2295	Dioctyl phthalate isomer	1880	NQ		In blank
2357	Unknown 57,43,71,85	622	NQ		In blank

Internal standards used: Bz = d6-benzene, Cl = d5-chlorobenzene, Xy = d10-p-xylene, Po = d5-Phenol, Na = d8-Naphthelene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P211	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	GRP	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	GRP Unchlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
2372	d62-Squalane	8924	8.0		IS
2426	Unknown 57,45,71,85	610	NQ		In blank

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Ph=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene.

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P141	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	GRP	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	GRP B2				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0023	d6-Benzene	347	2.0		IS
0195	Toluene	9354	53.9	Bz	Test material
0338	d5-Chlorobenzene	1433	2.0		IS
0382	m-Xylene	571	0.7	Xy	Test material
0392	d10-p-Xylene	416	0.5		IS
0608	d5-Phenol	2638	8.0		IS
0686	Isophorone	5289	9.8	Na	Test material
0721	Unknown 105,77,51,120	351	0.6	Na	Test material
0917	d8-Naphthalene	271	0.5		IS
0936	Benzoic acid	45803	84.5	Na	Test material
0975	t-Butylcyclohexanone 57,98,41,40	790	1.5	Na	Test material
0998	Unknown 57,81,67,41	26690	49.2	Na	Test material
1193	Unknown 57,97,41,115	3060	5.6	Na	Test material
1286	Unknown 57,41,56,83 (Dodecyl oxirane ?)	9219	17.0	Na	Test material
1321	Unknown 90,57,41,91	10043	18.5	Na	Test material
1343	d20-BHT	10424	8.0		IS
1424	Diethylphthalate isomer	599	NQ		In blank
1452	d34-Hexadecane	1324	0.5		IS
1459	Texanol isobutyrate		NQ		GC artefact
1636	d10-Phenanthrene	2484	2.0		IS
1717	Dibutylphthalate isomer	2083	NQ		In blank
1806	Dibutylphthalate isomer	36753	29.6	Ph	Test material
1982	Unknown 41,55,56,69	942	0.8	Ph	Test material
2015	Phthalate	3775	3.0	Ph	Test material

Internal standards used: Bz = d6-benzene, Cl = d5-chlorobenzene, Xy = d10-p-xylene, Po = d5-Phenol, Na = d8-Naphthelene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P141	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	GRP	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	GRP B2				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
2359	Unknown 57,43,71,85	465	NQ		In blank
2373	d62-Squalane	12974	8.0		IS
2428	Unknown 57,43,71,85	728	0.4	Sq	Test material

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Ph=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene.

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P201	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	GRP	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	GRP Chlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0022	d6-Benzene	303	2.0		IS
0069	Chloromethylbutene		NQ		Chlorination artefact
0194	Toluene	6284	41.5	Bz	Test material
0253	Unknown 59,107,109		NQ		Chlorination artefact
0337	d5-Chlorobenzene	1027	2.0		IS
0382	Xylene isomer	839	1.0	Xy	Test material
0390	d10-p-Xylene	418	0.5		IS
0398	Xylene isomer & Unknown 57,43,41,72	714	0.9	Xy	Test material
0535	Benzaldehyde	23840	22.8	Na	Test material
0607	d5-Phenol	1417	8.0		IS
0686	Isophorone	6045	5.8	Na	Test material
0720	1-Phenylethanone (?)	541	0.5	Na	Test material
0778	Methylbenzoate	916	0.9	Na	Test material
0916	d8-Naphthalene	522	0.5		IS
0969	Benzoic acid	249000	238.5	Na	Test material
1001	Unknown 57,81,67,41	20450	19.6	Na	Test material
1157	Unknown 105,77,148,51 (Phenylbutanone isomer ?)	378	0.4	Na	Test material
1192	Unknown 57,97,41,115	3282	3.1	Na	Test material
1257	Dimethylphthalate	1213	1.2	Na	Test material
1286	Unknown 57,41,56,83 (Dodecyl oxirane ?)	10304	9.9	Na	Test material
1307	Unknown 172,220,188,238 (Breakdown product from d20-BHT)	469	0.4	Na	
1320	Unknown 57,41,90,56	10555	10.1	Na	Test material
1342	d20-BHT	14511	8.0		IS
1363	Unknown 57,43,71,41	602		Ph	Test material

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Po=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P201	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	GRP	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	GRP Chlorinated				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
1424	Diethylphthalate	845	NQ		In blank
1451	d34-Hexadecane	1028	0.5		IS
1458	Texanol Isobutyrate		NQ		GC artefact
1571	Unknown 166,57,151,41	302		Ph	Test material
1636	d10-Phenanthrene	3025	2.0		IS
1716	Dibutylphthalate isomer	2064	NQ		In blank
1805	Dibutylphthalate isomer	37005	24.5	Ph	Test material
1825	Palmitic acid	688	0.5	Ph	Test material ?
1982	Unknown 41,55,69,43	4098	2.7	Ph	Test material
2014	Phthalate	3217	2.1	Ph	Test material
2294	Unknown 149,57,41,55 Phthalate	1231	NQ		In blank
2358	Unknown 57,43,71,85 HC	566	NQ		In blank
2372	d62-Squalane	11766	8.0		IS
2427	Unknown 57,43,71,85 HC	783	NQ		In blank

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Ph=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene.

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P121	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	GRP	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	GRP				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
0023	d6-Benzene	308	2.0		IS
0071	Chloromethylbutene		NQ		Chlorination artefact
0195	Toluene	6019	39.1	Bz	Test material
0254	Unknown 59,107,109		NQ		Chlorination artefact
0338	d5-Chlorobenzene	1068	2.0		IS
0382	d10-p-Xylene	257	0.5		IS
0536	Benzaldehyde	23456	24.2	Na	Test material
0608	d5-Phenol	1537	8.0		IS
0687	Isophorone	5502	5.7	Na	Test material
0721	Unknown 77,105,51,120	746	0.8	Na	Test material
0778	Methylbenzoate	421	0.4	Na	Test material
0916	d8-Naphthalene	485	0.5		IS
0951	Benzoic acid	95700	98.7	Na	Test material
1001	Unknown 57,41,81,67	20150	20.8	Na	Test material
1193	Unknown 57,97,41,115	2726	2.8	Na	Test material
1285	Unknown 57,41,56,83 (Dodecyl oxirane ?)	4356	4.5	Na	Test material
1323	Unknown 90,78,91,164	4167	4.3	Na	Test material
1343	d20-BHT	0	0.0		IS
1365	Unknown 57,43,71,85	1588	0.9	Ph	Test material
1452	d34-Hexadecane	768	0.5		IS
1459	Texanol isobutyrate		NQ		GC artefact
1637	d10-Phenanthrene	3533	2.0		IS
1718	Dibutylphthalate isomer	1307	NQ		In blank
1807	Dibutylphthalate isomer	22664	12.8	Ph	Test material

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Po=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene

GC-MS General Survey Results

Project No:	8782-0	**Form No:**	1307	**Date Sampled:**	Unknown
Analyst:	RK	**Data System Code:**	0780P121	**Date Received:**	09-Nov-94
Sample Volume:	1 litre	**Sample Code :**	GRP	**Date Analysed:**	15-Nov-94
Final Extract Vol:	500 ul	**Associated Blank:**	Borehole		
Sample Type:	GRP				

Scan	Compound	Peak Area	Conc. (ug/l)	Internal Standard	Origin of Peak
1984	Unknown 55,41,69,83	3199	NQ		In blank
2005	Stearic acid	2961	1.7	Ph	Test material ?
2015	Unknown 149,104,76,193	1681	1.0	Ph	Test material
2047	Unknown 70,55,83,69	562	0.3	Ph	Test material
2297	Dioctylphthalate isomer	2904	NQ		In blank
2372	d62-Squalene	7974	8.0		IS

Internal standards used: Bz=d6-benzene, Cl=d5-chlorobenzene, Xy=d10-p-xylene, Ph=d5-Phenol, Na=d8-Naphthelene, BHT=d20-BHT, Hx=d34-hexadecane, Ph=d10-phenanthrene and Sq=d62-Squalene.

APPENDIX D

DATA TABLES (INTERNAL STANDARDS) FOR GCMS RUNS UNDERTAKEN FOR INTERLABORATORY PERFORMANCE TESTING OF PROTOCOLS

GC-MS General Survey AQC results

Peak areas of internal standards detected in leachates

Laboratory WRc - Batch 1

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys	Peak Area								
	Code	d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (1)	0030S021	7211	8522	2981	9844	2739	56153	2768	14583	90672
PE-TW-CL (1)	0030S031	5588	10633	2815	10635	3700	56423	4668	19328	100599
GRP-TW (1)	0030S041	5754	9923	3685	13097	2753	61581	3160	16134	99354
GRP-TW-CL (1)	0030S051	8994	10401	3820	14973	3369	57335	4567	21121	101328
BIT-TW (1)	0030S061	5603	6327	1318	7156	2302	48171	3638	16048	85730
BIT-TW-CL (1)	0030S071	6916	11074	2969	9627	4402	57669	4469	16901	54348
PB-TW (1)	0030S091	12190	13005	2887	10916	4029	65931	4728	15546	102366
PB-TW-CL (1)	0030S101	9215	10259	2671	8351	3744	48374	4523	14240	78070
Mean		**7684**	**10018**	**2893**	**10575**	**3380**	**56455**	**4065**	**16738**	**89058**
SD		**2318**	**1945**	**759**	**2504**	**724**	**6004**	**766**	**2363**	**16501**
%RSD		**30%**	**19%**	**26%**	**24%**	**21%**	**11%**	**19%**	**14%**	**19%**

Column test Results:

CCM Coltest	0030S011	16577	16584	4166	36049	3324	64448	7760	18748	82567
CCM Coltest	0030S081	16811	14743	3509	43139	3866	63526	8429	16263	80833
CCM Coltest	0030S151	17261	17059	4202	39062	3002	69540	6221	18546	74540
CCM Coltest	0030S221	18417	14874	4481	32013	3260	63360	5920	15776	87494
Mean		**17267**	**15815**	**4090**	**37566**	**3363**	**65219**	**7083**	**17333**	**76359**
SD		**818**	**1179**	**412**	**4706**	**363**	**2920**	**1206**	**1532**	**6842**
%RSD		**5%**	**7%**	**10%**	**13%**	**11%**	**4%**	**17%**	**9%**	**9%**

GC-MS General Survey AQC results

Peak area ratios (normalised to d62-squalane)

Laboratory WRc - Batch 1

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Peak Area ratios								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (1)	0030S021	0.0795	0.0940	0.0329	0.1086	0.0302	0.6193	0.0305	0.1608	1.0000
PE-TW-CL (1)	0030S031	0.0555	0.1057	0.0280	0.1057	0.0368	0.5609	0.0464	0.1921	1.0000
GRP-TW (1)	0030S041	0.0579	0.0999	0.0371	0.1318	0.0277	0.6198	0.0318	0.1624	1.0000
GRP-TW-CL (1)	0030S051	0.0888	0.1026	0.0377	0.1478	0.0332	0.5658	0.0451	0.2084	1.0000
BIT-TW (1)	0030S061	0.0654	0.0738	0.0154	0.0835	0.0269	0.5619	0.0424	0.1872	1.0000
BIT-TW-CL (1)	0030S071	0.1273	0.2038	0.0546	0.1771	0.0810	1.0611	0.0822	0.3110	1.0000
PB-TW (1)	0030S091	0.1191	0.1270	0.0282	0.1066	0.0394	0.6441	0.0462	0.1519	1.0000
PB-TW-CL (1)	0030S101	0.1180	0.1314	0.0342	0.1070	0.0480	0.6196	0.0579	0.1824	1.0000
Mean		**0.0889**	**0.1173**	**0.0335**	**0.1210**	**0.0404**	**0.6566**	**0.0478**	**0.1945**	**1.0000**
SD		**0.0291**	**0.0394**	**0.0111**	**0.0297**	**0.0178**	**0.1666**	**0.0164**	**0.0506**	**0.0000**
%RSD		33%	34%	33%	25%	44%	25%	34%	26%	0%

Column test Results:

CCM Coltest	0030S011	0.2008	0.2009	0.0505	0.4366	0.0403	0.7806	0.0940	0.2271	1.0000
CCM Coltest	0030S081	0.2080	0.1824	0.0434	0.5337	0.0478	0.7859	0.1043	0.2012	1.0000
CCM Coltest	0030S151	0.2316	0.2289	0.0564	0.5240	0.0403	0.9329	0.0835	0.2488	1.0000
CCM Coltest	0030S221	0.2729	0.2204	0.0664	0.4743	0.0483	0.9388	0.0877	0.2337	1.0000
Mean		**0.2283**	**0.2081**	**0.0542**	**0.4922**	**0.0442**	**0.8595**	**0.0924**	**0.2277**	**1.0000**
SD		**0.0325**	**0.0208**	**0.0097**	**0.0453**	**0.0045**	**0.0882**	**0.0090**	**0.0199**	**0.0000**
%RSD		14%	10%	18%	9%	10%	10%	10%	9%	0%

GC-MS General Survey AQC results

Retention times of internal standards

Laboratory WRc - Batch 1

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Retention time (minutes)								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalene
PE-TW (1)	0030S021	3.50	7.70	8.44	11.44	15.61	21.53	23.05	25.51	35.72
PE-TW-CL (1)	0030S031	3.51	7.72	8.47	11.45	15.63	21.53	23.05	25.51	35.72
GRP-TW (1)	0030S041	3.50	7.72	8.45	11.45	15.61	21.53	23.03	25.51	35.71
GRP-TW-CL (1)	0030S051	3.48	7.72	8.44	11.45	15.63	21.53	23.03	25.51	35.72
BIT-TW (1)	0030S061	3.48	7.72	8.44	11.45	15.63	21.52	23.03	25.51	35.71
BIT-TW-CL (1)	0030S071	3.48	7.69	8.42	11.44	15.60	21.50	23.02	25.48	35.69
PB-TW (1)	0030S091	3.48	7.69	8.41	11.42	15.60	21.50	23.00	25.46	35.68
PB-TW-CL (1)	0030S101	3.47	7.69	8.42	11,42	15.58	21.50	23.02	25.48	35.69
Mean		3.49	7.71	8.44	11.44	15.61	21.52	23.03	25.50	35.71
SD		0.014	0.015	0.019	0.011	0.018	0.015	0.016	0.020	0.016
%RSD		0.40%	0.20%	0.23%	0.10%	0.12%	0.07%	0.07%	0.08%	0.04%

Column test Results:

CCM Coltest	0030S011	3.45	7.69	8.42	11.44	q5.63	21.55	23.06	25.53	35.74
CCM Coltest	0030S081	3.48	7.69	8.42	11.42	15.60	21.50	23.02	25.49	35.69
CCM Coltest	0030S151	3.45	7.66	8.39	11.39	15.58	21.49	23.00	25.48	35.66
CCM Coltest	0030S221	3.47	7.67	8.41	11.39	15.58	21.49	23.00	25.46	35.68
Mean		3.46	7.68	8.41	11.41	15.59	21.51	23.02	25.50	35.69
SD		0.015	0.015	0.014	0.024	0.012	0.029	0.028	0.029	0.034
%RSD		0.43%	0.20%	0.17%	0.21%	0.07%	0.13%	0.12%	0.12%	0.10%

GC-MS General Survey AQC results

Relative retention times of internal standards (normalised to d62-squalane)

Laboratory WRc - Batch 1

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Relative Retention times								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (1)	0030S021	0.0980	0.2156	0.2363	0.3202	0.4370	0.6027	0.6453	0.7142	1.0000
PE-TW-CL (1)	0030S031	0.0983	0.2161	0.2371	0.3207	0.4376	0.6027	0.6453	0.7142	1.0000
GRP-TW (1)	0030S041	0.0980	0.2162	0.2366	0.3208	0.4371	0.6029	0.6449	0.7144	1.0000
GRP-TW-CL (1)	0030S051	0.0974	0.2161	0.2363	0.3207	0.4376	0.6027	0.6447	0.7142	1.0000
BIT-TW (1)	0030S061	0.0975	0.2162	0.2363	0.3208	0.4377	0.6026	0.6449	0.7144	1.0000
BIT-TW-CL (1)	0030S071	0.0975	0.2155	0.2359	0.3205	0.4371	0.6024	0.6450	0.7139	1.0000
PB-TW (1)	0030S091	0.0975	0.2155	0.2357	0.3202	0.4372	0.6026	0.6446	0.7136	1.0000
PB-TW-CL (1)	0030S101	0.0972	0.2155	0.2359	0.3201	0.4365	0.6024	0.6450	0.7139	1.0000
Mean		**0.0977**	**0.2158**	**0.2363**	**0.3205**	**0.4372**	**0.6026**	**0.6450**	**0.7141**	**1.0000**
SD		**0.0004**	**0.0003**	**0.0005**	**0.0003**	**0.0004**	**0.0002**	**0.0002**	**0.0003**	**0.0000**
%RSD		**0.37%**	**0.16%**	**0.19%**	**0.09%**	**0.09%**	**0.03%**	**0.04%**	**0.04%**	**0.00%**

Column test Results:

CCM Coltest	0030S011	0.0965	0.2152	0.2356	0.3201	0.4373	0.6030	0.6452	0.7143	1.0000
CCM Coltest	0030S081	0.0975	0.2155	0.2359	0.3200	0.4371	0.6024	0.6450	0.7142	1.0000
CCM Coltest	0030S151	0.0967	0.2148	0.2353	0.3194	0.4369	0.6026	0.6450	0.7145	1.0000
CCM Coltest	0030S221	0.0973	0.2150	0.2357	0.3192	0.4367	0.6023	0.6446	0.7136	1.0000
Mean		**0.0970**	**0.2151**	**0.2356**	**0.3197**	**0.4370**	**0.6026**	**0.6450**	**0.7142**	**1.0000**
SD		**0.0004**	**0.0003**	**0.0003**	**0.0004**	**0.0003**	**0.0003**	**0.0002**	**0.0004**	**0.0000**
%RSD		**0.46%**	**0.13%**	**0.11%**	**0.13%**	**0.06%**	**0.05%**	**0.04%**	**0.06%**	**0.00%**

GC-MS General Survey AQC results

Peak areas of internal standards detected in leachates

Laboratory WRc - Batch 2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Peak Area								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (2)	0030S121	6498	9329	3311	7335	3868	53707	3531	14276	85766
PE-TW-CL (2)	0030S131	4665	9183	3188	6197	2929	62658	4435	17728	113155
GRP-TW (2)	0030S141	5766	7986	2087	12357	2531	50362	5212	16965	87439
GRP-TW-CL (2)	0030S161	5410	11762	3473	13804	2375	61799	6283	20480	100379
BIT-TW (2)	0030S171	4714	6008	4194	9691	2534	39768	2037	13363	36799
BIT-TW-CL (2)	0030S181	5702	9184	3345	14796	2609	47853	3479	15293	54239
PB-TW (2)	0030S191	7207	11073	3163	6846	2765	49139	4875	14024	88095
PB-TW-CL (2)	0030S201	6444	7862	2702	7097	2175	54553	6013	15490	85893
Mean		**5801**	**9048**	**3183**	**9765**	**2723**	**52480**	**4483**	**15952**	**81471**
SD		**887**	**1827**	**607**	**3436**	**516**	**7511**	**1423**	**2347**	**24542**
%RSD		**15%**	**20%**	**19%**	**35%**	**19%**	**14%**	**32%**	**15%**	**30%**

Column test Results:

CCM Coltest	0030S011	16577	16584	4166	36049	3324	64448	7760	18748	82567
CCM Coltest	0030S081	16811	14743	3509	43139	3866	63526	8429	16263	80833
CCM Coltest	0030S151	17261	17059	4202	39062	3002	69540	6221	18546	74540
CCM Coltest	0030S221	18417	14874	4481	32013	3260	63360	5920	15776	67494
Mean		**17267**	**15815**	**4090**	**37566**	**3363**	**65219**	**7083**	**17333**	**76359**
SD		**818**	**1179**	**412**	**4706**	**363**	**2920**	**1206**	**1532**	**6842**
%RSD		**5%**	**7%**	**10%**	**13%**	**11%**	**4%**	**17%**	**9%**	**9%**

GC-MS General Survey AQC results

Peak area ratios (normalised to d62-squalane)

Laboratory WRc - Batch 2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Peak Area ratios								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (2)	0030S121	0.0758	0.1088	0.0386	0.0855	0.0451	0.6262	0.0412	0.1665	1.0000
PE-TW-CL (2)	0030S131	0.0412	0.0812	0.0282	0.0548	0.0259	0.5537	0.0392	0.1567	1.0000
GRP-TW (2)	0030S141	0.0659	0.0913	0.0239	0.1413	0.0289	0.5760	0.0596	0.1940	1.0000
GRP-TW-CL (2)	0030S161	0.0539	0.1172	0.0346	0.1375	0.0237	0.6157	0.0626	0.2040	1.0000
BIT-TW (2)	0030S171	0.1281	0.1633	0.1140	0.2633	0.0689	1.0807	0.0554	0.3631	1.0000
BIT-TW-CL (2)	0030S181	0.1051	0.1693	0.0617	0.2728	0.0481	0.8823	0.0641	0.2820	1.0000
PB-TW (2)	0030S191	0.0818	0.1257	0.0359	0.0777	0.0314	0.5578	0.0553	0.1592	1.0000
PB-TW-CL (2)	0030S201	0.0750	0.0915	0.0315	0.0826	0.0253	0.6351	0.0700	0.1803	1.0000
Mean		**0.0784**	**0.1185**	**0.0460**	**0.1395**	**0.0372**	**0.6909**	**0.0559**	**0.2132**	**1.0000**
SD		**0.0277**	**0.0329**	**0.0297**	**0.0847**	**0.0158**	**0.1895**	**0.0108**	**0.0727**	**0.0000**
%RSD		**35%**	**28%**	**65%**	**61%**	**42%**	**27%**	**19%**	**34%**	**0%**

Column test Results:

CCM Coltest	0030S011	0.2008	0.2009	0.0505	0.4366	0.0403	0.7806	0.0940	0.2271	1.0000
CCM Coltest	0030S081	0.2080	0.1824	0.0434	0.5337	0.0478	0.7859	0.1043	0.2012	1.0000
CCM Coltest	0030S151	0.2316	0.2289	0.0564	0.5240	0.0403	0.9329	0.0835	0.2488	1.0000
CCM Coltest	0030S221	0.2729	0.2204	0.0664	0.4743	0.0483	0.9388	0.0877	0.2337	1.0000
Mean		**0.2283**	**0.2081**	**0.0542**	**0.4922**	**0.0442**	**0.8595**	**0.0924**	**0.2277**	**1.0000**
SD		**0.0325**	**0.0208**	**0.0097**	**0.0453**	**0.0045**	**0.0882**	**0.0090**	**0.0199**	**0.0000**
%RSD		**14%**	**10%**	**18%**	**9%**	**10%**	**10%**	**10%**	**9%**	**0%**

GC-MS General Survey AQC results

Retention times of internal standards

Laboratory WRc - Batch 2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Retention time (minutes)								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (2)	0030S121	3.47	7.67	8.39	11.42	15.57	21.49	22.99	25.46	35.66
PE-TW-CL (2)	0030S131	3.47	7.67	8.41	11.44	15.58	21.49	23.00	25.46	35.68
GRP-TW (2)	0030S141	3.47	7.67	8.42	11.42	15.58	21.49	22.99	25.46	35.68
GRP-TW-CL (2)	0030S161	3.47	7.67	8.42	11.42	15.60	21.49	23.00	25.46	35.68
BIT-TW (2)	0030S171	3.45	7.66	8.39	11.42	15.57	21.47	22.99	25.46	35.66
BIT-TW-CL (2)	0030S181	3.45	7.66	8.39	11.41	15.57	21.47	22.99	25.45	35.66
PB-TW (2)	0030S191	3.45	7.66	8.39	11.39	15.55	21.47	22.99	25.45	35.66
PB-TW-CL (2)	0030S201	3.47	7.67	8.41	11.41	15.58	21.49	23.00	25.46	35.68
Mean		**3.46**	**7.67**	**8.40**	**11.42**	**15.58**	**21.48**	**22.99**	**25.46**	**35.67**
SD		**0.010**	**0.005**	**0.014**	**0.014**	**0.014**	**0.010**	**0.005**	**0.005**	**0.011**
%RSD		**0.30%**	**0.07%**	**0.17%**	**0.12%**	**0.09%**	**0.05%**	**0.02%**	**0.02%**	**0.03%**

Column test Results:

CCM Coltest	0030S011	3.45	7.69	8.42	11.44	15.63	21.55	23.06	25.53	35.74
CCM Coltest	0030S081	3.48	7.69	8.42	11.42	15.60	21.50	23.02	25.49	35.69
CCM Coltest	0030S151	3.45	7.66	8.39	11.39	15.58	21.49	23.00	25.48	35.66
CCM Coltest	0030S221	3.47	7.67	8.41	11.39	15.58	21.49	23.00	25.46	35.68
Mean		**3.46**	**7.68**	**8.41**	**11.41**	**15.60**	**21.51**	**23.02**	**25.49**	**35.69**
SD		**0.015**	**0.015**	**0.014**	**0.024**	**0.024**	**0.029**	**0.028**	**0.029**	**0.034**
%RSD		**0.43%**	**0.20%**	**0.17%**	**0.21%**	**0.15%**	**0.13%**	**0.12%**	**0.12%**	**0.10%**

GC-MS General Survey AQC results

Relative retention times of internal standards (normalised to d62-squalane)

Laboratory WRc - Batch 2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Relative Retention times								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW (2)	0030S121	0.0973	0.2151	0.2353	0.3202	0.4366	0.6026	0.6447	0.7140	1.0000
PE-TW-CL (2)	0030S131	0.0973	0.2150	0.2357	0.3206	0.4367	0.6023	0.6446	0.7136	1.0000
GRP-TW (2)	0030S141	0.0973	0.2150	0.2360	0.3201	0.4367	0.6023	0.6443	0.7136	1.0000
GRP-TW-CL (2)	0030S161	0.0973	0.2150	0.2360	0.3201	0.4372	0.6023	0.6446	0.7136	1.0000
BIT-TW (2)	0030S171	0.0967	0.2148	0.2353	0.3200	0.4366	0.6021	0.6447	0.7137	1.0000
BIT-TW-CL (2)	0030S181	0.0967	0.2148	0.2353	0.3194	0.4361	0.6021	0.6447	0.7137	1.0000
PB-TW (2)	0030S191	0.0967	0.2148	0.2353	0.3194	0.4361	0.6021	0.6447	0.7137	1.0000
PB-TW-CL (2)	0030S201	0.0973	0.2150	0.2357	0.3198	0.4367	0.6023	0.6446	0.7136	1.0000
Mean		**0.0971**	**0.2149**	**0.2356**	**0.3199**	**0.4366**	**0.6023**	**0.6446**	**0.7137**	**1.0000**
SD		**0.0003**	**0.0001**	**0.0003**	**0.0004**	**0.0004**	**0.0002**	**0.0001**	**0.0001**	**0.0000**
%RSD		**0.28%**	**0.05%**	**0.14%**	**0.13%**	**0.09%**	**0.03%**	**0.02%**	**0.02%**	**0.00%**

Column test Results:

CCM Coltest	0030S011	0.0965	0.2152	0.2356	0.3201	0.4373	0.6030	0.6452	0.7143	1.0000
CCM Coltest	0030S081	0.0975	0.2155	0.2359	0.3200	0.4371	0.6024	0.6450	0.7142	1.0000
CCM Coltest	0030S151	0.0967	0.2148	0.2353	0.3194	0.4369	0.6026	0.6450	0.7145	1.0000
CCM Coltest	0030S221	0.0973	0.2150	0.2357	0.3192	0.4367	0.6023	0.6446	0.7136	1.0000
Mean		**0.0970**	**0.2151**	**0.2356**	**0.3197**	**0.4370**	**0.6026**	**0.6450**	**0.7142**	**1.0000**
SD		**0.0004**	**0.0003**	**0.0003**	**0.0004**	**0.0003**	**0.0003**	**0.0002**	**0.0004**	**0.0000**
%RSD		**0.46%**	**0.13%**	**0.11%**	**0.13%**	**0.06%**	**0.05%**	**0.04%**	**0.06%**	**0.00%**

GC-MS General Survey AQC results

Peak areas of internal standards detected in leachates

Laboratory WQC- Batch1

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Peak Area								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW(1)	C38903	510725	955198	283713	2105341	481045	9045261	606358	2219511	7247330
PE-TW-CI(1)	C38904	510121	885862	542940	1482416	423138	6363865	371529	1923732	6266574
GRP-TW(1)	C38905	488602	752021	244095	2098301		5788530	669675	2457007	12374654
GRP-TW-CL(1)	C38906	431959	668223	236576	2131447		5273854	345287	2014393	12172250
BIT-TW(1)	C38907	440723	808938	279793	1302116	474738	5712972	71733	1832596	9991398
BIT-TW-CI(1)	C38908	505832	733796	224201	1259306	387752	4766984	232142	1535164	6381086
BL-TW(1)	C38909	519492	873034	288267	1532059	378900	5352015	420284	1722400	9025045
BL-TW-CI(1)	C38910	296974	610123	219180	1252827	282152	4859589	280807	1745045	9503304
Mean		463054	785899	289846	1645477	404621	5895384	374702	1931231	9120205
SD		74837	116733	105851	398809	73545	1373920	194420	295536	2389706
%RSD		16%	15%	37%	24%	18%	23%	52%	15%	26%

Column test Results:

CCM Coltest	0101001	2635176	1512171	455618	4618337	628743	8024491	478131	4246508	9341948
CCM Coltest	0101007	2527226	1442073	458999	3926930	572867	6981229	535613	2116400	9426020
CCM Coltest	0101012	2749426	1519202	466173	3811608	536442	7465715	575570	2535597	12930676
Mean		2637276	1491149	460263	4118958	579351	7490478	529771	2966168	10566215
SD		111115	42646	5390	436302	46491	522072	48981	1128443	2048115
%RSD		4%	3%	1%	11%	8%	7%	9%	38%	19%

GC-MS General Survey AQC results

Peak area ratios (normalised to d62-squalane)

Laboratory WQC- Batch1

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Peak Area ratios								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW(1)	C38903	0.0705	0.1318	0.0391	0.2905	0.0664	1.2481	0.0837	0.3063	1.0000
PE-TW-Cl(1)	C38904	0.0814	0.1414	0.0866	0.2366	0.0675	1.0155	0.0593	0.3070	1.0000
GRP-TW(1)	C38905	0.0395	0.0608	0.0197	0.1696		0.4678	0.0541	0.1986	1.0000
GRP-TW-CL(1)	C38906	0.0355	0.0549	0.0194	0.1751		0.4333	0.0284	0.1655	1.0000
BIT-TW(1)	C38907	0.0441	0.0810	0.0280	0.1303	0.0475	0.5718	0.0072	0.1834	1.0000
BIT-TW-Cl(1)	C38908	0.0793	0.1150	0.0351	0.1973	0.0608	0.7470	0.0364	0.2406	1.0000
BL-TW(1)	C38909	0.0576	0.0967	0.0319	0.1698	0.0420	0.5930	0.0466	0.1908	1.0000
BL-TW-Cl(1)	C38910	0.0312	0.0642	0.0231	0.1318	0.0297	0.5114	0.0295	0.1836	1.0000
Mean		**0.0549**	**0.0932**	**0.0354**	**0.1876**	**0.0523**	**0.6985**	**0.0431**	**0.2220**	**1.0000**
SD		**0.0201**	**0.0334**	**0.0219**	**0.0538**	**0.0151**	**0.2905**	**0.0232**	**0.0565**	**0.0000**
%RSD		37%	36%	62%	29%	29%	42%	54%	25%	0%

Column test Results:

CCM Coltest	O101001	0.2821	0.1619	0.0488	0.4944	0.0673	0.8590	0.0512	0.4546	1.0000
CCM Coltest	O101007	0.2681	0.1530	0.0487	0.4166	0.0608	0.7406	0.0568	0.2245	1.0000
CCM Coltest	O101012	0.2126	0.1175	0.0361	0.2948	0.0415	0.5774	0.0445	0.1961	1.0000
Mean		**0.2543**	**0.1441**	**0.0445**	**0.4019**	**0.0565**	**0.7257**	**0.0508**	**0.2917**	**1.0000**
SD		**0.0367**	**0.0235**	**0.0073**	**0.1006**	**0.0134**	**0.1414**	**0.0062**	**0.1417**	**0.0000**
%RSD		14%	16%	16%	25%	24%	19%	12%	49%	0%

GC-MS General Survey AQC results

Retention times of internal standards

Laboratory WQC- Batch1

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Retention time (minutes)								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW(1)	C38903	8.4	16	16.8	19.49	23.24	27.63	28.72	30.87	41.6
PE-TW-CI(1)	C38904	8.37	15.97	16.78	19.5	23.22	27.65	28.72	30.86	41.57
GRP-TW(1)	C38905	8.4	15.97	16.8	19.5		27.56	28.71	30.86	41.56
GRP-TW-CL(1)	C38906	8.35	15.95	16.79	19.49		27.61	28.7	30.84	41.55
BIT-TW(1)	C38907	8.37	15.97	16.77	19.5	23.19	27.63	28.61	30.84	41.51
BIT-TW-CI(1)	C38908	8.31	15.94	16.76	19.49	23.2	27.64	28.6	30.84	41.51
BL-TW(1)	C38909	8.34	15.94	16.75	19.48	23.19	27.62	28.6	30.83	41.22
BL-TW-CI(1)	C38910	8.33	15.91	16.75	19.49	23.2	27.65	28.72	30.86	41.22
Mean		**8.36**	**15.96**	**16.78**	**19.49**	**23.21**	**27.62**	**28.67**	**30.85**	**41.47**
SD		**0.032**	**0.027**	**0.021**	**0.007**	**0.020**	**0.029**	**0.058**	**0.014**	**0.156**
%RSD		**0.39%**	**0.17%**	**0.12%**	**0.04%**	**0.08%**	**0.11%**	**0.20%**	**0.05%**	**0.38%**

Column test Results:

CCM Coltest	O101001	8.46	16.01	16.84	19.54	23.25	27.67	28.75	30.87	41.62
CCM Coltest	O101007	8.28	15.94	16.73	19.47	23.21	27.64	28.69	30.84	41.54
CCM Coltest	O101012	8.34	15.94	16.75	19.48	23.19	27.62	28.20	30.83	41.52
Mean		**8.36**	**15.96**	**16.77**	**19.50**	**23.22**	**27.64**	**28.55**	**30.85**	**41.56**
SD		**0.092**	**0.040**	**0.059**	**0.038**	**0.031**	**0.025**	**0.302**	**0.021**	**0.053**
%RSD		**1.10%**	**0.25%**	**0.35%**	**0.19%**	**0.13%**	**0.09%**	**1.06%**	**0.07%**	**0.13%**

GC-MS General Survey AQC results

Relative retention times of internal standards (normalised to d62-squalane)

Project No: WQC- Batch1

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Relative Retention times								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW(1)	C38903	0.2019	0.3846	0.4038	0.4685	0.5587	0.6642	0.6904	0.7421	1.0000
PE-TW-CI(1)	C38904	0.2013	0.3842	0.4037	0.4691	0.5586	0.6651	0.6909	0.7424	1.0000
GRP-TW(1)	C38905	0.2021	0.3843	0.4042	0.4692		0.6631	0.6908	0.7425	1.0000
GRP-TW-CL(1)	C38906	0.2010	0.3839	0.4041	0.4691		0.6645	0.6907	0.7422	1.0000
BIT-TW(1)	C38907	0.2016	0.3847	0.4040	0.4698	0.5587	0.6656	0.6892	0.7430	1.0000
BIT-TW-CI(1)	C38908	0.2002	0.3840	0.4038	0.4695	0.5589	0.6659	0.6890	0.7430	1.0000
BL-TW(1)	C38909	0.2023	0.3867	0.4064	0.4726	0.5626	0.6701	0.6938	0.7479	1.0000
BL-TW-CI(1)	C38910	0.2021	0.3860	0.4064	0.4728	0.5628	0.6708	0.6967	0.7487	1.0000
Mean		**0.2016**	**0.3848**	**0.4045**	**0.4701**	**0.5600**	**0.6662**	**0.6915**	**0.7440**	**1.0000**
SD		**0.0007**	**0.0010**	**0.0011**	**0.0017**	**0.0021**	**0.0028**	**0.0026**	**0.0027**	**0.0000**
%RSD		**0.36%**	**0.26%**	**0.28%**	**0.35%**	**0.37%**	**0.42%**	**0.38%**	**0.36%**	**0.00%**

Column test Results:

CCM Coltest	O101001	0.2033	0.3847	0.4046	0.4695	0.5586	0.6648	0.6908	0.7417	1.0000
CCM Coltest	O101007	0.1993	0.3837	0.4027	0.4687	0.5587	0.6654	0.6907	0.7424	1.0000
CCM Coltest	O101012	0.2009	0.3839	0.4034	0.4692	0.5585	0.6652	0.6792	0.7425	1.0000
Mean		**0.2012**	**0.3841**	**0.4036**	**0.4691**	**0.5586**	**0.6651**	**0.6869**	**0.7422**	**1.0000**
SD		**0.0020**	**0.0005**	**0.0009**	**0.0004**	**0.0001**	**0.0003**	**0.0067**	**0.0004**	**0.0000**
%RSD		**0.99%**	**0.13%**	**0.23%**	**0.08%**	**0.02%**	**0.04%**	**0.97%**	**0.06%**	**0.00%**

GC-MS General Survey AQC results

Peak areas of internal standards detected in leachates

Laboratory WQC - Batch 2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Peak Area								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW(2)	C39490	347486	758673	230241	935535	255894	3774193	322742	1131263	6184212
PE-TW-Cl(2)	C39494	322485	497705	144016	652910	252412	2914795	302697	955487	5113840
GRP-TW(2)	C39489	289764	584351	145449	1133871		3084475	304904	1249908	4620742
GRP-TW-Cl(2)	C39493	452698	651052	233306	1892358		3842178	335491	1239527	6003063
BIT-TW(2)	C39488	267939	548909	114490	1308954	337359	2963802	188086	1375920	3283917
BIT-TW-Cl(1)	C39492	263290	503971	140201	1221107	526488	3229347	259376	1234639	3689198
BL-TW(2)	C39487	224657	456599	135499	792517	206095	2618607	246956	922097	4816386
BL-TW-Cl(2)	C39491	229928	458272	157010	157327	196256	2441110	196177	766377	4967272
Mean		**299781**	**557442**	**162527**	**1011822**	**295751**	**3108563**	**269564**	**1109402**	**4834829**
SD		**74779**	**104530**	**44395**	**512030**	**123592**	**498637**	**56265**	**207126**	**1003207**
%RSD		**25%**	**19%**	**27%**	**51%**	**42%**	**16%**	**21%**	**19%**	**21%**

Column test Results:

CCM Coltest	0101001	173777	652002	191123	2164824	298640	3849794	277005	1058679	4946411
CCM Coltest	0101007	357848	1025981	300614	2854498	336890	4899779	522100	1404724	7546491
CCM Coltest	0101012	355170	1019405	361957	1975226	420243	5519653	559453	1545770	7936734
Mean		**295598**	**899129**	**284565**	**2331516**	**351924**	**4756409**	**452853**	**1336391**	**6809879**
SD		**105509**	**214044**	**86540**	**462730**	**62180**	**844111**	**153430**	**250632**	**1625563**
%RSD		**36%**	**24%**	**30%**	**20%**	**18%**	**18%**	**34%**	**19%**	**24%**

GC-MS General Survey AQC results

Peak area ratios (normalised to d62-squalane)

Project No: WQC - Batch 2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Peak Area ratios								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW(2)	C39490	0.0562	0.1227	0.0372	0.1513	0.0414	0.6103	0.0522	0.1829	1.0000
PE-TW-Cl(2)	C39494	0.0631	0.0973	0.0282	0.1277	0.0494	0.5700	0.0592	0.1868	1.0000
GRP-TW(2)	C39489	0.0627	0.1265	0.0315	0.2454		0.6675	0.0660	0.2705	1.0000
GRP-TW-Cl(2)	C39493	0.0754	0.1085	0.0389	0.3152		0.6400	0.0559	0.2065	1.0000
BIT-TW(2)	C39488	0.0816	0.1672	0.0349	0.3986	0.1027	0.9025	0.0573	0.4190	1.0000
BIT-TW-Cl(1)	C39492	0.0714	0.1366	0.0380	0.3310	0.1427	0.8754	0.0703	0.3347	1.0000
BL-TW(2)	C39487	0.0466	0.0948	0.0281	0.1645	0.0428	0.5437	0.0513	0.1914	1.0000
BL-TW-Cl(2)	C39491	0.0463	0.0923	0.0316	0.0317	0.0395	0.4914	0.0395	0.1543	1.0000
Mean		**0.0629**	**0.1182**	**0.0335**	**0.2207**	**0.0697**	**0.6626**	**0.0565**	**0.2433**	**1.0000**
SD		**0.0129**	**0.0256**	**0.0043**	**0.1229**	**0.0431**	**0.1503**	**0.0094**	**0.0916**	**0.0000**
%RSD		**20%**	**22%**	**13%**	**56%**	**62%**	**23%**	**17%**	**38%**	**0%**

Column test Results:

CCM Coltest	O101001	0.0351	0.1318	0.0386	0.4377	0.0604	0.7783	0.0560	0.2140	1.0000
CCM Coltest	O101007	0.0474	0.1360	0.0398	0.3783	0.0446	0.6493	0.0692	0.1861	1.0000
CCM Coltest	O101012	0.0448	0.1284	0.0456	0.2489	0.0529	0.6955	0.0705	0.1948	1.0000
Mean		**0.0424**	**0.1321**	**0.0414**	**0.3549**	**0.0527**	**0.7077**	**0.0652**	**0.1983**	**1.0000**
SD		**0.0065**	**0.0038**	**0.0037**	**0.0965**	**0.0079**	**0.0654**	**0.0080**	**0.0143**	**0.0000**
%RSD		**15%**	**3%**	**9%**	**27%**	**15%**	**9%**	**12%**	**7%**	**0%**

GC-MS General Survey AQC results

Retention times of internal standards

Laboratory WQC - Batch 2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Retention time (minutes)								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW(2)	C39490	8.33	15.93	16.77	19.49	23.20	27.60	28.63	30.81	41.43
PE-TW-Cl(2)	C39494	8.30	15.94	16.76	19.49	23.18	27.59	28.66	30.80	41.43
GRP-TW(2)	C39489	8.33	15.93	16.77	19.50		27.62	28.67	30.82	41.47
GRP-TW-Cl(2)	C39493	8.31	15.94	16.75	19.51		27.61	28.67	30.82	41.44
BIT-TW(2)	C39488	8.36	15.96	16.77	19.52	23.21	27.61	28.67	30.82	41.47
BIT-TW-Cl(1)	C39492	8.32	15.95	16.77	19.52	23.20	27.61	28.68	30.83	41.47
BL-TW(2)	C39487	8.35	15.96	16.77	19.49	23.20	27.60	28.68	30.83	41.46
BL-TW-Cl(2)	C39491	8.33	15.93	16.77	19.49	23.17	27.60	28.68	30.80	41.44
Mean		8.33	15.94	16.77	19.50	23.19	27.61	28.67	30.82	41.45
SD		0.020	0.013	0.007	0.014	0.015	0.009	0.017	0.012	0.018
%RSD		0.24%	0.08%	0.04%	0.07%	0.06%	0.03%	0.06%	0.04%	0.04%

Column test Results:

CCM Coltest	O101001	8.36	15.96	16.77	19.49	23.20	27.62	28.70	30.82	41.48
CCM Coltest	O101007	8.39	15.96	16.77	19.49	23.16	27.60	28.67	30.82	41.44
CCM Coltest	O101012	8.36	15.92	16.76	19.48	23.17	27.60	28.64	30.80	41.44
Mean		8.37	15.95	16.77	19.49	23.17	27.61	28.67	30.81	41.45
SD		0.017	0.023	0.006	0.006	0.021	0.012	0.030	0.012	0.023
%RSD		0.21%	0.14%	0.03%	0.03%	0.09%	0.04%	0.10%	0.04%	0.06%

GC-MS General Survey AQC results

Relative retention times of internal standards (normalised to d62-squalane)

Laboratory WQC - Batch 2

Sample Volume: 1 litre
Final Extract Vol: 500ul
Sample Type: Lab test leachate

Sample	Data sys Code	Relative Retention times								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW(2)	C39490	0.2011	0.3845	0.4048	0.4704	0.5600	0.6662	0.6910	0.7437	1.0000
PE-TW-CI(2)	C39494	0.2003	0.3847	0.4045	0.4704	0.5595	0.6659	0.6918	0.7434	1.0000
GRP-TW(2)	C39489	0.2009	0.3841	0.4044	0.4702	0.0000	0.6660	0.6913	0.7432	1.0000
GRP-TW-CI(2)	C39493	0.2005	0.3847	0.4042	0.4708	0.0000	0.6663	0.6918	0.7437	1.0000
BIT-TW(2)	C39488	0.2016	0.3849	0.4044	0.4707	0.5597	0.6658	0.6913	0.7432	1.0000
BIT-TW-CI(1)	C39492	0.2006	0.3846	0.4044	0.4707	0.5594	0.6658	0.6916	0.7434	1.0000
BL-TW(2)	C39487	0.2014	0.3849	0.4045	0.4701	0.5596	0.6657	0.6918	0.7436	1.0000
BL-TW-CI(2)	C39491	0.2010	0.3844	0.4047	0.4703	0.5591	0.6660	0.6921	0.7432	1.0000
Mean		**0.2009**	**0.3846**	**0.4045**	**0.4705**	**0.4197**	**0.6660**	**0.6916**	**0.7434**	**1.0000**
SD		**0.0004**	**0.0003**	**0.0002**	**0.0003**	**0.2590**	**0.0002**	**0.0003**	**0.0002**	**0.0000**
%RSD		**0.21%**	**0.07%**	**0.05%**	**0.05%**	**61.72%**	**0.03%**	**0.05%**	**0.03%**	**0.00%**

Column test Results:

CCM Coltest	O101001	0.2015	0.3848	0.4043	0.4699	0.5593	0.6659	0.6919	0.7430	1.0000
CCM Coltest	O101007	0.2025	0.3851	0.4047	0.4703	0.5589	0.6660	0.6918	0.7437	1.0000
CCM Coltest	O101012	0.2017	0.3842	0.4044	0.4701	0.5591	0.6660	0.6911	0.7432	1.0000
Mean		**0.2019**	**0.3847**	**0.4045**	**0.4701**	**0.5591**	**0.6660**	**0.6916**	**0.7433**	**1.0000**
SD		**0.0005**	**0.0005**	**0.0002**	**0.0002**	**0.0002**	**0.0001**	**0.0004**	**0.0004**	**0.0000**
%RSD		**0.24%**	**0.13%**	**0.05%**	**0.05%**	**0.04%**	**0.01%**	**0.06%**	**0.05%**	**0.00%**

GC-MS General Survey AQC results

Peak areas of internal standards detected in leachates

Laboratory CRECEP - Batch 1

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys	Peak Area								
	Code	d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PB-TW(1)	D336-1		30	88.8	59.9	26.6	150.9	10.6	62.6	121.7
PB-TW-CL(1)	D336-2		21.8	55.1	27.9	21.5	120.4	13.8	58.2	158.1
PE-TW(1)	D336-3		27.5	104	39.9	25.9	165.2	13.6	59.3	157.6
PE-TW-CL(1)	D336-4		29.7	62.9	53.1	21.4	123	12.7	67.4	150
GRP-TW(1)	D336-5		23.7	50.4		39.1	157	16.5	55.4	200.3
GRP-TW-CL(1)	D336-6		29	13.5	40.7		46.6	8.5	61.2	88.1
BIT-TW(1)	D336-7		35.6	16.4	27.8	34.8	79.6		69.8	65.5
BIT-TW-CL(1)	D336-8		31.8	14.7	28.5	19.5		9	52.2	107.3
Mean			**28.6375**	**50.7250**	**39.6857**	**26.9714**	**120.3857**	**12.1000**	**60.7625**	**131.0750**
SD			**4.3756**	**34.4697**	**12.8772**	**7.3765**	**43.5788**	**2.8775**	**5.8564**	**43.7268**
%RSD			**15%**	**68%**	**32%**	**27%**	**36%**	**24%**	**10%**	**33%**

Column test Results:

Mean										
SD										
%RSD										

GC-MS General Survey AQC results

Peak area ratios (normalised to d62-squalane)

Laboratory CRECEP - Batch 1

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys	Peak Area ratios								
	Code	d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PB-TW(1)	D336-1		0.2465	0.7297	0.4922	0.2186	1.2399	0.0871	0.5144	1.0000
PB-TW-CL(1)	D336-2		0.1379	0.3485	0.1765	0.1360	0.7615	0.0873	0.3681	1.0000
PE-TW(1)	D336-3		0.1745	0.6599	0.2532	0.1643	1.0482	0.0863	0.3763	1.0000
PE-TW-CL(1)	D336-4		0.1980	0.4193	0.3540	0.1427	0.8200	0.0847	0.4493	1.0000
GRP-TW(1)	D336-5		0.1183	0.2516		0.1952	0.7838	0.0824	0.2766	1.0000
GRP-TW-CL(1)	D336-6		0.3292	0.1532	0.4620		0.5289	0.0965	0.6947	1.0000
BIT-TW(1)	D336-7		0.5435	0.2504	0.4244	0.5313	1.2153		1.0656	1.0000
BIT-TW-CL(1)	D336-8		0.2964	0.1370	0.2656	0.1817		0.0839	0.4865	1.0000
Mean			**0.2555**	**0.3687**	**0.3468**	**0.2243**	**0.9140**	**0.0869**	**0.5289**	**1.0000**
SD			**0.1377**	**0.2223**	**0.1189**	**0.1384**	**0.2621**	**0.0046**	**0.2498**	**0.0000**
%RSD			**54%**	**60%**	**34%**	**62%**	**29%**	**5%**	**47%**	**0%**

Column test Results:

Mean										
SD										
%RSD										

GC-MS General Survey AQC results

Retention times of internal standards

Laboratory CRECEP - Batch 1

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Retention time (minutes)								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PB-TW(1)	D336-1		4.85	5.05	5.98	8.15	12.4	13.54	18.27	34.27
PB-TW-CL(1)	D336-2		4.85	5.05	6.04	8.2	12.47	13.6	18.25	34.27
PE-TW(1)	D336-3		4.85	5.08	6.01	8.15	12.45	13.55	18.25	34.3
PE-TW-CL(1)	D336-4		4.89	5.04	6.04	8.16	12.45	13.59	18.23	34.29
GRP-TW(1)	D336-5		4.85	5.06	6.05	8.18	12.46	13.6	18.24	34.3
GRP-TW-CL(1)	D336-6		5.61	5.8	7.08	9.6	14.25	15.3	19.98	36.19
BIT-TW(1)	D336-7		5.65	5.86	7.12	9.62	14.23	15.34	20.05	36.24
BIT-TW-CL(1)	D336-8		5.58	5.85	7.13	9.57	14.28	15.3	19.98	36.19
Mean			**5.14**	**5.35**	**6.43**	**8.70**	**13.12**	**14.23**	**18.91**	**35.01**
SD			**0.392**	**0.405**	**0.563**	**0.740**	**0.936**	**0.900**	**0.909**	**0.994**
%RSD			**7.62%**	**7.56%**	**8.75%**	**8.50%**	**7.13%**	**6.32%**	**4.81%**	**2.84%**

Column test Results:

CCM Coltest	ETESTN4	3.04	4.69	4.87	5.96	8.19	12.50	13.64	18.33	34.36
CCM Coltest	ETESTN5		4.89	5.04	6.03	8.22	12.50	13.65	18.33	34.37
Mean		**3.04**	**4.79**	**4.96**	**6.00**	**8.21**	**12.50**	**13.65**	**18.33**	**34.37**
SD			**0.141**	**0.120**	**0.049**	**0.021**	**0.000**	**0.007**	**0.000**	**0.007**
%RSD			**2.95%**	**2.43%**	**0.83%**	**0.26%**	**0.00%**	**0.05%**	**0.00%**	**0.02%**

GC-MS General Survey AQC results

Relative retention times of internal standards (normalised to d62-squalane)

Laboratory CRECEP - Batch 1

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys	Relative Retention times								
	Code	d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PB-TW(1)	D336-1		0.1415	0.1474	0.1745	0.2378	0.3618	0.3951	0.5331	1.0000
PB-TW-CL(1)	D336-2		0.1415	0.1474	0.1762	0.2393	0.3639	0.3968	0.5325	1.0000
PE-TW(1)	D336-3		0.1414	0.1481	0.1752	0.2376	0.3630	0.3950	0.5321	1.0000
PE-TW-CL(1)	D336-4		0.1426	0.1470	0.1761	0.2380	0.3631	0.3963	0.5316	1.0000
GRP-TW(1)	D336-5		0.1414	0.1475	0.1764	0.2385	0.3633	0.3965	0.5318	1.0000
GRP-TW-CL(1)	D336-6		0.1550	0.1603	0.1956	0.2653	0.3938	0.4228	0.5521	1.0000
BIT-TW(1)	D336-7		0.1559	0.1617	0.1965	0.2655	0.3927	0.4233	0.5533	1.0000
BIT-TW-CL(1)	D336-8		0.1542	0.1616	0.1970	0.2644	0.3946	0.4228	0.5521	1.0000
Mean			**0.1467**	**0.1526**	**0.1835**	**0.2483**	**0.3745**	**0.4061**	**0.5398**	**1.0000**
SD			**0.0069**	**0.0071**	**0.0107**	**0.0139**	**0.0159**	**0.0140**	**0.0105**	**0.0000**
%RSD			**4.73%**	**4.67%**	**5.85%**	**5.60%**	**4.24%**	**3.44%**	**1.94%**	**0.00%**

Column test Results:

CCM Coltest	ETEST4N	0.0885	0.1365	0.1417	0.1735	0.2384	0.3638	0.3970	0.5335	1.0000
CCM Coltest	ETEST5N		0.1423	0.1466	0.1754	0.2392	0.3637	0.3971	0.5333	1.0000
Mean		**0.0885**	**0.1394**	**0.1442**	**0.1745**	**0.2388**	**0.3637**	**0.3971**	**0.5334**	**1.0000**
SD			**0.0041**	**0.0035**	**0.0014**	**0.0006**	**0.0001**	**0.0001**	**0.0001**	**0.0000**
%RSD			**2.93%**	**2.41%**	**0.81%**	**0.24%**	**0.02%**	**0.03%**	**0.02%**	**0.00%**

GC-MS General Survey AQC results

Peak areas of internal standards detected in leachates

Laboratory CRECEP - Batch 2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys	Peak Area								
	Code	d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PB-TW(2)	D509-1		17.21	19.53	13.47	9.57	99.68	3.32	27.54	37.34
PB-TW-CL(2)	D509-2		13.11	17.05	12.67	11.03	99.26	3.9	27.97	35.63
PE-TW(2)	D509-3		25.89	27.59	44.33	22.78	164.9	13.09	56.69	176.2
PE-TW-CL(2)	D509-4		12.94	14.82	15.58	8.65	67.8	5.22	24.05	62.24
GRP-TW(2)	D509-5		16.14	19.57	20.19		82.78	3.59	30.1	39.65
GRP-TW-CL(2)	D509-6		7.26	12.09	8.82		52.18	2.14	18.82	22.4
BIT-TW(2)	D509-7		13.93	13.24	13.06	12.73	42.26	2.03	34.8	30.37
BIT-TW-CL(2)	D509-8		13.6	19.98	13.94	8.98	31.56	1.82	30.93	21.85
Mean			**15.0100**	**17.9838**	**17.7575**	**12.2900**	**80.0625**	**4.3888**	**31.3625**	**53.2100**
SD			**5.2861**	**4.9178**	**11.1966**	**5.3558**	**42.5399**	**3.6963**	**11.2883**	**51.2797**
%RSD			**35%**	**27%**	**63%**	**44%**	**53%**	**84%**	**36%**	**96%**

Column test Results:

GC-MS General Survey AQC results

Peak area ratios (normalised to d62-squalane)

Laboratory CRECEP - Batch 2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Peak Area ratios								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PB-TW(2)	D509-1		0.4609	0.5230	0.3607	0.2563	2.6695	0.0889	0.7375	1.0000
PB-TW-CL(2)	D509-2		0.3679	0.4785	0.3556	0.3096	2.7859	0.1095	0.7850	1.0000
PE-TW(2)	D509-3		0.1469	0.1566	0.2516	0.1293	0.9359	0.0743	0.3217	1.0000
PE-TW-CL(2)	D509-4		0.2079	0.2381	0.2503	0.1390	1.0893	0.0839	0.3864	1.0000
GRP-TW(2)	D509-5		0.4071	0.4936	0.5092		2.0878	0.0905	0.7591	1.0000
GRP-TW-CL(2)	D509-6		0.3241	0.5397	0.3938		2.3295	0.0955	0.8402	1.0000
BIT-TW(2)	D509-7		0.4587	0.4360	0.4300	0.4192	1.3915	0.0668	1.1459	1.0000
BIT-TW-CL(2)	D509-8		0.6224	0.9144	0.6380	0.4110	1.4444	0.0833	1.4156	1.0000
Mean			**0.3745**	**0.4725**	**0.3987**	**0.2774**	**1.8417**	**0.0866**	**0.7989**	**1.0000**
SD			**0.1508**	**0.2264**	**0.1295**	**0.1269**	**0.7194**	**0.0130**	**0.3597**	**0.0000**
%RSD			40%	48%	32%	46%	39%	15%	45%	0%

Column test Results:

GC-MS General Survey AQC results

Retention times of internal standards

Laboratory CRECEP - Batch 2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Retention time (minutes)								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PB-TW(2)	D509-1		5.62	5.87	7.09	9.6	14.15	15.34	20.03	36.27
PB-TW-CL(2)	D509-2		5.65	5.93	7.1	9.61	14.16	15.36	20.05	36.27
PE-TW(2)	D509-3		5.68	5.93	7.1	9.62	14.17	15.35	20.05	36.28
PE-TW-CL(2)	D509-4		5.63	5.89	7.09	9.61	14.16	15.34	20.03	36.24
GRP-TW(2)	D509-5		5.62	5.88	7.12	9.89	14.19	15.37	20.07	36.29
GRP-TW-CL(2)	D509-6		5.66	5.83	7.11	9.61	14.18	15.37	20.06	36.29
BIT-TW(2)	D509-7		5.67	5.92	7.15	9.64	14.27	15.4	20.11	36.32
BIT-TW-CL(2)	D509-8		5.68	5.87	7.15	9.64	14.3	15.38	20.08	36.29
Mean			**5.65**	**5.89**	**7.11**	**9.65**	**14.20**	**15.36**	**20.06**	**36.28**
SD			**0.025**	**0.035**	**0.024**	**0.097**	**0.056**	**0.021**	**0.027**	**0.023**
%RSD			**0.45%**	**0.60%**	**0.34%**	**1.01%**	**0.39%**	**0.13%**	**0.13%**	**0.06%**

Column test Results:

CCM Coltest	ETESTN6		5.60	5.80	7.06	9.58	14.10	15.28	19.96	36.18
CCM Coltest	ETESTN7		5.62	5.85	7.08	9.60	14.13	15.33	20.02	36.22
Mean			**5.61**	**5.83**	**7.07**	**9.59**	**14.12**	**15.31**	**19.99**	**36.20**
SD			**0.014**	**0.035**	**0.014**	**0.014**	**0.021**	**0.035**	**0.042**	**0.028**
%RSD			**0.25%**	**0.61%**	**0.20%**	**0.15%**	**0.15%**	**0.23%**	**0.21%**	**0.08%**

GC-MS General Survey AQC results

Relative retention times of internal standards (normalised to d62-squalane)

Laboratory CRECEP - Batch 2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Relative Retention times								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PB-TW(2)	D509-1		0.1549	0.1618	0.1955	0.2647	0.3901	0.4229	0.5522	1.0000
PB-TW-CL(2)	D509-2		0.1558	0.1635	0.1958	0.2650	0.3904	0.4235	0.5528	1.0000
PE-TW(2)	D509-3		0.1566	0.1635	0.1957	0.2652	0.3906	0.4231	0.5526	1.0000
PE-TW-CL(2)	D509-4		0.1554	0.1625	0.1956	0.2652	0.3907	0.4233	0.5527	1.0000
GRP-TW(2)	D509-5		0.1549	0.1620	0.1962	0.2725	0.3910	0.4235	0.5530	1.0000
GRP-TW-CL(2)	D509-6		0.1560	0.1607	0.1959	0.2648	0.3907	0.4235	0.5528	1.0000
BIT-TW(2)	D509-7		0.1561	0.1630	0.1969	0.2654	0.3929	0.4240	0.5537	1.0000
BIT-TW-CL(2)	D509-8		0.1565	0.1618	0.1970	0.2656	0.3940	0.4238	0.5533	1.0000
Mean			**0.1558**	**0.1623**	**0.1961**	**0.2660**	**0.3913**	**0.4235**	**0.5529**	**1.0000**
SD			**0.0007**	**0.0010**	**0.0006**	**0.0026**	**0.0014**	**0.0004**	**0.0004**	**0.0000**
%RSD			**0.42%**	**0.60%**	**0.30%**	**0.99%**	**0.36%**	**0.08%**	**0.08%**	**0.00%**

Column test Results:

CCM Coltest	ETESTN6		0.1548	0.1603	0.1951	0.2648	0.3897	0.4223	0.5517	1.0000
CCM Coltest	ETESTN7		0.1552	0.1615	0.1955	0.2650	0.3901	0.4232	0.5527	1.0000
Mean			**0.1550**	**0.1609**	**0.1953**	**0.2649**	**0.3899**	**0.4228**	**0.5522**	**1.0000**
SD			**0.0003**	**0.0009**	**0.0002**	**0.0002**	**0.0003**	**0.0006**	**0.0007**	**0.0000**
%RSD			**0.17%**	**0.53%**	**0.12%**	**0.07%**	**0.07%**	**0.15%**	**0.13%**	**0.00%**

GC-MS General Survey AQC results

Peak areas of internal standards detected in leachates

Laboratory KIWA - Batch 1/2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys	Peak Area								
	Code	d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW	O-950288/9		162	51	370	52	776	59	210	362
PE-TW-Cl	O-950299/0		183	51	375	42	742	45	182	147
GRP-TW	O-950290/1		195	55	476	53	907	50	185	110
GRP-TW-CL	O-950301/2		198	53	443	58	950	56	209	147
BIT-TW	O-950292/3		165	54	406	54	721	68	173	203
BIT-TW-Cl	O-950303/4		192	48	419	54	794	61	182	140
BL-TW	O-950286/7		154	52	383	56	715	59	207	320
BL-TW-Cl	O-950297/8		166	42	324	42	609	44	163	154
Mean			177	51	400	51	777	55	189	198
SD			17	4	47	6	109	8	18	93
%RSD			10%	8%	12%	12%	14%	15%	9%	47%

Column test Results:

CCM Coltest										
CCM Coltest										
CCM Coltest										
Mean										
SD										
%RSD										

GC-MS General Survey AQC results

Peak area ratios (normalised to d62-squalane)

Laboratory KIWA - Batch 1/2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys Code	Peak Area ratios								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW	O-950288/9		0.4475	0.1409	1.0221	0.1436	2.1436	0.1630	0.5801	1.0000
PE-TW-CI	O-950299/0		1.2449	0.3469	2.5510	0.2857	5.0476	0.3061	1.2381	1.0000
GRP-TW	O-950290/1		1.7727	0.5000	4.3273	0.4818	8.2455	0.4545	1.6818	1.0000
GRP-TW-CL	O-950301/2		1.3469	0.3605	3.0136	0.3946	6.4626	0.3810	1.4218	1.0000
BIT-TW	O-950292/3		0.8128	0.2660	2.0000	0.2660	3.5517	0.3350	0.8522	1.0000
BIT-TW-CI	O-950303/4		1.3714	0.3429	2.9929	0.3857	5.6714	0.4357	1.3000	1.0000
BL-TW	O-950286/7		0.4813	0.1625	1.1969	0.1750	2.2344	0.1844	0.6469	1.0000
BL-TW-CI	O-950297/8		1.0779	0.2727	2.1039	0.2727	3.9545	0.2857	1.0584	1.0000
Mean			**1.0694**	**0.2991**	**2.4010**	**0.3007**	**4.6639**	**0.3182**	**1.0974**	**1.0000**
SD			**0.4617**	**0.1158**	**1.0730**	**0.1143**	**2.1119**	**0.1068**	**0.3854**	**0.0000**
%RSD			**43%**	**39%**	**45%**	**38%**	**45%**	**34%**	**35%**	**0%**

Column test Results:

CCM Coltest										
CCM Coltest										
CCM Coltest										
Mean										
SD										
%RSD										

GC-MS General Survey AQC results

Retention indices (Kovats) of internal standards

Laboratory KIWA - Batch 1/2

Sample Volume: 1 litre
Final Extract Vol: 500ul
Sample type Lab test leachate

Sample	Data sys Code	Kovats Index								
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW	O-950288/9		839	862	972	1162	1496	1581	1800	2620
PE-TW-CL	O-950299/0		839	862	972	1162	1496	1581	1800	2620
GRP-TW	O-950290/1		839	862	972	1162	1496	1581	1800	2620
GRP-TW-CL	O-950301/2		839	862	972	1162	1496	1581	1800	2620
BIT-TW	O-950292/3		839	862	972	1162	1496	1581	1800	2620
BIT-TW-CL	O-950303/4		839	862	972	1162	1496	1581	1800	2620
BL-TW	O-950286/7		839	862	972	1162	1496	1581	1800	2620
BL-TW-CL	O-950297/8		839	862	972	1162	1496	1581	1800	2620
Mean			839.00	862.00	972.00	1162.00	1496.00	1581.00	1800.00	2620.00
SD			0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
%RSD			0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%

Column test Results:

CCM Coltest										
CCM Coltest										
CCM Coltest										
Mean										
SD										
%RSD										

GC-MS General Survey AQC results

Relative retention indices of internal standards (normalised to d62-squalane)

Laboratory KIWA - Batch 1/2

Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

Sample	Data sys	Relative retention indices								
	Code	d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane
PE-TW	O-950288/9		0.3202	0.3290	0.3710	0.4435	0.5710	0.6034	0.6870	1.0000
PE-TW-CI	O-950299/0		0.3202	0.3290	0.3710	0.4435	0.5710	0.6034	0.6870	1.0000
GRP-TW	O-950290/1		0.3202	0.3290	0.3710	0.4435	0.5710	0.6034	0.6870	1.0000
GRP-TW-CL	O-950301/2		0.3202	0.3290	0.3710	0.4435	0.5710	0.6034	0.6870	1.0000
BIT-TW	O-950292/3		0.3202	0.3290	0.3710	0.4435	0.5710	0.6034	0.6870	1.0000
BIT-TW-CI	O-950303/4		0.3202	0.3290	0.3710	0.4435	0.5710	0.6034	0.6870	1.0000
BL-TW	O-950286/7		0.3202	0.3290	0.3710	0.4435	0.5710	0.6034	0.6870	1.0000
BL-TW-CI	O-950297/8		0.3202	0.3290	0.3710	0.4435	0.5710	0.6034	0.6870	1.0000
Mean			**0.3202**	**0.3290**	**0.3710**	**0.4435**	**0.5710**	**0.6034**	**0.6870**	**1.0000**
SD			0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
%RSD			0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%

Column test Results:

CCM Coltest										
CCM Coltest										
CCM Coltest										
Mean										
SD										
%RSD										