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

Final Report

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

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

EXECUTIVE 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. 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.

The protocols produced are therefore satisfactory, in that they allow organic compounds in leachates from materials intended for use in contact with drinking water to be reliably determined at concentrations in the range 1-10 $\mu\text{g l}^{-1}$. The limit of detection is about 0.75 $\mu\text{g l}^{-1}$.

CONTENTS

Page

EXECUTIVE SUMMARY	i
1. INTRODUCTION	1
2. WORK PROGRAMME	3
2.1 Protocols	3
2.2 Materials used for performance testing of the protocols	3
2.3 Laboratories undertaking performance testing	4
3. RESULTS	5
3.1 Protocols	5
3.2 Within-laboratory performance testing of protocols	7
3.3 Interlaboratory performance testing of protocols	9
4. DISCUSSION	12
5. CONCLUSIONS	13

1. INTRODUCTION

Manufacturers wishing to use new materials (e.g. pipes or pipe-lining materials) designed to come into contact with drinking water, must gain approval for the intended use from the Committee (CCM) which advises the Secretary of State on such matters. Tests on these materials are necessary 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. Additionally in the UK, there is a requirement to apply a basic gas chromatography-mass spectrometry (GCMS) method, intended to detect, identify and quantify unsuspected contaminants in materials leachates.

Currently the 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 and, when agreed, these standards will also become national standards within the EU and EFTA countries. In order to promote the adoption of the UK view (*viz.* that general survey GCMS analysis should be a requirement for this type of materials testing) standardised procedures of known performance are required. The 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, and 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 DWI's 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, as 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. $\pm 1-2$ 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 write this protocol in such a way as 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.

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.

It 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} -phenanthrene) 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_{10} -phenanthrene
d_{20} -BHT	d_5 -phenol
d_5 -chlorobenzene	d_{62} -squalane
d_{34} -hexadecane	d_{10} - <i>p</i> -xylene
d_8 -naphthalene	

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 specifications 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 $\mu\text{g l}^{-1}$, three (d_6 -benzene, d_5 -chlorobenzene and d_{10} -phenanthrene) are present at 2 $\mu\text{g l}^{-1}$, and the remaining three (d_{20} -BHT, d_5 -phenol and d_{62} -squalane) are present at 8 $\mu\text{g l}^{-1}$. These levels were chosen to allow the protocol to be performance tested over the range 0-10 $\mu\text{g l}^{-1}$, 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 $0.5 \mu\text{g l}^{-1}$; d_{10} -phenanthrene, present at $2.0 \mu\text{g l}^{-1}$; d_{62} -squalane, present at $8.0 \mu\text{g l}^{-1}$) 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 on the temperature programming rate, 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.

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 $\mu\text{g l}^{-1}$, and it was necessary to demonstrate that a detection limit of $<1 \mu\text{g l}^{-1}$ could be achieved. The internal standards added to the samples at 2 $\mu\text{g l}^{-1}$ (d_6 -benzene, d_5 -chlorobenzene and d_{10} -phenanthrene) represent the lower end of the range of interest, those added at 8 $\mu\text{g l}^{-1}$ (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 $\mu\text{g l}^{-1}$ (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 $\mu\text{g l}^{-1}$) to 49% (for d_{20} -BHT, present at 8.0 $\mu\text{g l}^{-1}$). For the internal standards present at 0.5 $\mu\text{g l}^{-1}$ (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 $4.65 \times$ standard deviation) for these three internal standards is about 0.75 $\mu\text{g l}^{-1}$, which is below the desired LOD (1 $\mu\text{g l}^{-1}$). 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 $\mu\text{g l}^{-1}$ 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 rating 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 the Department.

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, for there was an improvement in the data produced. The RSD of the peak areas for the internal standards ranged from 13% (for d₂₀-BHT) to 29% (for d₆-benzene and d₅-phenol). Obviously the inclusion of the dechlorination step resulted in the marked change noted for d₂₀-BHT (RSD reduced from 49% to 13%). 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. However, when the variation was expressed as a percentage, the RSD for the retention time of d₆-benzene (3.5%) was greater than that specified in the protocol (2%). This is mainly due to the fact that this internal standard has the shortest retention time, and although in terms of absolute time the variation is no worse than for the other internal standards, when expressed as a percentage this variation is exaggerated.

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.

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.

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.

4. DISCUSSION

The results obtained from the inter-laboratory performance testing indicate that competent laboratories following the protocols for the production and GCMS analysis of leachates from materials intended for use in contact with drinking water can produce comparable results.

Following the GCMS protocol allows organic compounds present in materials leachates in the range 1-100 $\mu\text{g l}^{-1}$ to be quantified, and consistent mass spectra can be obtained. While this latter factor does not automatically lead to the identification of detected compounds, it does maximise the chances of doing so.

Some differences noted when comparing results produced by the various laboratories were apparently due to instrumental factors, but these are not considered significant.

5. CONCLUSIONS

Two protocols for materials testing have been written. One relates to the production of leachates from non-metallic (non-cementitious) factory-produced products. The other relates to the analysis of these leachates for unspecified organic compounds using GCMS. Both protocols have been drafted in BSi format.

Interlaboratory performance testing of these protocols by four laboratories (two within the UK, two outside the UK but within the EU) has shown that they can be successfully utilised to produce comparable results.

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 cm² /ml. For cylindrical pipes $S/V = 4/(\text{internal diameter in cm})$.

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 $0.2 \pm 0.05 \text{ mg l}^{-1}$.

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

$\pm 0.2 \text{ mg l}^{-1}$, 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 $25 \pm 2^\circ\text{C}$. 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 $25 \pm 2^\circ\text{C}$.

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 test 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 $25 \pm 2^\circ\text{C}$.

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 contact 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: ~843.5cm²

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 Theoretical: 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:** 2250cm³
- **Surface to volume ratio:** 0.37cm²cm⁻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: ~843.5cm²

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 Theoretical: 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:** 2250cm³

- **Surface to volume ratio:** 0.37cm²cm⁻³

- **Temperature °C:** 25

Deviations from test procedure

TEST LABORATORY WRC MEDMENHAM.

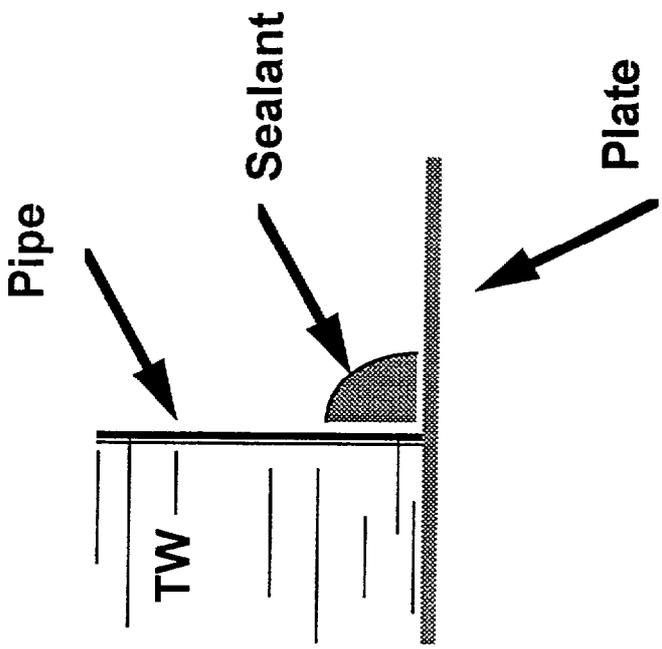
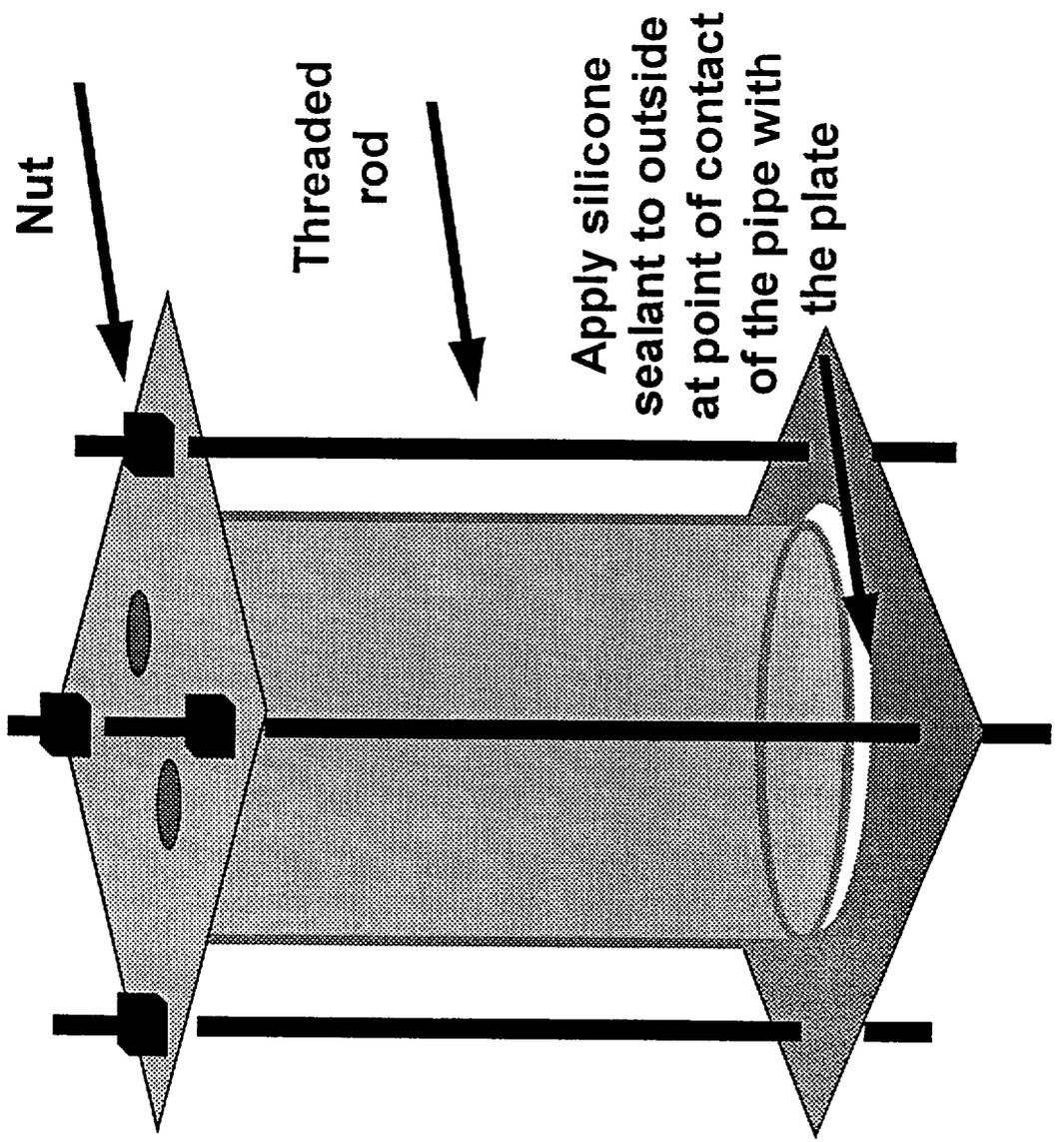
BATCH No 1.

SAMPLES PE, GRP, BiEumen

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 pre washing	16 th Jan 95	10-55	17 th Jan 95	9-00	22.1
Flush	17 th Jan 95	9-15	17 th Jan 95	10-20	1.1
Rinse	17 th Jan 95	10-25	17 th Jan 95	10-45	0.3
leaching	17 th Jan 95	11-00	20 th Jan 95	11-35	72.6
Sample collection	20 th Jan 95	11-35	20 th Jan 95	11-55	0.3



APPENDIX B

**GCMS GENERAL SURVEY PROCEDURE FOR THE
ANALYSIS OF LEACHATES PRODUCED FROM LEACH-
ING 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.

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

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

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 (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:

d₆-benzene
d₂₀-2,6-di-t-butyl-4-methylphenol
d₅-chlorobenzene
d₃₄-hexadecane
d₈-naphthalene
d₁₀-phenanthrene
d₅-phenol
d₆₂-squalane
d₁₀-p-xylene

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_{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 μ l +/- 2 μ l 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 μ l and 500 μ l 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 μ l. 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 µl 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 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.

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 heptacosafuorotributylamine (Heptacosafuorotributylamine) 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 manufacturer's 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 i.e. d_6 -benzene must be separated from the solvent peak, and the retention time of d_{62} -squalane must be between 35 and 45 minutes. All of the internal standards should 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 of 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) or the scan number of the peak maximum of all of the peaks detected; this should include the retention times or scan numbers 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:

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

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 d₅-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 d₈-naphthalene and d₅-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 d₈-naphthalene, d₁₀-phenanthrene and d₆₂-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, should 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 or scan number 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 suspected 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.

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: Dr Huw James

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Report serial number: 9076-1307

Project Number: 09076-0

Date of issue: 16-November 1994

Report produced by: WRc Analysis
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MEDMENHAM

GC-MS General Survey Results

Page 1 of 2

Project No: 8782-0
 Analyst: RK
 Sample Volume: 1 litre
 Final Extract Vol: 500 ul
 Sample Type: Example results table

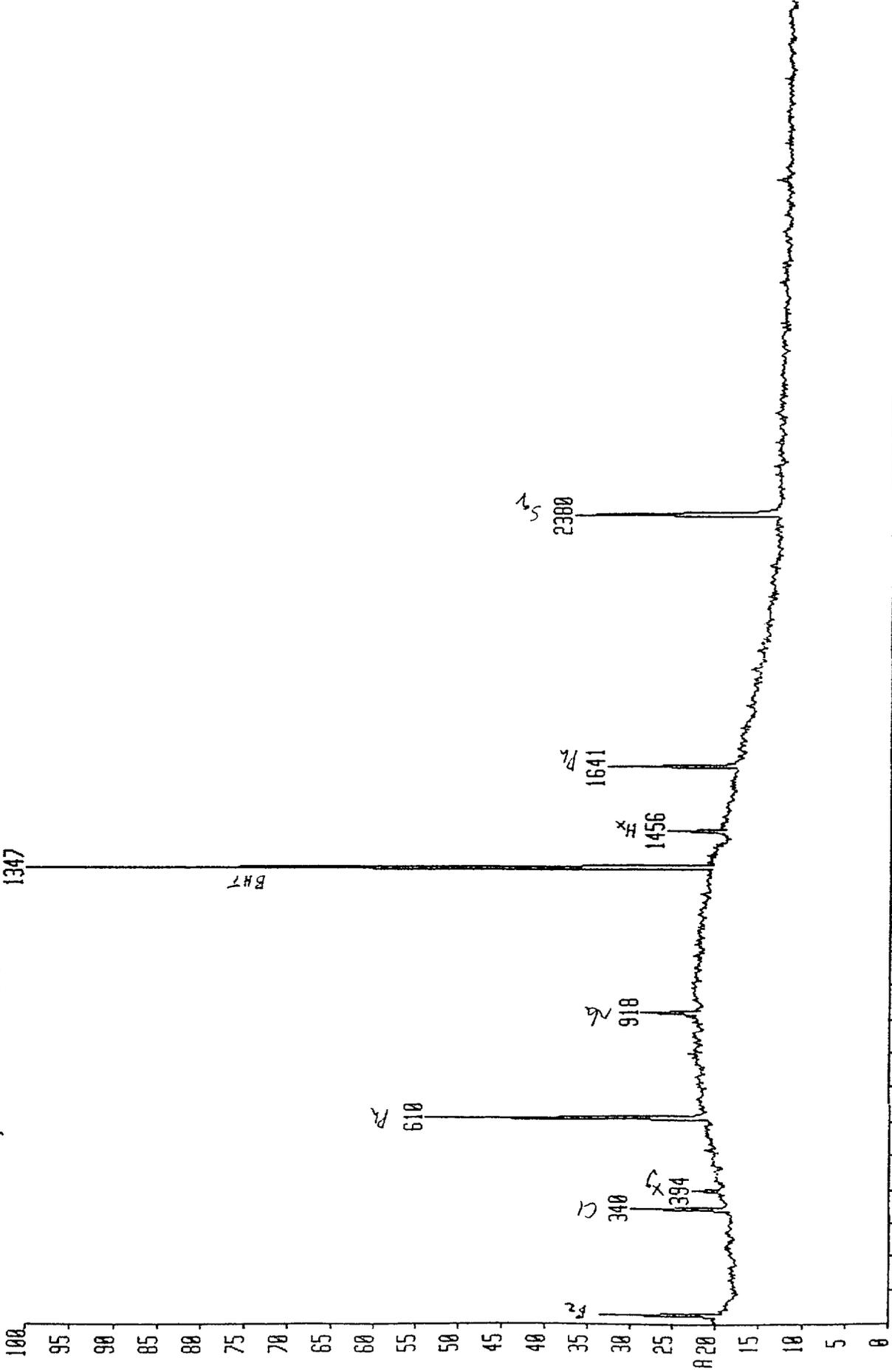
Form No: xxxx
 Data System Code: xxxx
 Sample Code : xxxx
 Associated Blank: Borehole

Date Sampled: 09-Nov-94
 Date Received: 09-Nov-94
 Date Analysed: 15-Nov-94

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-butandioic 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

Chromatogram Identifiers : A : ATIC
Text: EXTRACT IN DCM, 30C 4M 8C/M 300C

Fl: 10917

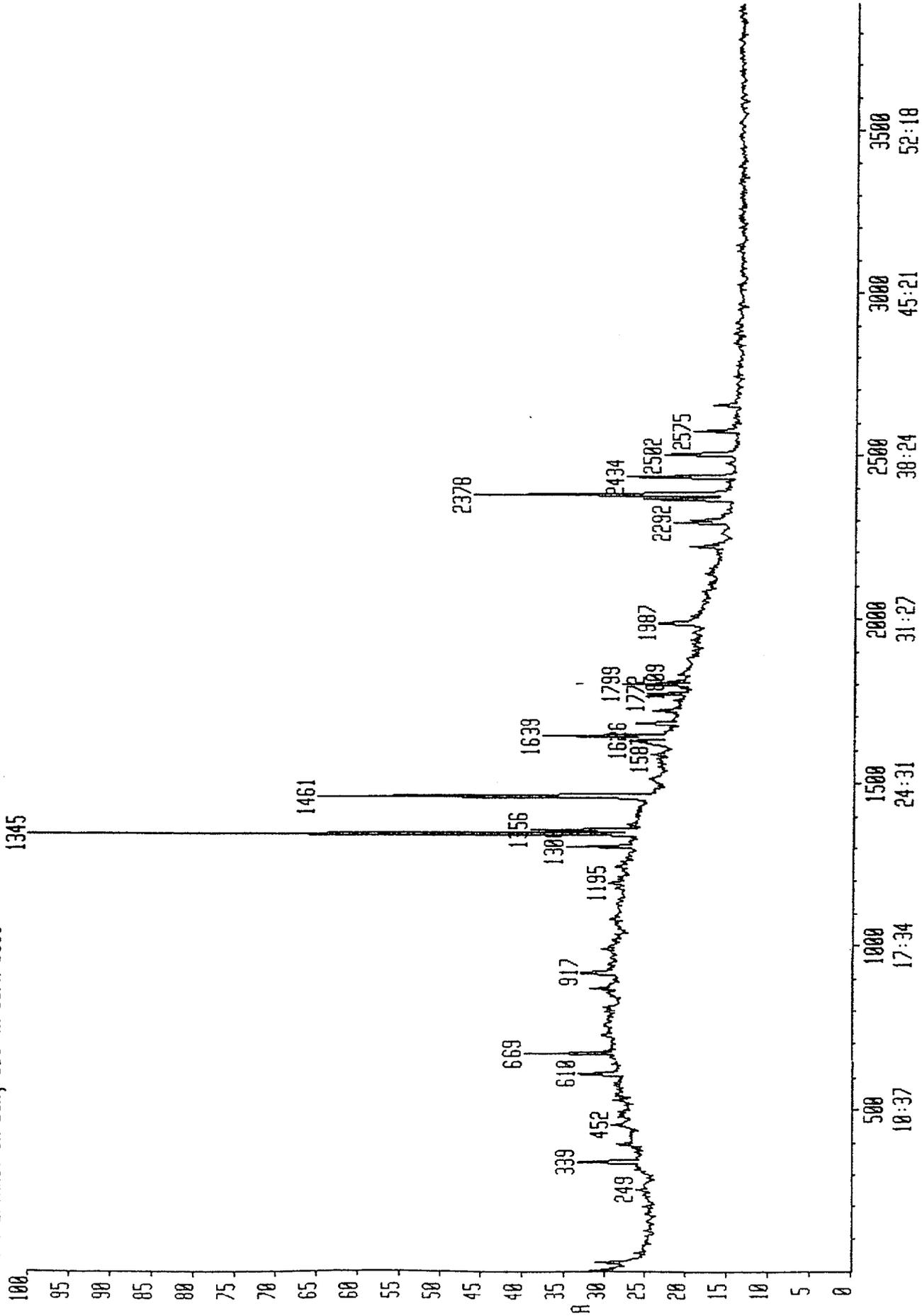


SCAN	TIME
500	10:37
1000	17:35
1500	24:32
2000	31:29
2500	38:26
3000	45:24
3500	52:21

0780P031 #1-3890 15-DEC-94 16:18 70E
Chromatogram Identifiers : A :PTIC
Text:EXTRACT IN DCM, 30C 4M 8C/M 300C

(E1+)
Suj:GENSURVEY

IHP
AI: 8275



SPEL000001 XI R00=0
VpI=0 I=64uvs H#271 TIC=0
SPEL000001-SPEL000001

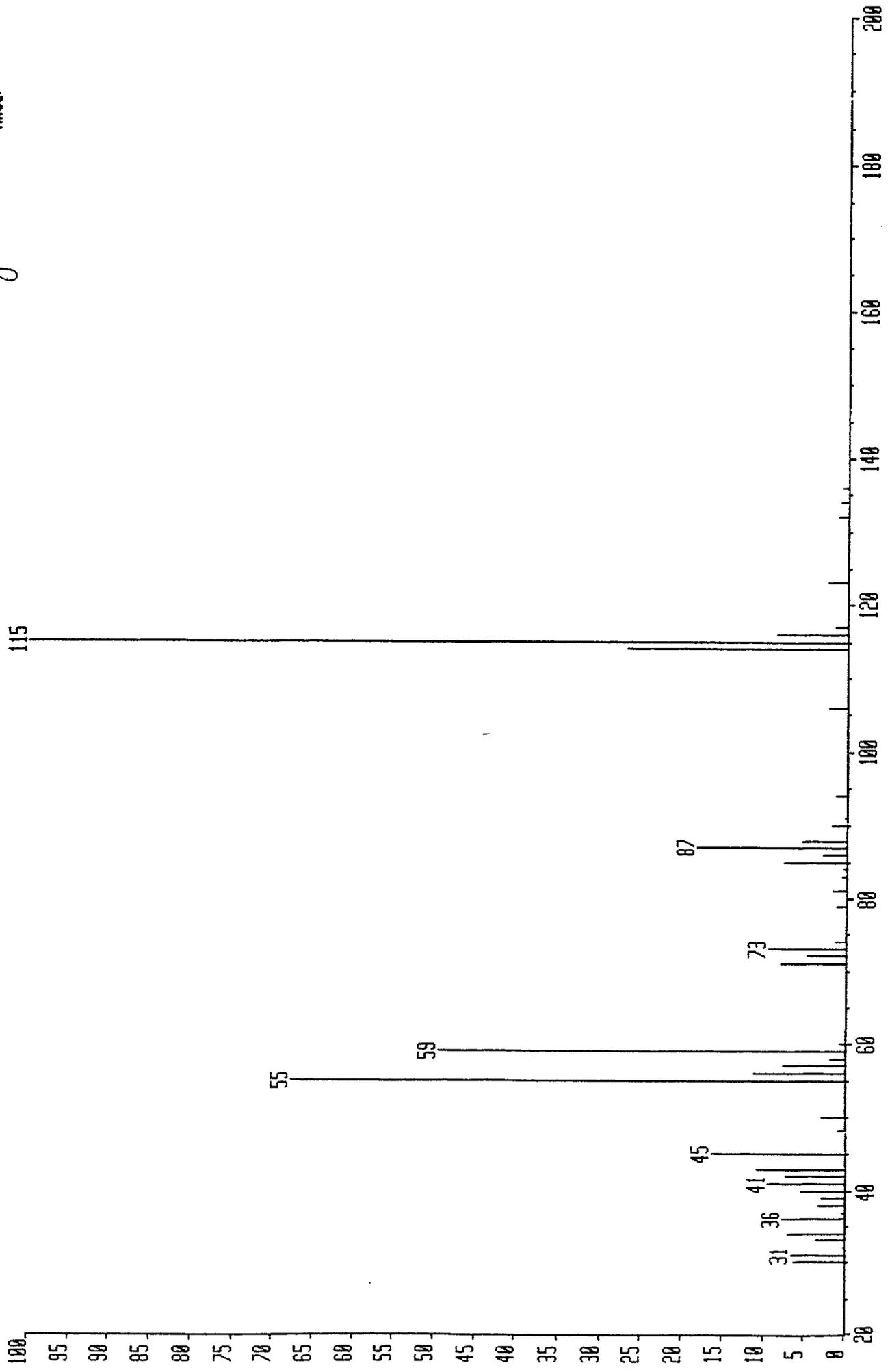
+0:00:00
SU

Acnt:
PT=0°

Sys:
Cal:

Scan 68 (background subtracted,
averaged)

940000
115



APPENDIX C

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

Project No: 8782-0
Form Number: 1307/1313
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-B1	0780P031	694	1622	832	2749	964	14691	941	3381	12275	
BL-CL-B2	0780P041	521	1212	422	1965	723	3494	717	2869	7846	
BIT-CL	0780P051	836	1401	697	3348	933	10668	882	2902	8974	
1CLB	0780P061	582	943	423	1997	750	6356	911	2679	9093	
UNCLB	0780P071	364	1489	392	2329	801	12885	863	2870	10332	
BIT-B1	0780P101	185	655	480	2523	632	7802	700	3017	5796	
PE-CL	0780P111	800	1265	531	3202	848	7726	776	3234	11517	
GRP-CL	0780P121	308	1068	257	1537	485	0	768	3533	7974	
BL-B2	0780P131	479	1500	366	1679	674	11848	620	2124	9142	
GRP-B2	0780P141	347	1433	416	2638	271	10424	1324	2484	12974	
BIT-B2	0780P171	316	929	403	895	622	7421	628	2463	4899	
BIT-CL	0780P181	637	1736	480	1202	529	11673	459	4035	5752	
PE-B2	0780P191	260	710	416	690	492	4320	211	1733	3656	
GRP-CL	0780P201	303	1027	418	1417	522	14511	1028	3025	11766	
GRP-B1	0780P211	270	1212	426	1773	914	7934	465	2008	8924	
Mean		460	1213	464	1996	677	8784	753	2824	8728	
SD		208	323	138	797	197	4176	266	606	2800	
%RSD		45%	27%	30%	40%	29%	48%	35%	21%	32%	

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
CCM Coltest	0780P221	3333	3101	1277	3417	1068	19723	1390	3492	12981
CCM Coltest	0780P151	3895	3400	986	7903	1086	18756	1695	4506	11325
Mean		3524	3072	1125	6709	1280	19599	1728	4172	11960
SD		252	240	141	2248	240	1044	321	680	715
%RSD		7%	8%	13%	34%	19%	5%	19%	16%	6%

GC-MS General Survey AQC results

Project No: 8782-0
 Form Number: 1307/1313
 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-B1	0780P031	0.0565	0.1321	0.0678	0.2240	0.0785	1.1968	0.0767	0.2754	1.0000	
BL-CL-B2	0780P041	0.0664	0.1545	0.0538	0.2504	0.0921	0.4453	0.0914	0.3657	1.0000	
BIT-CL	0780P051	0.0932	0.1561	0.0777	0.3731	0.1040	1.1888	0.0983	0.3234	1.0000	
1CLB	0780P061	0.0640	0.1037	0.0465	0.2196	0.0825	0.6990	0.1002	0.2946	1.0000	
UNCLB	0780P071	0.0352	0.1441	0.0379	0.2254	0.0775	1.2471	0.0835	0.2778	1.0000	
BIT-B1	0780P101	0.0319	0.1130	0.0828	0.4353	0.1090	1.3461	0.1208	0.5205	1.0000	
PE-CL	0780P111	0.0695	0.1098	0.0461	0.2780	0.0736	0.6708	0.0674	0.2808	1.0000	
GRP-CL	0780P121	0.0386	0.1339	0.0322	0.1928	0.0608	0.0000	0.0963	0.4431	1.0000	
BL-B2	0780P131	0.0524	0.1641	0.0400	0.1837	0.0737	1.2960	0.0678	0.2323	1.0000	
GRP-B2	0780P141	0.0267	0.1105	0.0321	0.2033	0.0209	0.8035	0.1021	0.1915	1.0000	
BIT-B2	0780P171	0.0645	0.1896	0.0823	0.1827	0.1270	1.5148	0.1282	0.5028	1.0000	
BIT-CL	0780P181	0.1107	0.3018	0.0834	0.2090	0.0920	2.0294	0.0798	0.7015	1.0000	
PE-B2	0780P191	0.0711	0.1942	0.1138	0.1887	0.1346	1.1816	0.0577	0.4740	1.0000	
GRP-CL	0780P201	0.0258	0.0873	0.0355	0.1204	0.0444	1.2333	0.0874	0.2571	1.0000	
GRP-B1	0780P211	0.0303	0.1358	0.0477	0.1987	0.1024	0.8891	0.0521	0.2250	1.0000	
Mean		0.0558	0.1487	0.0586	0.2323	0.0849	1.0494	0.0873	0.3577	1.0000	
SD		0.0251	0.0523	0.0245	0.0789	0.0296	0.4837	0.0215	0.1419	0.0000	
%RSD		45%	35%	42%	34%	35%	46%	25%	40%	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
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		0.2961	0.2578	0.0938	0.5694	0.1075	1.6414	0.1453	0.3510	1.0000
SD		0.0360	0.0289	0.0072	0.2074	0.0221	0.0977	0.0303	0.0678	0.0000
%RSD		12%	11%	8%	36%	21%	6%	21%	19%	0%

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

Sample	Data sys Code	Retention Time/min									
		d6-Benzene	d5-Chlorobenzene	d10-p-Xylene	d5-Phenol	d8-Naphthalene	d20-BHT*	d34-Hexadecane	d10-Phenanthrene	d62-Squalane	
PE-B1	0780P031	4.00	8.38	9.13	12.15	16.41	22.36	23.88	26.45	36.72	
BL-CL-B2	0780P041	4.00	8.38	9.12	12.15	16.40	22.36	23.88	26.45	36.73	
BIT-CL	0780P051	4.00	8.39	9.13	12.16	16.41	22.36	23.88	26.46	36.72	
1CLB	0780P061	4.00	8.39	9.12	12.13	16.41	22.36	23.88	26.45	36.72	
UNCLB	0780P071	4.01	8.39	9.14	12.13	16.41	22.36	23.88	26.45	36.72	
BIT-B1	0780P101	3.99	8.36	9.12	12.15	16.39	22.35	23.86	26.43	36.69	
PE-CL	0780P111	4.01	8.39	9.13	12.13	16.41	22.36	23.88	26.45	36.71	
GRP-CL	0780P121	4.00	8.36	9.10	12.12	16.39	22.33	23.85	26.42	36.68	
BL-B2	0780P131	3.99	8.35	9.10	12.12	16.40	22.33	23.84	26.43	36.67	
GRP-B2	0780P141	3.99	8.36	9.10	12.15	16.39	22.33	23.85	26.42	36.67	
BIT-B2	0780P171	4.00	8.36	9.12	12.15	16.39	22.35	23.86	26.43	36.65	
BIT-CL	0780P181	4.00	8.36	9.10	12.15	16.40	22.33	23.88	26.41	36.64	
PE-B2	0780P191	4.00	8.35	9.12	12.15	16.39	22.32	23.85	26.41	36.64	
GRP-CL	0780P201	3.96	8.35	9.12	12.12	16.39	22.39	23.84	26.42	36.65	
GRP-B1	0780P211	4.01	8.38	9.12	12.13	16.39	22.35	23.85	26.45	36.67	
Mean		4.00	8.37	9.12	12.14	16.40	22.35	23.86	26.44	36.68	
SD		0.014	0.016	0.012	0.013	0.013	0.018	0.016	0.017	0.034	
%RSD		0.35%	0.19%	0.13%	0.10%	0.08%	0.08%	0.07%	0.06%	0.09%	

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
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	9.11	12.14	16.39	22.35	23.85	26.42	36.65
SD		0.024	0.013	0.007	0.013	0.007	0.030	0.017	0.020	0.013
%RSD		0.60%	0.16%	0.08%	0.11%	0.04%	0.13%	0.07%	0.07%	0.04%

GC-MS General Survey AQC results

Project No: 8782-0
 Form Number: 1307/1313
 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-B1	0780P031	0.1089	0.2282	0.2486	0.3308	0.4470	0.6090	0.6502	0.7203	1.0000	
BL-CL-B2	0780P041	0.1089	0.2281	0.2481	0.3306	0.4464	0.6087	0.6500	0.7200	1.0000	
BIT-CL	0780P051	0.1089	0.2286	0.2486	0.3311	0.4470	0.6090	0.6502	0.7206	1.0000	
1CLB	0780P061	0.1089	0.2286	0.2482	0.3304	0.4470	0.6090	0.6502	0.7203	1.0000	
UNCLB	0780P071	0.1093	0.2286	0.2490	0.3304	0.4470	0.6090	0.6502	0.7203	1.0000	
BIT-B1	0780P101	0.1086	0.2280	0.2484	0.3310	0.4465	0.6091	0.6504	0.7204	1.0000	
PE-CL	0780P111	0.1094	0.2286	0.2487	0.3305	0.4471	0.6092	0.6505	0.7205	1.0000	
GRP-CL	0780P121	0.1091	0.2281	0.2481	0.3304	0.4467	0.6089	0.6502	0.7203	1.0000	
BL-B2	0780P131	0.1087	0.2278	0.2482	0.3305	0.4473	0.6091	0.6501	0.7210	1.0000	
GRP-B2	0780P141	0.1087	0.2281	0.2482	0.3313	0.4469	0.6091	0.6505	0.7206	1.0000	
BIT-B2	0780P171	0.1091	0.2282	0.2487	0.3314	0.4471	0.6098	0.6511	0.7213	1.0000	
BIT-CL	0780P181	0.1092	0.2283	0.2484	0.3315	0.4476	0.6096	0.6517	0.7208	1.0000	
PE-B2	0780P191	0.1092	0.2279	0.2488	0.3315	0.4472	0.6092	0.6510	0.7208	1.0000	
GRP-CL	0780P201	0.1080	0.2278	0.2487	0.3306	0.4471	0.6109	0.6503	0.7209	1.0000	
GRP-B1	0780P211	0.1095	0.2285	0.2486	0.3309	0.4469	0.6095	0.6505	0.7214	1.0000	
Mean		0.1090	0.2282	0.2485	0.3309	0.4470	0.6093	0.6505	0.7206	1.0000	
SD		0.0004	0.0003	0.0003	0.0004	0.0003	0.0005	0.0005	0.0004	0.0000	
%RSD		0.333%	0.13%	0.11%	0.13%	0.06%	0.09%	0.07%	0.05%	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
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		0.1090	0.2282	0.2486	0.3311	0.4472	0.6098	0.6509	0.7209	1.0000
SD		0.0007	0.0003	0.0002	0.0004	0.0003	0.0007	0.0006	0.0003	0.0000
%RSD		0.60%	0.14%	0.07%	0.14%	0.07%	0.12%	0.10%	0.04%	0.00%

GC-MS General Survey Results

Page 1 of 2

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	Diocetyl adipate	1146	NQ		Contaminant
2217	Unknown 57,71,43,85	618	NQ		Contaminant
2291	Unknown 57,43,71,85	1402	NQ		Contaminant
2300	Diocetylphthalate isomer	6364	NQ		Contaminant
2364	Unknown 57,43,71,85	2699	NQ		Contaminant
2378	d62-Squalene	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-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Page 1 of 1

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
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-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Page 1 of 1

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
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	Diethylphthalate isomer	3974	NQ		Contaminant
2363	Unknown 57,43,71,85	1052	NQ		Contaminant
2378	d62-Squalene	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-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Page 1 of 2

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-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Page 1 of 2

Project No: 8782-0
 Analyst: RK
 Sample Volume: 1 litre
 Final Extract Vol: 500 ul
 Sample Type: Bitumen chlorinated

Form No: 1307
 Data System Code: 0780P181
 Sample Code: BIT
 Associated Blank: Borehole

Date Sampled: Unknown
 Date Received: 09-Nov-94
 Date Analysed: 15-Nov-94

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-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey results

Page 1 of 2

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-butandioic 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-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Page 1 of 3

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-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Page 1 of 2

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-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalane

GC-MS General Survey Results

Page 1 of 2

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	Test material
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	Diocetyl 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-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-5-squalene

GC-MS General Survey Results

Page 1 of 2

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

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-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Page 1 of 2

Project No: 8782-0
 Analyst: RK
 Sample Volume: 1 litre
 Final Extract Vol: 500 ul
 Sample Type: GRP Chlorinated

Form No: 1307
 Data System Code: 0780P201
 Sample Code: GRP
 Associated Blank: Borehole

Date Sampled: Unknown
 Date Received: 09-Nov-94
 Date Analysed: 15-Nov-94

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 (7)	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-Naphthalene, BHT = d20-BHT, Hx = d34-hexadecane, Ph = d10-phenanthrene and Sq = d62-Squalene

GC-MS General Survey Results

Page 1 of 2

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

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-Naphthalene, 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 PER-
FORMANCE TESTING OF PROTOCOLS

WASTEWATER TREATMENT PLANT SURVEY ANALYSIS RESULTS

Project No: 8782-0
Form Number: 1347/1352
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)	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	
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	4876	14024	88095	
PB-TW-CL (2)	0030S201	6444	7862	2702	7097	2175	54553	6013	15490	85893	
Mean		6742	9533	3038	10170	3052	54467	4274	16345	85265	
SD		1955	1890	681	2934	695	6882	1125	2311	20579	
%RSD		29%	20%	22%	29%	23%	13%	26%	14%	24%	

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

Project No: 8782-0
Form Number: 1347/1352
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	
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		0.0836	0.1179	0.0398	0.1302	0.0388	0.6737	0.0519	0.2039	1.0000	
SD		0.0280	0.0351	0.0226	0.0621	0.0163	0.1733	0.0141	0.0613	0.0000	
%RSD		33%	30%	57%	48%	42%	26%	27%	30%	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%

Project No: 8782-0
Form Number: 1347/1352
Sample Volume: 1 litre
Final Extract Vol: 500 ul
Sample Type: Lab test leachate

0.901376147

Sample	Data sys Code	Retention Time/min									
		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	3.48	7.69	8.42	11.42	15.58	21.50	23.02	25.48	35.69	
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	22.99	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	25.45	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		3.47	7.68	8.42	11.43	15.59	21.50	23.01	25.48	35.69	
SD		0.017	0.023	0.023	0.018	0.024	0.023	0.022	0.023	0.022	
%RSD		0.50%	0.29%	0.27%	0.16%	0.15%	0.10%	0.10%	0.09%	0.06%	

Column test Results:

CCM Coltest	0780P011	3.45	7.69	8.42	11.44	15.63	21.55	23.06	25.53	35.74
CCM Coltest	0780P081	3.48	7.69	8.42	11.42	15.60	21.50	23.02	25.49	35.69
CCM Coltest	0780P221	3.45	7.66	8.39	11.39	15.58	21.49	23.00	25.48	35.66
CCM Coltest	0780P151	3.47	7.67	8.41	11.39	15.58	21.49	23.00	25.46	35.68
Mean		3.46	7.67	8.41	11.41	15.60	21.50	23.02	25.49	35.69
SD		0.014	0.014	0.014	0.023	0.021	0.028	0.028	0.026	0.032
%RSD		0.42%	0.19%	0.17%	0.20%	0.14%	0.13%	0.12%	0.10%	0.09%

GC-MS General Survey AQC results

Project No: 8782-0
 Form Number: 1347/1352
 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	0.0979	0.2156	0.2362	0.3202	0.4371	0.6027	0.6452	0.7141	1.0000	
PE-TW-CL (1)	0030S031	0.0983	0.2160	0.2370	0.3207	0.4375	0.6027	0.6452	0.7141	1.0000	
GRP-TW (1)	0030S041	0.0979	0.2161	0.2367	0.3208	0.4373	0.6030	0.6450	0.7144	1.0000	
GRP-TW-CL (1)	0030S051	0.0974	0.2160	0.2362	0.3207	0.4375	0.6027	0.6448	0.7141	1.0000	
BIT-TW (1)	0030S061	0.0975	0.2161	0.2363	0.3208	0.4377	0.6026	0.6450	0.7144	1.0000	
BIT-TW-CL (1)	0030S071	0.0975	0.2153	0.2359	0.3205	0.4371	0.6024	0.6449	0.7139	1.0000	
PB-TW (1)	0030S081	0.0976	0.2154	0.2356	0.3202	0.4372	0.6027	0.6447	0.7138	1.0000	
PB-TW-CL (1)	0030S101	0.0975	0.2153	0.2359	0.3201	0.4366	0.6024	0.6449	0.7139	1.0000	
PE-TW (2)	0030S121	0.0972	0.2151	0.2353	0.3204	0.4366	0.6025	0.6446	0.7141	1.0000	
PE-TW-CL (2)	0030S131	0.0971	0.2150	0.2356	0.3206	0.4368	0.6022	0.6447	0.7138	1.0000	
GRP-TW (2)	0030S141	0.0971	0.2150	0.2360	0.3202	0.4368	0.6022	0.6443	0.7138	1.0000	
GRP-TW-CL (2)	0030S161	0.0971	0.2150	0.2360	0.3202	0.4372	0.6022	0.6447	0.7138	1.0000	
BIT-TW (2)	0030S171	0.0968	0.2147	0.2353	0.3199	0.4366	0.6021	0.6448	0.7137	1.0000	
BIT-TW-CL (2)	0030S181	0.0968	0.2147	0.2353	0.3195	0.4362	0.6021	0.6446	0.7137	1.0000	
PB-TW (2)	0030S191	0.0968	0.2147	0.2353	0.3195	0.4362	0.6021	0.6446	0.7137	1.0000	
PB-TW-CL (2)	0030S201	0.0971	0.2150	0.2356	0.3198	0.4368	0.6024	0.6447	0.7138	1.0000	
Mean		0.0973	0.2153	0.2359	0.3203	0.4370	0.6024	0.6448	0.7139	1.0000	
SD		0.0004	0.0005	0.0005	0.0004	0.0005	0.0003	0.0002	0.0003	0.0000	
%RSD		0.44%	0.24%	0.21%	0.13%	0.11%	0.05%	0.04%	0.04%	0.00%	

Column test Results:

CCM Coltest	0030S011	0.0966	0.2151	0.2356	0.3201	0.4373	0.6029	0.6453	0.7143	1.0000
CCM Coltest	0030S081	0.0975	0.2153	0.2359	0.3201	0.4371	0.6024	0.6449	0.7143	1.0000
CCM Coltest	0030S151	0.0968	0.2147	0.2353	0.3195	0.4370	0.6025	0.6450	0.7145	1.0000
CCM Coltest	0030S221	0.0971	0.2150	0.2356	0.3194	0.4368	0.6022	0.6447	0.7138	1.0000
Mean		0.0970	0.2150	0.2356	0.3198	0.4371	0.6025	0.6450	0.7142	1.0000
SD		0.0004	0.0003	0.0003	0.0004	0.0002	0.0003	0.0003	0.0003	0.0000
%RSD		0.44%	0.13%	0.11%	0.12%	0.05%	0.05%	0.04%	0.04%	0.00%