Final Report

Study Title	(^{14}C)-8-2 Telomer B Alcohol: Adsorption/Desorption using a Batch Equilibrium Method
Data Requirement	Protocol designed to meet the known requirements of OECD Guideline 106 (January 2000)
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Covance Study Number	2304/001
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Report Issued	16 July 2004
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STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

(^{14}C)-8-2 Telomer B Alcohol: Adsorption/Desorption using a Batch Equilibrium Method

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA Sections § 10(d)(1)(A), (B) or (C).

Company Telomer Research Program

Company Agent: _____ Date: _____

These data are the property of the Telomer Research Program, and as such are considered to be confidential for all purposes other than compliance with FIFRA § 10. Submission of these data in compliance with FIFRA does not constitute a waiver of any right to confidentiality, which may exist under any statue or in any other country.

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

(^{14}C)-8-2 Telomer B Alcohol: Adsorption/Desorption using a Batch Equilibrium Method

The study was performed in accordance with the agreed protocol and with Covance Laboratories Limited, Standard Operating Procedures, unless otherwise stated, and the study objectives were achieved. The study was conducted in compliance with the United Kingdom Statutory Instrument No. 3106, The Good Laboratory Practice Regulations 1999, from 27th April 2004 as amended by the Good Laboratory Practice (Codification Amendments Etc.) Regulations 2004, and the OECD Principles on Good Laboratory Practice (revised 1997, Issued January 1998) ENV/MC/CHEM (98)17.

Studies performed in compliance with the GLP regulations and standards referenced above will be fully acceptable to the US EPA regulatory authorities.

S E Swales — 16 July 2004

S Swales
Study Director,
Covance Laboratories Ltd

Name — Date
Submitter
Department or Company

Name — Date
Submitter
Department or Company

Name — Date
Submitter
Department or Company

FLAGGING STATEMENTS

(^{14}C)-8-2 Telomer B Alcohol: Adsorption/Desorption using a Batch Equilibrium Method

STUDY DIRECTOR AUTHENTICATION AND GLP COMPLIANCE STATEMENT

(^{14}C)-8-2 Telomer B Alcohol: Adsorption/Desorption using a Batch Equilibrium Method

I, the undersigned, hereby declare that the work was performed by me or under my supervision and that the findings provide a true and accurate record of the results obtained.

The study was performed in accordance with the agreed protocol and with Covance Laboratories Limited Standard Operating Procedures, unless otherwise stated, and the study objectives were achieved. The study was conducted in accordance with the following international Codes of Good Laboratory Practice:

- United Kingdom Statutory Instrument 1999 No.3106 the Good Laboratory Practice Regulations 1999, from 27th April 2004 as amended by the Good Laboratory Practice (Codification Amendments Etc.) Regulations 2004
- The OECD Principles on Good Laboratory Practice (revised 1997, issued Jan 1998) ENV/MC/CHEM(98)17

Soils used for the study were sterilised by gamma irradiation at Isotron (Bradford, West Yorkshire). Isotron is not part of the UK GLP compliance-monitoring program.

S Swales

S Swales BSc MSc PhD
Study Director

16 July 2004
Date

QUALITY ASSURANCE STATEMENT

(^{14}C)-8-2 Telomer B Alcohol: Adsorption/Desorption using a Batch Equilibrium Method

This study has been reviewed by the Quality Assurance Unit of Covance Laboratories Ltd. and the report accurately reflects the raw data. The following inspections were conducted and findings reported to the Study Director (SD) and associated management.

Critical procedures which are performed routinely in an operational area, may be audited as part of a "process" inspection program. This can be in addition to phases scheduled on an individual study basis. Selected process inspections conducted and considered applicable to this study are included below.

Inspection Dates		Phase	Date Reported to SD and SD Management
From	To		
17 Mar 2003	17 Mar 2003	Protocol Review	17 Mar 2003
26 Mar 2003	26 Mar 2003	Protocol Amendment Review	26 Mar 2003
03 Apr 2003	03 Apr 2003	Test Article Receipt	03 Apr 2003
13 Aug 2003	13 Aug 2003	Protocol Amendment Review	13 Aug 2003
27 Nov 2003	27 Nov 2003	Protocol Amendment Review	27 Nov 2003
23 Dec 2003	23 Dec 2003	Dose Administration	23 Dec 2003
18 Feb 2004	09 Mar 2004	Draft Report and Data Review	09 Mar 2004

Process

Inspection Dates		Phase	Date Reported to SD and SD Management
From	To		
13 Mat 2003	13 May 2003	Data Processing	13 May 2003
30 May 2003	30 May 2003	High Performance Liquid Chromatography Analysis	30 May 2003
24 Jun 2003	24 Jun 2003	Sample Receipt	24 Jun 2003
30 Jun 2003	30 Jun 2003	Sample Preparation	30 Jun 2003

I Morrison
Section Head, Quality Assurance

16 July 2004
Date

RESPONSIBLE SCIENTIST'S STATEMENT

(^{14}C)-8-2 Telomer B Alcohol: Adsorption/Desorption using a Batch Equilibrium Method

I, the undersigned, hereby declare that I have reviewed this report in conjunction with the Study Director and that the interpretation and presentation of the data in the report are consistent with the results obtained.

A Hill BSc PhD
Director, Environmental Sciences

16 July 2024
Date

RESPONSIBLE PERSONNEL

(^{14}C)-8-2 Telomer B Alcohol: Adsorption/Desorption using a Batch Equilibrium Method

The following personnel were responsible for key elements of the study:

STUDY MANAGEMENT	NAME
Study Director	S Swales
Deputy Study Director	C Lewis
Scientific Reviewer	A Hill
Study Co-ordinator	C Unsworth

OPERATIONAL SUPERVISION	
Study Supervisor	K Dixon
Formulation	K Dixon
Chemical Analysis	K Dixon
Data Processing	K Dixon
In Life Supervisor	P Davies

QUALITY ASSURANCE	
Director, Quality Systems	C Clare

ARCHIVE STATEMENT

(^{14}C)-8-2 Telomer B Alcohol: Adsorption/Desorption using a Batch Equilibrium Method

All primary data, or authenticated copies thereof, samples and the final report will be retained in the Covance Laboratories Limited archives for one year after issue of the final report. At this time, the sponsor will be contacted to determine whether data should be returned, retained or destroyed on their behalf. Sponsors will be notified of the financial implications of each of these options at that time. All details supplied with the soils and all raw data generated by National Soil Resource Institute (NSRI) will be archived at CLE.

Samples requiring frozen storage are specifically excluded from the above. Samples requiring chromatographic analysis and adsorption/desorption solutions will be retained for as long as the quality of the material permits but for no longer than six months after issue of the draft report or three months after the issue of the final report, whichever is shorter. The study sponsor will be notified before such samples are destroyed on their behalf. All other samples may be disposed of in an ongoing manner.

CONTENTS

SUMMARY

The adsorption/desorption characteristics of (^{14}C)-8-2 TBA were examined in four soil types and one sediment. The study was conducted to the OECD Guideline 106 (January 2000).

Preliminary experiments were conducted in the dark at 20 ± 2°C. Equilibrium time determination and isotherm tests were conducted in the dark at 4 ± 1°C. The air-dried soils were pre-conditioned by mixing overnight with 0.01M calcium chloride.

Preliminary tests were performed to assess the solubility and adsorption to containers. These tests demonstrated that in a sealed vial with minimal headspace 8-2 TBA may not exist fully in solution over a concentration range of 0.01 - 0.1 µg/mL. Loss of radioactivity occurred when the vials were opened thereby confirming the volatile nature of 8-2 TBA. No definitive conclusions could be drawn from the preliminary solubility test due to the volatile nature of the compound. The amount of 8-2 TBA adsorbed to the test vessels from aqueous solution was ≤2% of the applied radioactivity. The concentrations used for the remainder of the study were selected such that they were below the stated water solubility (0.14 mg/L), but were high enough to ensure that the radioactivity could be detected throughout each stage of the study.

Three soils and a sediment from sites in the United Kingdom were used for the study. A fourth soil (Eurosoil 5) was obtained from the Institute for Reference Materials and Measurements in Belgium:

Soil name	pH (0.01M $CaCl_2$)	Organic carbon content (%)	Organic Matter (%)	Clay content (%)	Soil texture
SK961089	7.6	4.6	7.9	34	Clay loam
SK566696	4.2	0.8	1.4	11	Sandy loam
Emperor Son's Lake	6.0	1.2	2.1	17	Sandy loam
SK179618	5.6	3.8	6.6	20	Clay loam
Eurosoil 5	3.1	6.7	11.6	5	Loamy sand

Further preliminary tests demonstrated that:

- The percentage adsorption of 8-2 TBA to the test soils (not including Eurosoil 5) was >80% at ratios of soil to solution of 1:1 w/v and 1:5 w/v. For a soil:solution ratio of 1:25 the percentage adsorption of 8-2 TBA was >60% for the test soils. The recovery of applied radioactivity from the samples was dependent on the proportion of soil present with >90% being recovered at a soil:solution ratio of 1:1 w/v. The 1:1 ratio was, therefore, selected for the remainder of the study.

- 3 hours was a suitable adsorption and desorption equilibrium time for the study. The 8-2 TBA was not stable in the test system at 20°C and therefore, the isotherms test was conducted using sterile soil, sterile 0.01M calcium chloride and at a reduced temperature of 4 ± 1°C.

Five dose formulations of (^{14}C)-8-2 TBA were prepared at appropriate concentrations in sterile 0.01M calcium chloride. Aliquots (2.2, 5.2, 6.9 or 8.6 µL) of the appropriate formulations were added to the pre-conditioned sterile soils to obtain duplicate units at five concentrations, nominally 0.125, 0.1, 0.075, 0.05 and 0.0125 µg/mL.

Tests were performed in sealed glass vials with minimal headspace and aliquots were taken using a Hamilton SGE syringe (glass syringe with stainless steel needle and piston) inserted through the PTFE-lined rubber septum. All samples were mixed continuously for 3 hours on an end-over-end shaker at 4 ± 1°C. Each sample was then centrifuged to separate the soil and solution. The radioactivity in the supernatant was determined by assaying duplicate aliquots by LSC and the supernatant was removed.

The supernatant from each sample after the adsorption phase was replaced with an equivalent weighed portion of fresh 0.01M calcium chloride. The tubes were shaken to break up the compacted soil and were then placed back on the end-over-end shaker and mixed continuously for a further 3 hours for the desorption phase. Each sample was centrifuged to separate the soil and solution. The radioactivity in the supernatant was determined by assaying duplicate aliquots by LSC and the supernatant was removed.

The 0.125 µg/mL samples were extracted three times with acetone (10 mL) and the supernatants for each sample pooled. The soil extracts for each sample were quantified by LSC. The extracted soil samples (0.125 µg/mL) were air-dried then ground prior to combustion in oxygen.

The Freundlich adsorption constants (K_f) were calculated by linear regression analysis and the results were as follows:

Soil	Freundlich constant (K_f)
SK961089 (clay loam)	35
SK566696 (loamy sand)	40
Emperor Son's lake sediment (sandy loam)	20
SK 179618 (loam)	130
Eurosoil 5 (loamy sand)	38

The Freundlich adsorption constants related to organic carbon content (K_{oc}) as shown below:

Soil	(K_{oc})
SK961089 (clay loam)	765
SK566696 (loamy sand)	4941
Emperor Son's lake sediment (sandy loam)	1667
SK 179618 (loam)	3414
Eurosoil 5 (loamy sand)	572

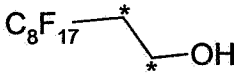
The adsorption and desorption isotherm showed a strong positive monotonic relationship between log C_e and log X/m, indicated by the correlation coefficients being close to 1. These values characterise 8-2 TBA as slightly mobile (K_{oc} 500-4000) to non-mobile (K_{oc}>4000) in the four soils and one sediment used in this study (Hollis, 1991)[a]

Mass balance for the four soils and one sediment were in the range 86 to 96%.

[a] Hollis, J M (1991). Mapping the vulnerability of aquifers and surface water to pesticide contamination at the national/regional scale. Pesticides in Soil and Water: Current Perspectives (A Walker Ed), BCPC Monograph 47, pp 169 to 174

INTRODUCTION

(^{14}C)-8-2 TBA (2-perfluorooctylethanol) used in this study had the following structure:



*Position of the ^{14}C radiolabel

The objective of the study was to:

- determine the adsorption/desorption characteristics of (^{14}C)-8-2 TBA in four soil types and one sediment.

The study was conducted to meet the requirements of the OECD Guideline 106 (January 2000).

The study was initiated on 11 March 2003 (date the protocol was signed by the Study Director) and completed on 16 July 2004 (date the final report was signed by the Study Director). The practical phase of the study was conducted at Covance Laboratories Europe (CLE), Otley Road, Harrogate, North Yorkshire between 4 April 2003 (test article quantification) and 8 January 2004 (combustion of residues).

MATERIALS AND METHODS

Protocol adherence

The study described in this report was carried out according to the agreed protocol and three amendments. Minor deviations that did not affect the integrity or outcome of the study are listed in Appendix 5.

Test article

The following physical information on the (^{14}C)-test substance is known:

Common Names:	8-2 Telomer B Alcohol 8-2 TBA
Chemical Name:	2-Perfluorooctylethanol [Ethanol-1,2-^{14}C]
CAS Name:	1-Decanol, 3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluoro-
CAS Number:	678-39-7
Lot Number:	3489-169
Radiochemical Purity:	98.6% (HPLC)
Specific Activity:	33 mCi/mmole
Molecular Weight:	464.12 g/mole
Chemical Formula:	$C_8F_{17}{}^{14}CH_2{}^{14}CH_2OH$
Solubility:	*ca* 140 µg/L
Storage Conditions:	frozen (<-10 °C) in the dark
Vapor Pressure:	0.023 mmHg

The radiolabelled test article was supplied by Perkin Elmer Life Sciences and was received at Covance on 3 April 2003. It was stored at <-10°C in the dark.

The specific activity was not re-determined at Covance but the value stated above was used, where applicable. A Certificate of Analysis for the radiolabelled test article was provided by the supplier (Appendix 1).

Non-radiolabelled test article

Non-radiolabelled test article was supplied by the sponsor and was received at Covance on 27 February 2003. It was stored at room temperature in the dark. The following information was supplied:

Name:	8-2 Telomer B alcohol
Batch number:	Haskell 24691
Quantity received:	5.6 g

A Certificate of Analysis for the non-radiolabelled test article was provided by the sponsor (Appendix 1).

Solvents and reagents

Reagents and solvents were of Analytical grade (or a suitable alternative) and were obtained principally from VWR International Limited (formerly Merck, BDH) (Poole, Dorset) or Rathburn Chemicals Limited (Walkerburn, Peebleshire).

Starscint™, obtained from Perkin Elmer Life Sciences Ltd (Pangbourne, Berkshire) was the liquid scintillant used for general LSC. Quickszint Flow 302 (Zinsser Analytic, Maidenhead, Berkshire) was the liquid scintillant used in HPLC liquid mixing cells.

Permafluor® E^+ and Carbo-sorb® E, obtained from Perkin Elmer Life Sciences Ltd were mixed (2:1 v/v) to form the scintillation cocktail used in the sample oxidiser.

Test System

Three soils (SK961089, SK566696 and SK179618) and one sediment (Emperor Son's Lake) from sites in the United Kingdom, and one soil (Eurosoil 5) from Belgium were used for the study. The soils were supplied by Land Research Associates (Derby, Derbyshire) and the Institute for Reference Materials and Measurements (Belgium).

On arrival at Covance the test soils and sediment were thoroughly mixed and passed through a 2 mm mesh sieve. The soils were then air-dried and stored in the dark at room temperature. Information on the sampling site, sampling method and pesticide history was supplied with the soils[a].

The UK soils were characterised for the following parameters by Covance:

- pH (0.01 M $CaCl_2$)
- organic carbon content
- organic matter content
- particle size distribution (UK/BBA/USDA)
- textural class (UK, BBA and USDA)

[a] Information on the sampling site, sampling method and pesticide history was not supplied with Eurosoil 5 and, therefore, this information has not been reported.

- cation exchange capacity (mmol/kg)
- moisture holding capacity at pF 0 (maximum water holding capacity, MWHC) and pF 2.5 (33 kPa)
- C/N ratio

Nitrogen content of the soils was determined by The National Soil Resource Institute, formerly known as the Soil Survey and Land Research Centre (Silsoe, Bedford) under their own study number and protocol.

Certificates of Analysis were produced for each soil. These are shown in Appendix 2.

Soil Eurosoil 5 was supplied with a certificate of analysis detailing the pH in water and pH in calcium chloride ($CaCl_2$). Eurosoil 5 also had an unaudited certificate of analysis detailing:

- organic carbon content
- organic matter content
- particle size distribution (UK/BBA/USDA)
- textural class (UK/BBA/USDA)
- cation exchange capacity (mmol/kg)

The soil characteristics are summarised in the following table:

Soil name	pH (0.01M $CaCl_2$)	Organic carbon Content (%)	Clay content (%)	Soil texture (USDA)
SK961089	7.6	4.6	34	Clay loam
SK566696	4.2	0.8	11	Loamy sand
Emperor Son's Lake	6.0	1.2	17	Sandy loam
SK179618	5.6	3.8	20	Loam
Eurosoil 5*	3.1	6.7	5	Loamy sand

* Eurosoil 5 included after initial equilibrium time test, due to stability problems of (^{14}C)-8,2 TBA with SK179618 soil.

All future references to soil, for the purpose of this report, include soil and sediment.

Prior to dispensing, the moisture contents of the stored soils were determined at Covance by drying a portion at *ca* 105°C. This allowed the dry weight equivalent of

the dispensed soil to be calculated from the wet weight. All calculations were based upon soil dry weight.

All experiments were conducted in the dark at either 20 ± 2°C or 4 ± 1°C.

Soils were dispensed into pre-weighed centrifuge tubes and pre-conditioned by mixing with either 0.01M calcium chloride solution or sterile 0.01M calcium chloride solution overnight before the day of the experiment.

Soils and solutions were mixed mechanically using a Stuart Scientific Rotator Drive STR4 end-over-end shaker at a speed that ensured efficient mixing.

Centrifugation was performed in an IEC Centra GP8R centrifuge at 2000 rpm (*ca* 700 g) for a length of time calculated to be sufficient to remove particles larger than 0.2 µm from the portion of the solution from which aliquots were taken.

Test article application

The radiochemical purity of (^{14}C)-8-2 TBA was assessed by high performance liquid chromatography (HPLC, page 27) and by thin layer chromatography (TLC, page 27) after receipt at Covance and prior to use.

A stock solution (stock solution 1) was prepared by dissolving one ampoule containing (^{14}C)-8-2 TBA (approx. 4.22 mg) in ethanol (6 mL) to produce a stock solution at a concentration of 0.679 mg/mL (by assay). The radiochemical purity of (^{14}C)-8-2 TBA in stock solution 1 (determined 4 April 2003) was >98% by HPLC and >96% by TLC (Appendix 3).

Stock solution 2 was prepared by removing an aliquot (500 µL) from stock solution 1 and adding this to ethanol (4.5 mL). The concentration of stock solution 2 (determined by assay) was 0.07 mg/mL.

Stock solution 1 was used for the preparation of dose formulations for the equilibrium time determination and isotherms tests. Stock solution 2 was used for the solubility and soil:solution ratio tests.

Dose formulations for the isotherms test were prepared through the addition of appropriate amounts of radiolabelled test article, as detailed below. The specific activities of the dose formulations were calculated and the concentration of 8-2 TBA

in each formulation prior to dosing was checked by removing triplicate aliquots for LSC.

Test	Formulation number	Volume of stock solution (mL)	Radiolabelled 8-2 TBA (µg)	Solvent volume (mL)	Specific activity (MBq/mg)
SSR	SSR	0.357[2]	25	0.643	2.63
EQT	2	0.147[1]	100	0.853	2.63
EQT-Rpt	3	0.191[1]	130	1.109	2.63
Isotherms	4	0.130[1]	88	0.570	2.63
Isotherms	5	0.045[1]	30	0.555	2.63

[1] stock solution 1, [2] stock solution 2
SSR = soil:solution ratio
EQT = equilibrium time
EQT-Rpt = repeat equilibrium time

Tests were initiated by the addition of an appropriate amount of (^{14}C)-8-2 TBA dose formulation. Application of test article complied with OECD Guideline 106 (January 2000).

Due to the low level of radioactivity in the individual formulations, the radiochemical purity of (^{14}C)-8-2 TBA in stock solution 1 was assessed by HPLC and TLC immediately prior to preparing the individual dose formulations. The radiochemical purity was >98% by HPLC and >96% by TLC.

Solubility test and adsorption to vessels

Tests were performed in sealed glass vials with minimal headspace after a series of initial tests indicated that there was potentially a problem with the volatile nature of this compound. Aliquots for liquid scintillation counting (LSC) were removed using a Hamilton SGE syringe (glass syringe with stainless steel needle and piston) that was inserted through the PTFE-lined rubber septum to minimise any volatile losses. A control vial containing ethanol only was used to confirm that the dosing procedure was acceptable. The test was performed over the concentration range 0.01 µg/mL to 0.1 µg/mL.

Solubility tests were inconclusive due to the volatile nature of the compound. The volatile nature of 8-2 TBA was considered less of a problem in the presence of soil. Therefore, for the remainder of the study the concentrations used were selected such that they were below the stated water solubility of the compound (0.14 mg/L), but

were high enough to ensure that the radioactivity could be detected throughout each stage of the study.

Ratio of soil to aqueous phase

Tests were performed in sealed glass vials with minimal headspace. All soils (SK961089, SK566696, Emperor Son's Lake and SK179618) were pre-equilibrated overnight with 0.01M calcium chloride prior to dose application. The test article (dissolved in ethanol) was applied to each of the test units by a syringe inserted through the PTFE-lined rubber septum. The test article was applied such that the target concentration in solution was 0.025 µg/mL. The sample vials were then placed on an end-over-end shaker set at a speed sufficient to keep the soils in suspension. After 24 hours the samples were removed from the shaker and centrifuged (*ca* 1700 g for 10 min). Aliquots for LSC were removed using a Hamilton SGE syringe that was inserted through the PTFE-lined rubber septum to minimise any volatile losses. The centrifuge speed (*ca* 1700 g) resulted in the loss of a number of the glass vials, therefore subsequent centrifugation was performed at *ca* 700 g.

Once the aliquots for LSC had been taken the lids were removed from the sample vials, the supernatant removed and acetone added (10 mL). All samples were shaken then centrifuged and the supernatant removed, the process was repeated a further two times and the supernatants for each sample pooled. The soil extracts for each sample were quantified by LSC.

The extracted soil samples were air-dried overnight then ground in a pestle and mortar prior to combustion in oxygen.

The PTFE-lined lids were soaked in acetone prior to quantification by LSC.

The following weights of soil and volumes of 0.01M calcium chloride were used for the test:

1:1 Ratio. 9 g of soil to 9 mL calcium chloride solution

1:5 Ratio. 3 g of soil to 15 mL calcium chloride solution

1:25 Ratio. 0.6 g of soil to 15 mL of calcium chloride solution.

The percentage adsorption of 8-2 TBA to the test soils (not including Eurosoil 5) was >80% at ratios of soil to solution of 1:1 w/v and 1:5 w/v. For a soil:solution ratio of

1:25 the percentage adsorption of 8-2 TBA was >60% for the test soils. The recovery of applied radioactivity from the samples was dependent on the proportion of soil present with >90% being recovered at a soil:solution ratio of 1:1 w/v. The 1:1 ratio was, therefore, selected for the remainder of the study.

Equilibrium time determination and stability

The adsorption equilibrium time was determined by preparation of samples of 8-2 TBA (0.1 µg/mL) in 0.01M calcium chloride (9 mL) with 9 g soil (SK961089, SK566696, Emperor Son's Lake and SK179618). The sample vials were placed on an end-over-end shaker set at a speed sufficient to keep the soils in suspension. At intervals of 3, 6, 24 and 48 hours, duplicate samples of each soil type were removed from the shaker and centrifuged. Aliquots for LSC were removed using a Hamilton SGE syringe that was inserted through the PTFE-lined rubber septum to minimise volatile losses. Once the aliquots for LSC had been taken the lids were removed and the supernatant removed. The 24 hour samples were extracted three times with acetone (10 mL) and the supernatants for each sample pooled. The soil extracts for each sample were quantified by LSC and analysed by TLC.

The extracted soil samples were air-dried overnight then ground in a pestle and mortar prior to combustion in oxygen.

It was determined that 3 hours was a suitable adsorption equilibrium time. However, degradation of the 8-2 TBA had occurred over the 24 hour equilibration period.

The equilibrium time was established as 3 hours but the stability of the 8-2 TBA was unknown over this period of time, so further testing was carried out as detailed below.

Duplicate samples of 8-2 TBA (0.1 µg/mL) were prepared in 0.01M calcium chloride (9 mL) with 9 g soil (SK961089, SK566696, Emperor Son's Lake and SK179618). The sample vials were placed on an end-over-end shaker (set at a speed sufficient to keep the soils in suspension) in an incubator set at 4 ± 1°C. After 3 hours, duplicate samples of each soil type were removed from the shaker and centrifuged. Aliquots for LSC were removed using a Hamilton SGE syringe that was inserted through the PTFE-lined rubber septum to minimise volatile losses. Once the aliquots for LSC had been taken the lids were removed and the supernatant removed. The samples were extracted three times with acetone (10 mL) and the supernatants for each sample pooled. The soil extracts for each sample were quantified by LSC and analysed by TLC.

The extracted soil samples were air-dried overnight then ground in a pestle and mortar prior to combustion in oxygen.

The level of radioactivity remaining in the supernatants after 3 hours at 4°C was similar to that previously obtained at 20°C (i.e. <3% of applied radioactivity). This indicated that equilibrium had been established at 3 hours. The stability was checked for all soils and, with the exception of SK179618 soil, (^{14}C)-8-2 TBA was the only component present in the soil extracts.

Due to the ongoing stability problem with SK179618 soil, an additional soil (Eurosoil 5) was obtained. The previous test was repeated using both SK179618 soil and Eurosoil 5. The soils were sterilised by gamma irradiation (Isotron, Bradford, West Yorkshire) and the test was conducted at 4 ± 1°C using sterile 0.01M calcium chloride solution. Irradiation of the soils was performed to minimise the microbial degradation of the test substance during the test. 0.01M calcium chloride was sterilised by autoclaving.

Duplicate samples of 8-2 TBA (0.1 µg/mL) were prepared in sterile 0.01M calcium chloride (9 mL) with 9 g sterile soil (SK179618 and Eurosoil 5). The sample vials were placed on an end-over-end shaker (set at a speed sufficient to keep the soils in suspension) in an incubator set at 4 ± 1°C. After 3 hours, duplicate samples of each soil type were removed from the shaker and centrifuged. Aliquots for LSC were removed using a Hamilton SGE syringe that was inserted through the PTFE-lined rubber septum to minimise volatile losses. Once the aliquots for LSC had been taken the lids were removed and the supernatant removed. The samples were extracted three times with acetone (10 mL) and the supernatants for each sample pooled. The soil extracts for each sample were quantified by LSC and analysed by TLC.

The level of radioactivity remaining in the supernatants after 3 hours at 4°C was similar to that previously obtained for SK179618 (i.e. <3% of applied radioactivity). This indicated that equilibrium had been established at 3 hours. The stability was checked for both soils and (^{14}C)-8-2 TBA was the only component present in the soil extracts.

Under sterile conditions the results for SK179618 soil and Eurosoil 5 confirmed that the (^{14}C)-8-2 TBA was stable over the 3 hour equilibrium time at 4 ± 1°C.

The results of the adsorption equilibrium time led to the following changes being made to the study design:

- inclusion of an additional soil (Eurosoil 5)
- all five soils were sterilised by gamma irradiation prior to use
- 0.01M calcium chloride solutions were sterilised by autoclaving prior to use
- the temperature at which the test was performed was reduced from 20 ± 2°C to 4 ± 1°C.

The desorption equilibrium time for each soil type was determined by preparation of samples of 8-2 TBA (0.1 µg/mL) in sterile 0.01M calcium chloride (9 mL) with 9 g sterile soil (all five soils).

After mixing for the adsorption equilibrium time of 3 hours, samples were centrifuged and the supernatants removed. The supernatants were replaced by equal weights of fresh sterile 0.01M calcium chloride and the samples were placed back on the shaker.

At intervals of 1.5, 3, 6 and 24 hours, duplicate samples of each soil type were removed from the shaker and centrifuged. Aliquots for LSC were removed using a Hamilton SGE syringe that was inserted through the PTFE-lined rubber septum to minimise volatile losses.

It was determined that 3 hours was a suitable desorption equilibrium time.

Adsorption isotherms

Duplicate samples at five concentrations of 8-2 TBA in sterile 0.01M calcium chloride (9 mL) with 9 g sterile soil were prepared. The table below details how the test article formulations were used to achieve the initial concentration of 8-2 TBA in the units:

Formulation	Formulation conc.[1] (mg/mL)	Volume formulation (µL)	Weight of 8-2 TBA (µg)	Volume of 0.01 $MCaCl_2$ (mL)	Nominal conc.[1] (µg/mL)	Actual conc.[1] (µg/mL)
4[2]	0.131	8.6	1.127	9	0.125	0.125
4[2]	0.131	6.9	0.904	9	0.1	0.1
4[2]	0.131	5.2	0.681	9	0.075	0.076
5[2]	0.053	8.6	0.456	9	0.05	0.05
5[2]	0.053	2.2	0.117	9	0.0125	0.0129

[1] concentration
[2] refer to table on page 21 for preparation of the formulations.

The sample vials were placed on an end-over-end shaker in an incubator set at 4 ± 1°C. After mixing for the adsorption equilibrium time of 3 hours, the phases were separated by centrifugation. Aliquots (*ca* 1 mL or 1.5 mL) for LSC were removed using a Hamilton SGE syringe that was inserted through the PTFE-lined rubber

septum to minimise potential volatile losses. Once the aliquots for LSC had been taken, as much of the adsorption supernatant as possible was removed.

Desorption isotherms

The volume of adsorption supernatant removed was replaced by an equivalent weighed portion of fresh 0.01M calcium chloride. The samples were shaken vigorously to break up the soil packed at the bottom of the tube. The samples were then re-mixed for the desorption equilibrium time of 3 hours at 4 ± 1°C. After this time, each sample was centrifuged and the supernatant quantified by LSC.

The 0.125 µg/mL samples were extracted three times with acetone (10 mL) and the supernatants for each sample pooled. The soil extract for each sample was quantified by LSC. The extracted soil samples (0.125 µg/mL) were air-dried then ground in a pestle and mortar prior to combustion in oxygen and quantification by LSC.

The PTFE-lined lids used for each sample were soaked in acetone prior to quantification by LSC.

Mass balance

For each of the samples, the mass balance was determined from the sum of the radioactivity present in the adsorption supernatant, the desorption supernatant, the soil extract, the lid wash and the combusted residue.

pH measurements

The pH of 0.01 M calcium chloride containing the highest (0.125 µg/mL) and lowest (0.0125 µg/mL) concentrations of 8-2 TBA was measured. The pH of each adsorption supernatant from the adsorption isotherm test was also measured.

Analytical Methods and Measurements

HPLC

Radiochemical purity determinations were performed using the following high performance liquid chromatography (HPLC) method:

Column: Genesis C_{18}, 150 x 4.6 mm, 4µm

Gradient: A linear binary gradient system was used consisting of
Solvent A: 2mM ammonium acetate in water
Solvent B: Methanol

Time	%A	%B
0	97	3
15	0	100
35	0	100
35.1	97	3
40	97	3

Flow rate: 1 mL/min

Detection: UV absorbance of the eluent was monitored at 215 nm using an Agilent G1314A UV detector or a Jasco UV-975 detector. Radioactivity in the eluent was monitored using a β-ram flow through radioactivity monitor (LabLogic, Sheffield, South Yorkshire) equipped with a liquid mixing (500 µL) cell.

Samples were co-injected with non-radiolabelled 8-2 TBA. Chromatograms were evaluated using Laura (version 1.4a) software (LabLogic).

TLC

The following TLC system was used for determination of radiochemical purity and analysis of selected sample extracts:

Plates: Whatman silica gel K6F
Solvent: Toluene : Ethyl acetate (1:1)

Following chromatography, non-radiolabelled 8-2 TBA was visualised by spraying the plate with a 2% potassium permanganate solution. (^{14}C)-8-2 TBA was detected by preparation of a radioluminogram of the TLC plate using a Fuji BAS 1500 Bio-image analyser. Chromatograms were evaluated from the radioluminograms using the associated Tina software (version 2.09g).

Radioactivity

Radioactivity was measured using a Packard Tricarb model 1900TR liquid scintillation counter with facilities for computing quench corrected disintegrations per minute (dpm). Efficiency correlation curves were routinely checked by the use of commercially available quenched standards (Perkin Elmer) quenched over the range observed in test samples. The limit of detection was taken as 1.5 times the background

radioactivity, determined by counting vials containing scintillant only in the same batch as the samples.

The radioactivity in liquid samples was determined by calculating the total sample weight and assaying duplicate weighed aliquots by LSC, after mixing with liquid scintillant (*ca* 5 mL). The density of calcium chloride solutions and supernatants was assumed to be 1.00 g/mL so concentrations per g are equivalent to concentrations per mL.

The radioactivity in solid residues was determined, after air-drying and grinding using a pestle and mortar, by combustion of triplicate weighed aliquots in oxygen using a Harvey OX-500 biological oxidiser. The combusted products were absorbed in scintillation cocktail and the radioactivity absorbed assayed by LSC. Carbon-14 standards were combusted at the beginning and at regular intervals throughout the day to check the carry-over between samples and the efficiency of the combustion process. Combustion efficiencies were 99 ± 4% and therefore no correction factors were applied to the data.

Calculations

Definitions

Mass of test article available for adsorption (µg) = G

Dry weight of soil (g) = m

Volume of solution associated with soil (mL) = V_0

Concentration of test article in adsorption supernatant (µg/mL) = C_e

Concentration of test article in desorption supernatant (µg/mL) = C_1

Density of 0.01 M calcium chloride solutions and supernatants (g/mL) = ρ

Adsorption

Weight of stock solution initially added (g) = W_1

Volume of stock solution initially added (mL) = $V_1 = W_1 / \rho$

Weight of supernatant after adsorption step (g) = W_2

Volume of supernatant after adsorption step (mL) = $V_2 = W_2 / \rho$

Quantity of test substance adsorbed (µg) = $X = G - (C_e \times (V_0 + V_1))$

Percent of test substance adsorbed = $A = X \times 100 / G$

Desorption

Weight of supernatant after desorption step (g) = W_3

Volume of supernatant after desorption step (mL) = $V_3 = W_3 / \rho$

Quantity of test substance adsorbed following desorption (µg)
$= X_1 = X + (C_e \times (V_0 + V_1 - V_2)) - (C_1 \times (V_0 + V_1 - V_2 + V_3))$

Percent of adsorbed compound desorbed in desorption step = $(X - X_1) \times 100 / X$

Adsorption coefficients

The following equations were used to calculate adsorption coefficients (mL/g) for individual units:

Adsorption coefficient = $K_d = (X/m) / C_e$

Adsorption coefficient as a function of the organic carbon content of the soil
$= K_{doc} = K_d \times 100 /$ % organic carbon

Adsorption coefficient as a function of the organic matter content of the soil
$= K_{dom} = K_d \times 100 /$ % organic matter

Adsorption constants

The Freundlich constants, K_f, and slope of Freundlich isotherm, 1/n, for each adsorption and desorption isotherm were calculated from the following relationship:

$X/m = K \times C^{1/n}$

Where X/m is the concentration of test article in the soil and C is the concentration of test article in the supernatant.

Taking Logs of each side gives:

Log (X/m) = Log (K_f) + (1/n) Log (C)

Hence an isotherm of Log (X/m) against Log (C) was plotted for each adsorption and desorption phase. The intercept and the slope of the line of best fit gave Log (K) and (1/n) respectively.

The Freundlich adsorption constant was calculated as a function of the organic carbon content and organic matter content of the soil to give the values for K_{oc} and K_{om} respectively by using the following equations:

K_{oc} = K x 100 / % organic carbon

K_{om} = K x 100 / % organic matter

Determination of percent of applied radioactivity in samples

The individual estimates of concentration derived from the duplicate or triplicate aliquots were averaged to provide a mean concentration of radioactivity in the sample.

Total radioactivity applied to incubation unit (kBq) = T

Initial sample weight (g) = I

Sub-sample weight (g) = SS

Total weight of sample for LSC assay (g) = A

Weight of aliquots assayed by LSC (g) = W

Background corrected radioactivity in aliquots assayed by LSC (dpm) = D

Concentration of radioactivity in sample (dpm/g) = C = D / W

Radioactivity in assayed sample (kBq) = B = C x A / 60,000
(1 kBq = 60,000 dpm)

Percent of applied radioactivity in sample = (B / T) x (I / SS) x 100

Expression of Results

Tables presented in the report have been computer generated and data values displayed may be rounded values of those held in memory. As a result of this, calculation of mean and total values from individual data presented in the report will, in some instances, yield a minor variation to the value shown. Calculations were performed using Microsoft Excel software.

Volumes referred to in the methods section are approximate volumes only. When samples were quantified for radioactive content, liquid samples were accurately weighed and accurately weighed aliquots were removed for LSC.

RESULTS

Preliminary tests

Solubility and Adsorption to containers

The table below shows the results of the test solutions before and after centrifugation.

Test concentration	Dose applied kBq	Radioactivity in solution kBq	% ARR in solution	Radioactivity in solution after centrifugation kBq	% ARR in solution after centrifugation
0.1 µg/mL	5.166	2.933	56.8	2.293	44.5
0.05 µg/mL	2.583	2.031	78.6	1.502	58.1
0.025 µg/mL	1.291	0.999	77.4	0.765	59.3
0.01 µg/mL	0.553	0.467	84.4	0.351	63.5
0.1 µg/mL[1]	5.166	5.206	100.8	5.180	100.3

[1] Control vial containing ethanol only

The results show that in a sealed vial with minimal headspace, the test article may not exist fully in solution. This may account for the loss of radioactivity after centrifugation. However, this loss may also be attributed to volatilization of the test article because when the vial lids were removed, there was a further loss of radioactivity, confirming that the test article was volatile. The table below shows the results of the test solutions post-centrifugation and after the removal of the vial lid.

Test concentration	Dose applied kBq	Radioactivity in solution after removal of lid kBq	% ARR in solution	Radioactivity in methanol wash of vial kBq	% ARR in unit wash
0.1 µg/mL	5.166	1.838	35.6	0.1051	2.03
0.05 µg/mL	2.583	1.164	45.1	0.0258	1.00
0.025 µg/mL	1.291	0.600	46.5	0.0214	1.66
0.01 µg/mL	0.553	0.345	62.4	0.0081	1.46
0.1 µg/mL[1]	5.166	4.848	93.8	0.0244	0.47

[1] Control vial containing ethanol only

Solubility tests were inconclusive due to the volatile nature of the compound. Tests showed that there was not much adsorption of the test material to the glass vial.

Ratio of soil to aqueous phase

The percentage adsorption of 8-2 TBA to the test soils was >80% at ratios of soil to solution of 1:1 w/v and 1:5 w/v. For a soil:solution ratio of 1:25 the percentage adsorption of 8-2 TBA was >60% for the test soils. The recovery of applied radioactivity from the samples was dependent on the proportion of soil present with >90% being recovered at a soil:solution ratio of 1:1 w/v. Results are shown in Table 1 to Table 3. The 1:1 ratio was selected for the remainder of the study.

Equilibrium time determination

Results from the equilibrium time determination showed that for all four soils with a soil : aqueous phase ratio of 1:1, 8-2 TBA at a concentration of 0.1 µg/mL in 0.01M calcium chloride had reached adsorption and desorption equilibriums by 3 hours. After this time, the percent change in the quantity of 8-2 TBA in the supernatant was <10%. Results are shown in Figure 1 to Figure 4.

Results from the stability test (conducted using the 24 hour samples only) showed that the overall recovery of applied radioactivity extracted with acetone was 83% to 88% for soils SK961089, SK566696 and Emperor Son's Lake, but only 39% for soil SK179618. Thin layer chromatography of the soil extracts showed that the purity of 8-2 TBA was 72% to 87% in extracts from SK961089, SK566696 and Emperor Son's Lake soils. The test article was degraded to a greater extent in soil SK179618 with ≤ 5% of the applied radioactivity being present as 8-2 TBA in the acetone extract of the soil. Results are presented in Table 4 and Table 5. TLC chromatograms of the soil extracts are shown in Figure 5 to Figure 8.

A repeat test was performed by shaking duplicate samples for 3 hours, the proposed equilibrium time, at a reduced temperature of 4 ± 1°C. The level of radioactivity remaining in the supernatants after 3 hours was similar to that previously obtained at 20°C (i.e. <3% of applied radioactivity). This, therefore, demonstrated that equilibrium had been established at 3 hours. The stability was checked for all soils and, with the exception of SK179618 soil, (^{14}C)-8-2 TBA was the only component present in the soil extracts. Results are presented in Table 6 and Table 7. TLC chromatograms of the soil extracts are shown in Figure 9 to Figure 12.

Due to the ongoing stability problem with soil SK179618, an additional soil (Eurosoil 5) was obtained. The previous test was repeated using both SK179618 soil and Eurosoil 5 under sterile conditions and at 4 ± 1°C.

The level of radioactivity remaining in the supernatants after 3 hours at 4°C was again similar to that previously obtained for SK179618 (i.e. ≤3% of applied

radioactivity). This indicated that equilibrium had been established at 3 hours. The stability was checked for both soils and (^{14}C)-8-2 TBA was the only component present in the soil extracts. The results are shown in Table 8, Figure 13 and Figure 14.

Under sterile conditions the results for SK179618 soil and Eurosoil 5 confirm that the (^{14}C)-8-2 TBA was stable over the 3 hour equilibrium time at 4 ± 1°C.

The determination of the desorption equilibrium time demonstrated that 1% to 2% of the adsorbed radioactivity was desorbed from the soils at 1.5, 3, 6 and 24 hours. Therefore, the adsorbed 8-2 TBA is not desorbed from the soils even after 24 h. Therefore, 3 hours was a selected as the desorption equilibrium time. The results are shown in Table 9.

Adsorption and desorption isotherms

Values for the concentrations of 8-2 TBA in solution and on the soil at the adsorption and desorption equilibrium times are presented in Table 10 to Table 14.

Quantities of 8-2 TBA adsorbed to and desorbed from soil are presented in Table 15 to Table 19.

Adsorption coefficients (K_d, K_{doc} and K_{dom}) determined at each concentration for the soil used for the adsorption/desorption test are presented in Table 20 to Table 24.

Freundlich adsorption and desorption constants (K_f, K_{oc} and K_{om}) are presented in Table 25 to Table 29.

The Freundlich adsorption constants (K_f) calculated by linear regression analysis were 35, 40, 20, 130 and 38 for the SK961089, SK566696, Emperor Son's Lake, SK179618 and Eurosoil 5 respectively.

The Freundlich adsorption constants related to organic carbon content (K_{oc}) were 765, 4941, 1667, 3414 and 572 for the SK961089, SK566696, Emperor Son's Lake, SK179618 and Eurosoil 5 respectively.

The adsorption and desorption isotherms, presented in Figure 15 to Figure 19, showed a strong positive monotonic relationship between log C_e and log X/m, indicated by the correlation coefficients being close to 1.

Mass balance results are presented in Table 30. Mean recoveries of applied radioactivity were 86 to 96%.

The pH values of 0.01M calcium chloride solutions containing the highest (0.125 µg/mL) and lowest (0.0125 µg/mL) concentrations of 8-2 TBA are presented in Table 31. The pH values of all the adsorption supernatants are presented in Table 32.

SUMMARY AND CONCLUSIONS

The Freundlich adsorption constants (K_f) were calculated by linear regression analysis and the results were as follows:

Soil	Freundlich constant (K_f)
SK961089 (clay loam)	35
SK566696 (loamy sand)	40
Emperor Son's Lake sediment (sandy loam)	20
SK 179618 (loam)	130
Eurosoil 5 (loamy sand)	38

The Freundlich adsorption constants related to organic carbon content (K_{oc}) as shown below:

Soil	(K_{oc})
SK961089 (clay loam)	765
SK566696 (loamy sand)	4941
Emperor Son's Lake sediment (sandy loam)	1667
SK 179618 (loam)	3414
Eurosoil 5 (loamy sand)	572

These values characterise 8-2 TBA as slightly mobile (K_{oc} 500 to 4000) to non-mobile (K_{oc}>4000) in the four soils and one sediment used in this study (Hollis, 1991)[a]

[a] Hollis, J M (1991). Mapping the vulnerability of aquifers and surface water to pesticide contamination at the national/regional scale. Pesticides in Soil and Water: Current Perspectives (A Walker Ed), BCPC Monograph 47, pp 169 to 174

TABLES

Table 1

1:1 Ratio of soil to aqueous phase following treatment at 0.025 µg/mL

		% of applied radioactivity				
Unit	Soil type	Supernatant	Soil extract	Residue	Lid Wash	Total
A2	SK961089	1.2	74.6	18.1	3.9	97.8
B2	SK566696	1.5	86.6	5.6	6.1	99.8
C2	Emperor Son's Lake	1.1	80.4	6.0	10.6	98.1
D1	SK179618	2.5	39.1	51.3	1.4	94.3

Ratio Test not performed for Eurosoil 5.

Table 2

1:5 Ratio of soil to aqueous phase following treatment at 0.025 µg/mL

		% of applied radioactivity				
Unit	Soil type	Supernatant	Soil extract	Residue	Lid Wash	Total
A3	SK961089	1.6	71.5	19.5	2.6	95.2
A4	SK961089	1.6	71.3	19.4	3.3	95.6
B3	SK566696	2.6	76.0	4.8	4.8	88.2
B4	SK566696	2.5	77.7	4.1	5.5	89.8
C3	Emperor Son's Lake	2.6	78.9	4.0	9.6	95.1
D3	SK179618	6.3	35.9	45.4	1.3	88.9
D4	SK179618	5.8	37.6	43.7	1.9	89.0

Ratio Test not performed for Eurosoil 5.

Table 3

1:25 Ratio of soil to aqueous phase following treatment at 0.025 µg/mL

		% of applied radioactivity				
Unit	Soil type	Supernatant	Soil extract	Residue	Lid Wash	Total
A5	SK961089	4.8	61.1	20.8	7.9	94.6
A6	SK961089	5.1	59.9	19.4	7.7	92.1
B5	SK566696	8	67.0	5.2	9.3	89.5
C5	Emperor Son's Lake	10	60.8	3.4	12.0	86.2
C6	Emperor Son's Lake	11.3	56.6	3.8	15.8	87.5
D5	SK179618	8.4	30.0	45.1	2.9	86.4

Ratio Test not performed for Eurosoil 5.

Table 4

Recovery of applied radioactivity from the 24 hour equilibrium time samples at a soil to aqueous phase ratio of 1:1 and 20 ± 1°C

Soil	Sample	Percent of applied radioactivity				
		Supernatant	Soil Extract	Residue	Lid wash	Total
SK961089	EQ A5	1.0	84.1	13.9	1.5	100.5
	EQ A6	0.4	83.0	13.2	1.2	97.8
SK566696	EQ B5	1.3	87.4	3.9	2.1	94.7
	EQ B6	1.2	84.9	3.7	2.3	92.1
Emperor Son's Lake	EQ C5	0.7	87.9	5.5	3.3	97.4
	EQ C6	0.9	86.2	5.6	4.8	97.5
SK179618	EQ D5	0.6	38.9	52.7	0.0	92.2
	EQ D6	1.6	37.0	53.7	0.9	93.2

Stability Test not performed for Eurosoil 5.

Table 5

Stability over a 24 hour period (20°C) at a soil to aqueous phase ratio of 1:1

Soil	Sample	Percent of applied radioactivity	
		Amount in soil extract	8-2 TBA
SK961089	EQ A5	84.1	72.4
	EQ A6	83.0	73.2
SK566696	EQ B5	87.4	77.0
	EQ B6	84.9	76.6
Emperor Son's Lake	EQ C5	87.9	87.0
	EQ C6	86.2	85.7
SK179618	EQ D5	38.9	5.2
	EQ D6	37.0	4.4

Stability Test not performed for Eurosoil 5.

Table 6

Comparison of the applied radioactivity recovered in the supernatants from soils (soil to aqueous phase ratio of 1:1)

Soil	Sample	Percent of applied radioactivity	
		3 h @ 4°C	3 h @ 20°C
SK961089	EQ A9	1.9	1.1
	EQ A10	1.3	0.9
SK566696	EQ B9	1.7	2.1
	EQ B10	1.8	1.3
Emperor Son's Lake	EQ C9	1.8	2.0
	EQ C10	2.7	2.4
SK179618	EQ D9	1.3	2.4
	EQ D10	1.3	2.6

Stability Test not performed for Eurosoil 5.

Table 7

Stability over a 3 hour period (4°C) at a soil to aqueous phase ratio of 1:1

Soil	Sample	Percent of applied radioactivity	
		Amount in soil extract	8-2 TBA
SK961089	EQ A9	93.5	93.4
	EQ A10	101.6	100.0
SK566696	EQ B9	95.2	95.0
	EQ B10	95.4	95.1
Emperor Son's Lake	EQ C9	91.9	90.7
	EQ C10	88.5	88.0
SK179618	EQ D9	86.5	69.5
	EQ D10	84.4	66.9

Stability Test not performed for Eurosoil 5.

Table 8

Percent applied radioactivity recovered in the supernatants from sterile soils at 4°C and stability of 8-2 TBA in the soil extract of sterile soil

Soil	Sample	Percent of applied radioactivity		
		3 h @ 4°C	Amount in soil	8-2 TBA
SK179618	EQ D11	1.9	92.0	90.3
	EQ D12	3.0	82.6	81.1
Eurosoil 5	EQ E11	1.1	92.9	91.1
	EQ E12	1.2	94.1	91.5

Table 9

Percent applied radioactivity recovered in the desorption supernatants from sterile soils at 4°C following a 3 h equilibrium time

Desorption time (h)	Percent of applied radioactivity in solution				
	SK961089	SK566696	Emperor Son's Lake	SK179618	Eurosoil 5
0	0.0	0.0	0.0	0.0	0.0
1.5	0.8	1.2	1.0	1.0	0.6
3	0.7	1.2	1.5	1.3	0.7
6	1.0	1.3	2.0	1.4	0.7
24	1.4	1.8	1.3	1.7	0.8

Values presented are mean values of two replicate samples.

Table 10

Values for C_e, X/m, C_1 and X_1/m for 8-2 TBA on soil SK961089

Nominal Dose level (µg/mL)	Replicate	Adsorption		Desorption	
		C_e (µg/mL)	X/m (µg/g)	C_1 (µg/mL)	X_1/m (µg/g)
0.125	1	0.0021	0.1223	0.0010	0.1225
	2	0.0021	0.1224	0.0010	0.1225
	Mean	0.0021	0.1223	0.0010	0.1225
0.1	1	0.0013	0.0985	0.0008	0.0984
	2	0.0012	0.0989	0.0009	0.0986
	Mean	0.0012	0.0987	0.0009	0.0985
0.075	1	0.0010	0.0741	0.0005	0.0742
	2	0.0016	0.0744	0.0017	0.0736
	Mean	0.0013	0.0743	0.0011	0.0739
0.05	1	0.0009	0.0496	0.0004	0.0497
	2	0.0007	0.0497	0.0004	0.0497
	Mean	0.0008	0.0497	0.0004	0.0497
0.0125	1	0.0002	0.0126	0.0001	0.0126
	2	0.0001	0.0127	0.0001	0.0127
	Mean	0.0002	0.0127	0.0001	0.0127

C_e and C_1 are the aqueous phase concentrations; X/m and X_1/m are the concentrations in the soil

Table 11

Values for C_e, X/m, C_1 and X_1/m for 8-2 TBA on soil SK566696

Nominal Dose level (µg/mL)	Replicate	Adsorption		Desorption	
		C_e (µg/mL)	X/m (µg/g)	C_1 (µg/mL)	X_1/m (µg/g)
0.125	1	0.0023	0.1220	0.0020	0.1207
	2	0.0024	0.1221	0.0025	0.1204
	Mean	0.0023	0.1221	0.0023	0.1205
0.1	1	0.0020	0.0978	0.0015	0.0970
	2	0.0017	0.0981	0.0014	0.0972
	Mean	0.0018	0.0979	0.0014	0.0971
0.075	1	0.0012	0.0741	0.0010	0.0735
	2	0.0013	0.0740	0.0010	0.0735
	Mean	0.0013	0.0740	0.0010	0.0735
0.05	1	0.0010	0.0495	0.0010	0.0488
	2	0.0009	0.0495	0.0008	0.0491
	Mean	0.0010	0.0495	0.0009	0.0489
0.0125	1	0.0002	0.0126	0.0002	0.0124
	2	0.0002	0.0127	0.0002	0.0125
	Mean	0.0002	0.0127	0.0002	0.0125

C_e and C_1 are the aqueous phase concentrations; X/m and X_1/m are the concentrations in the soil

Table 12

Values for C_e, X/m, C_1 and X_1/m for 8-2 TBA on Emperor Son's Lake sediment

Nominal Dose level (µg/mL)	Replicate	Adsorption		Desorption	
		C_e (µg/mL)	X/m (µg/g)	C_1 (µg/mL)	X_1/m (µg/g)
0.125	1	0.0028	0.1212	0.0019	0.1205
	2	0.0027	0.1225	0.0018	0.1217
	Mean	0.0027	0.1218	0.0019	0.1211
0.1	1	0.0020	0.0977	0.0013	0.0975
	2	0.0021	0.0979	0.0013	0.0974
	Mean	0.0021	0.0978	0.0013	0.0974
0.075	1	0.0014	0.0735	0.0010	0.0730
	2	0.0026	0.0726	0.0009	0.0729
	Mean	0.0020	0.0731	0.0009	0.0729
0.05	1	0.0011	0.0492	0.0007	0.0491
	2	0.0009	0.0493	0.0005	0.0492
	Mean	0.0010	0.0492	0.0006	0.0491
0.0125	1	0.0002	0.0127	0.0001	0.0126
	2	0.0002	0.0127	0.0001	0.0126
	Mean	0.0002	0.0127	0.0001	0.0126

C_e and C_1 are the aqueous phase concentrations; X/m and X_1/m are the concentrations in the soil

Table 13

Values for C_e, X/m, C_1 and X_1/m for 8-2 TBA on soil SK179618

Nominal Dose level (µg/mL)	Replicate	Adsorption		Desorption	
		C_e (µg/mL)	X/m (µg/g)	C_1 (µg/mL)	X_1/m (µg/g)
0.125	1	0.0021	0.1218	0.0015	0.1212
	2	0.0020	0.1220	0.0015	0.1215
	Mean	0.0020	0.1219	0.0015	0.1214
0.1	1	0.0016	0.0976	0.0013	0.0971
	2	0.0016	0.0980	0.0014	0.0974
	Mean	0.0016	0.0978	0.0013	0.0972
0.075	1	0.0013	0.0735	0.0009	0.0732
	2	0.0011	0.0736	0.0010	0.0731
	Mean	0.0012	0.0735	0.0010	0.0731
0.05	1	0.0010	0.0492	0.0006	0.0492
	2	0.0008	0.0495	0.0006	0.0492
	Mean	0.0009	0.0493	0.0006	0.0492
0.0125	1	0.0003	0.0126	0.0002	0.0125
	2	0.0002	0.0126	0.0002	0.0125
	Mean	0.0003	0.0126	0.0002	0.0125

C_e and C_1 are the aqueous phase concentrations; X/m and X_1/m are the concentrations in the soil

Table 14

Values for C_e, X/m, C_1 and X_1/m for 8-2 TBA on Eurosoil 5

Nominal Dose level (µg/mL)	Replicate	Adsorption		Desorption	
		C_e (µg/mL)	X/m (µg/g)	C_1 (µg/mL)	X_1/m (µg/g)
0.125	1	0.0020	0.1225	0.0010	0.1223
	2	0.0016	0.1219	0.0010	0.1215
	Mean	0.0018	0.1222	0.0010	0.1219
0.1	1	0.0012	0.0990	0.0009	0.0985
	2	0.0011	0.0988	0.0009	0.0983
	Mean	0.0012	0.0989	0.0009	0.0984
0.075	1	0.0012	0.0737	0.0006	0.0736
	2	0.0013	0.0734	0.0008	0.0731
	Mean	0.0012	0.0735	0.0007	0.0733
0.05	1	0.0006	0.0499	0.0003	0.0497
	2	0.0006	0.0498	0.0003	0.0497
	Mean	0.0006	0.0499	0.0003	0.0497
0.0125	1	0.0002	0.0127	0.0001	0.0127
	2	0.0002	0.0127	0.0001	0.0127
	Mean	0.0002	0.0127	0.0001	0.0127

C_e and C_1 are the aqueous phase concentrations; X/m and X_1/m are the concentrations in the soil

Table 15

Quantities of 8-2 TBA adsorbed to and desorbed from soil SK961089

Nominal Dose level (µg/mL)	Replicate	Percent applied chemical adsorbed	Weight (µg) of adsorbed chemical following		Percent of adsorbed chemical desorbed	Percent of adsorbed chemical not desorbed
			Adsorption	Desorption		
0.125	1	98.2	1.1010	1.1026	0.0	100.1
	2	98.2	1.1013	1.1024	0.0	100.1
	Mean	98.2	1.1011	1.1025	0.0	100.1
0.1	1	98.7	0.8882	0.8872	0.1	99.9
	2	98.8	0.8888	0.8863	0.3	99.7
	Mean	98.7	0.8885	0.8868	0.2	99.8
0.075	1	98.6	0.6684	0.6687	0.0	100.0
	2	97.8	0.6631	0.6564	1.0	99.0
	Mean	98.2	0.6657	0.6625	0.5	99.5
0.05	1	98.2	0.4457	0.4461	0.0	100.1
	2	98.5	0.4470	0.4469	0.0	100.0
	Mean	98.3	0.4464	0.4465	0.0	100.0
0.0125	1	98.3	0.1140	0.1142	0.0	100.1
	2	98.8	0.1146	0.1144	0.2	99.8
	Mean	98.5	0.1143	0.1143	0.0	100.0

Table 16

Quantities of 8-2 TBA adsorbed to and desorbed from soil SK566696

Nominal Dose level (µg/mL)	Replicate	Percent applied chemical adsorbed	Weight (µg) of adsorbed chemical following		Percent of adsorbed chemical desorbed	Percent of adsorbed chemical not desorbed
			Adsorption	Desorption		
0.125	1	98.1	1.0996	1.0879	1.1	98.9
	2	98.1	1.0992	1.0834	1.4	98.6
	Mean	98.1	1.0994	1.0856	1.2	98.8
0.1	1	98.0	0.8818	0.8747	0.8	99.2
	2	98.3	0.8843	0.8763	0.9	99.1
	Mean	98.1	0.8830	0.8755	0.9	99.1
0.075	1	98.3	0.6668	0.6617	0.8	99.2
	2	98.2	0.6657	0.6608	0.7	99.3
	Mean	98.3	0.6662	0.6613	0.8	99.2
0.05	1	98.0	0.4448	0.4391	1.3	98.7
	2	98.1	0.4456	0.4414	0.9	99.1
	Mean	98.1	0.4452	0.4402	1.1	98.9
0.0125	1	98.2	0.1139	0.1124	1.4	98.6
	2	98.3	0.1141	0.1126	1.3	98.7
	Mean	98.3	0.1140	0.1125	1.3	98.7

Table 17

Quantities of 8-2 TBA adsorbed to and desorbed from Emperor Son's Lake sediment

Nominal Dose level (µg/mL)	Replicate	Percent applied chemical adsorbed	Weight (µg) of adsorbed chemical following		Percent of adsorbed chemical desorbed	Percent of adsorbed chemical not desorbed
			Adsorption	Desorption		
0.125	1	97.7	1.0951	1.0893	0.5	99.5
	2	97.8	1.0958	1.0889	0.6	99.4
	Mean	97.7	1.0954	1.0891	0.6	99.4
0.1	1	97.9	0.8810	0.8788	0.3	99.7
	2	97.8	0.8804	0.8754	0.6	99.4
	Mean	97.9	0.8807	0.8771	0.4	99.6
0.075	1	98.1	0.6649	0.6602	0.7	99.3
	2	96.4	0.6536	0.6561	0.0	100.4
	Mean	97.2	0.6593	0.6581	0.2	99.8
0.05	1	97.7	0.4438	0.4424	0.3	99.7
	2	98.1	0.4454	0.4445	0.2	99.8
	Mean	97.9	0.4446	0.4434	0.3	99.7
0.0125	1	98.4	0.1142	0.1138	0.4	99.6
	2	98.4	0.1142	0.1135	0.6	99.4
	Mean	98.4	0.1142	0.1136	0.5	99.5

Table 18

Quantities of 8-2 TBA adsorbed to and desorbed from soil SK 179618

Nominal Dose level (µg/mL)	Replicate	Percent applied chemical adsorbed	Weight (µg) of adsorbed chemical following		Percent of adsorbed chemical desorbed	Percent of adsorbed chemical not desorbed
			Adsorption	Desorption		
0.125	1	98.0	1.0972	1.0922	0.5	99.5
	2	98.2	1.0984	1.0935	0.4	99.6
	Mean	98.1	1.0978	1.0928	0.5	99.5
0.1	1	98.2	0.8819	0.8772	0.5	99.5
	2	98.2	0.8819	0.8759	0.7	99.3
	Mean	98.2	0.8819	0.8765	0.6	99.4
0.075	1	98.1	0.6630	0.6600	0.4	99.6
	2	98.3	0.6646	0.6603	0.7	99.3
	Mean	98.2	0.6638	0.6601	0.5	99.5
0.05	1	97.7	0.4435	0.4428	0.1	99.9
	2	98.1	0.4454	0.4431	0.5	99.5
	Mean	97.9	0.4444	0.4430	0.3	99.7
0.0125	1	97.4	0.1130	0.1125	0.4	99.6
	2	97.9	0.1135	0.1130	0.4	99.6
	Mean	97.6	0.1132	0.1128	0.4	99.6

Table 19

Quantities of 8-2 TBA adsorbed to and desorbed from Eurosoil 5

Nominal Dose level (µg/mL)	Replicate	Percent applied chemical adsorbed	Weight (µg) of adsorbed chemical following		Percent of adsorbed chemical desorbed	Percent of adsorbed chemical not desorbed
			Adsorption	Desorption		
0.125	1	98.4	1.1009	1.0989	0.2	99.8
	2	98.7	1.1040	1.1004	0.3	99.7
	Mean	98.5	1.1024	1.0997	0.3	99.7
0.1	1	98.8	0.8868	0.8828	0.4	99.6
	2	98.8	0.8876	0.8834	0.5	99.5
	Mean	98.8	0.8872	0.8831	0.5	99.5
0.075	1	98.4	0.6649	0.6639	0.2	99.8
	2	98.2	0.6641	0.6617	0.4	99.6
	Mean	98.3	0.6645	0.6628	0.3	99.7
0.05	1	98.8	0.4486	0.4475	0.2	99.8
	2	98.8	0.4488	0.4477	0.2	99.8
	Mean	98.8	0.4487	0.4476	0.2	99.8
0.0125	1	98.8	0.1146	0.1144	0.1	99.9
	2	98.8	0.1146	0.1143	0.2	99.8
	Mean	98.8	0.1146	0.1144	0.2	99.8

Table 20
Adsorption coefficients for 8-2 TBA on soil SK961089

Nominal Dose level (µg/mL)	Replicate	Adsorption coefficients		
		K_d	K_{doc}	K_{dom}
0.125	1	58.12	1263	736
	2	58.91	1281	746
	Mean	58.51	1272	741
0.1	1	78.71	1711	996
	2	84.09	1828	1064
	Mean	81.40	1770	1030
0.075	1	74.16	1612	939
	2	46.60	1013	590
	Mean	60.38	1313	764
0.05	1	57.49	1250	728
	2	68.03	1479	861
	Mean	62.76	1364	794
0.0125	1	60.84	1323	770
	2	87.74	1907	1111
	Mean	74.29	1615	940

Table 21
Adsorption coefficients for 8-2 TBA on soil SK566696

Nominal Dose level (µg/mL)	Replicate	Adsorption coefficients		
		K_d	K_{doc}	K_{dom}
0.125	1	52.56	6570	3754
	2	51.80	6475	3700
	Mean	52.18	6522	3727
0.1	1	48.88	6110	3492
	2	58.20	7275	4157
	Mean	53.54	6693	3824
0.075	1	61.12	7639	4365
	2	55.70	6962	3979
	Mean	58.41	7301	4172
0.05	1	49.53	6191	3538
	2	54.79	6849	3914
	Mean	52.16	6520	3726
0.0125	1	57.44	7181	4103
	2	60.71	7588	4336
	Mean	59.08	7384	4220

Table 22
Adsorption coefficients for 8-2 TBA on Emperor Son's Lake

Nominal Dose level (µg/mL)	Replicate	Adsorption coefficients		
		K_d	K_{doc}	K_{dom}
0.125	1	43.26	3605	2060
	2	45.82	3818	2182
	Mean	44.54	3712	2121
0.1	1	48.00	4000	2286
	2	46.97	3914	2237
	Mean	47.49	3957	2261
0.075	1	51.79	4316	2466
	2	27.54	2295	1311
	Mean	39.66	3305	1889
0.05	1	44.96	3747	2141
	2	53.66	4471	2555
	Mean	49.31	4109	2348
0.0125	1	64.21	5351	3057
	2	65.03	5420	3097
	Mean	64.62	5385	3077

Table 23
Adsorption coefficients for 8-2 TBA on soil SK179618

Nominal Dose level (µg/mL)	Replicate	Adsorption coefficients		
		K_d	K_{doc}	K_{dom}
0.125	1	57.96	1525	878
	2	61.66	1623	934
	Mean	59.81	1574	906
0.1	1	62.73	1651	950
	2	63.00	1658	955
	Mean	62.87	1654	953
0.075	1	58.06	1528	880
	2	66.71	1755	1011
	Mean	62.38	1642	945
0.05	1	48.21	1269	730
	2	59.43	1564	900
	Mean	53.82	1416	815
0.0125	1	43.44	1143	658
	2	52.86	1391	801
	Mean	48.15	1267	729

Table 24
Adsorption coefficients for 8-2 TBA on Eurosoil 5

Nominal Dose level (µg/mL)	Replicate	Adsorption coefficients		
		K_d	K_{doc}	K_{dom}
0.125	1	62.58	934	539
	2	75.24	1123	649
	Mean	68.91	1028	594
0.1	1	83.18	1242	717
	2	88.44	1320	762
	Mean	85.81	1281	740
0.075	1	61.56	919	531
	2	57.33	856	494
	Mean	59.44	887	512
0.05	1	85.77	1280	739
	2	88.83	1326	766
	Mean	87.30	1303	753
0.0125	1	82.96	1238	715
	2	84.55	1262	729
	Mean	83.75	1250	722

Table 25
Freundlich adsorption and desorption constants and regression analysis for 8-2 TBA on soil SK961089

Experimental Stage	Adsorption constant			1/n	r^2
	K_f	K_{oc}	K_{om}		
Adsorption Step	35.20	765	446	0.9105	0.9534
Desorption Step	91.57	1991	1159	0.9636	0.9922

Table 26

Freundlich adsorption and desorption constants and regression analysis for 8-2 TBA on soil SK566696

Experimental Stage	Adsorption constant			1/n	r^2
	K_f	K_{oc}	K_{om}		
Adsorption Step	39.53	4941	2823	0.9522	0.9937
Desorption Step	77.35	9669	5525	1.0340	0.9632

Table 27

Freundlich adsorption and desorption constants and regression analysis for 8-2 TBA on Emperor Son's Lake sediment

Experimental Stage	Adsorption constant			1/n	r^2
	K_f	K_{oc}	K_{om}		
Adsorption Step	20.00	1667	952	0.8633	0.9967
Desorption Step	34.25	2854	1631	0.8875	0.9902

Table 28

Freundlich adsorption and desorption constants and regression analysis for 8-2 TBA on soil SK179618

Experimental Stage	Adsorption constant			1/n	r^2
	K_f	K_{oc}	K_{om}		
Adsorption Step	129.73	3414	1966	1.1188	0.9864
Desorption Step	103.92	2735	1575	1.0422	0.9927

Table 29

Freundlich adsorption and desorption constants and regression analysis for 8-2 TBA on Eurosoil 5

Experimental Stage	Adsorption constant			1/n	r^2
	K_f	K_{oc}	K_{om}		
Adsorption Step	38.34	572	331	0.9049	0.9719
Desorption Step	47.85	714	412	0.8718	0.9834

Table 30

Recovery of radioactivity from all five soils treated with 8-2 TBA at 0.125 µg/mL after adsorption, desorption, organic wash and combustion

	% of Applied Radioactivity					
Sample	Ads Supernatant	Des Supernatant	Soil extract	Lid wash	Residue	Total
A1	0.8	0.4	85.6	0.9	4.9	92.6
A2	0.8	0.4	83.8	1.8	3.7	90.5
B1	1.3	1.1	87.0	1.6	1.8	92.8
B2	1.3	1.4	82.2	3.2	2.0	90.1
C1	1.3	0.8	77.5	3.0	3.8	86.4
C2	1.3	0.9	76.9	2.8	3.7	85.6
D1	1.0	0.7	86.0	0.8	4.3	92.8
D2	0.9	0.7	88.4	0.9	4.7	95.6
E1	1.0	0.5	89.7	0.7	1.6	93.5
E2	0.8	0.5	90.6	1.0	1.5	94.4

Table 31

pH of the blank solutions used for the main isotherms test

Nominal Concentration	pH
0.125 µg/mL	6.05
0.0125 µg/mL	5.90

Table 32

pH of the adsorption supernatants from the main isotherms test following treatment with 8-2 TBA

Dose group	Replicate	pH
A	1	7.82
	2	8.14
	3	7.45
	4	7.65
	5	7.88
	6	7.87
	7	7.93
	8	7.94
	9	7.86
	10	8.35

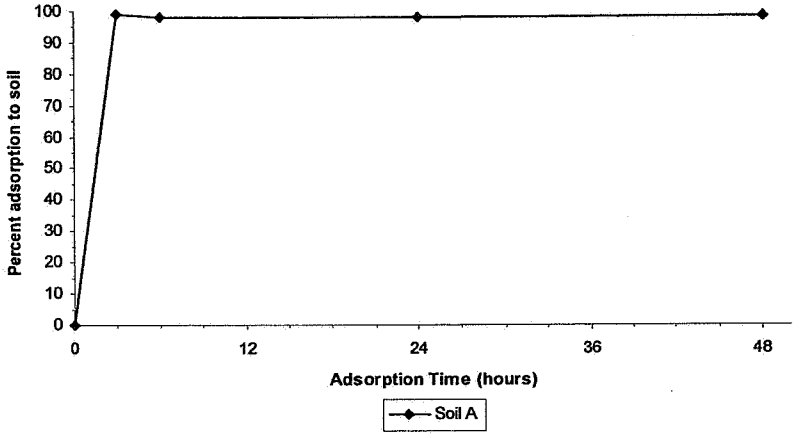
Table 32 continued

pH of the adsorption supernatants from the main isotherms test following treatment with 8-2 TBA

Dose group	Replicate	pH
B	1	4.26
	2	4.22
	3	4.11
	4	4.18
	5	4.14
	6	4.10
	7	4.17
	8	4.14
	9	4.14
	10	4.11
C	1	6.21
	2	6.17
	3	6.20
	4	6.24
	5	6.25
	6	6.16
	7	6.23
	8	6.25
	9	6.26
	10	6.31
D	1	5.74
	2	5.73
	3	5.43
	4	5.49
	5	5.47
	6	5.58
	7	5.54
	8	5.52
	9	5.58
	10	5.54
E	1	3.05
	2	3.00
	3	3.04
	4	3.02
	5	3.02
	6	2.99
	7	2.99
	8	2.99
	9	2.96
	10	2.99

FIGURES

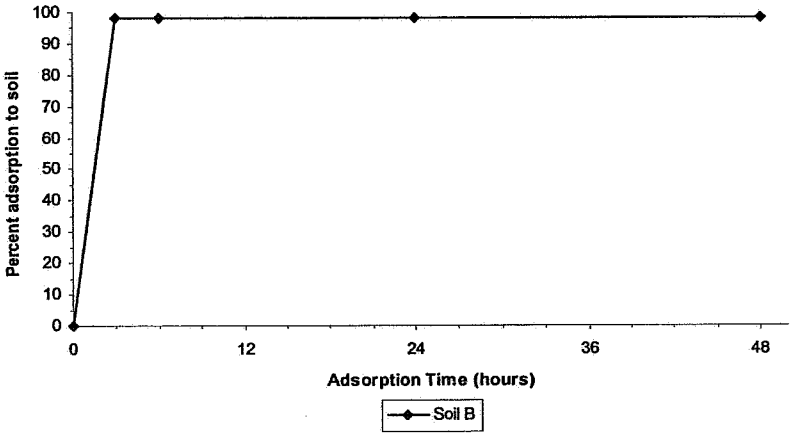
Figure 1
Adsorption equilibrium time data for soil SK961089 following treatment with 8-2 TBA



Adsorption Time (hours)	Percent adsorbed to soil
0	0
3	99
6	98.2
24	98.3
48	98.4

Figure 2

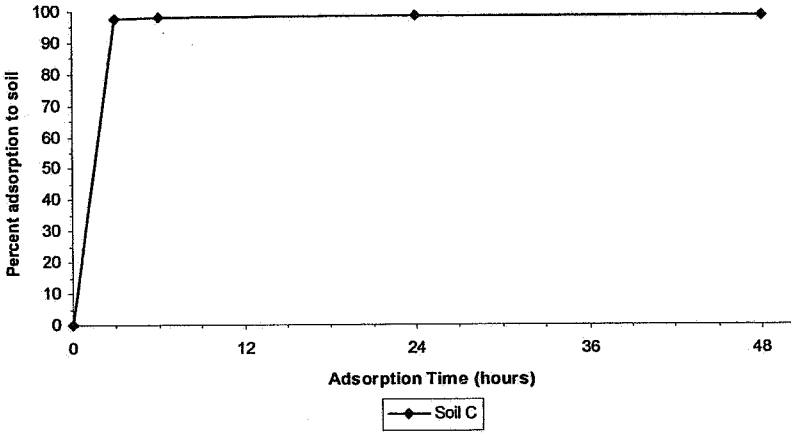
Adsorption equilibrium time data for soil SK566696 following treatment with 8-2 TBA



Adsorption Time (hours)	Percent adsorbed to soil
0	0
3	98.3
6	98.3
24	98.1
48	98.1

Figure 3

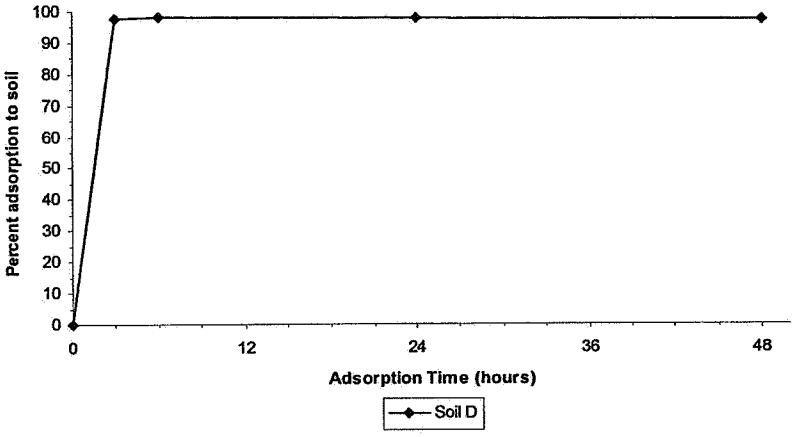
Adsorption equilibrium time data for Emperor Son's Lake sediment following treatment with 8-2 TBA



Adsorption Time (hours)	Percent adsorbed to soil
0	0
3	97.8
6	98.2
24	98.4
48	98.8

Figure 4

Adsorption equilibrium time data for soil SK179618 following treatment with 8-2 TBA



Adsorption Time (hours)	Percent adsorbed to soil
0	0
3	97.5
6	97.9
24	97.6
48	96.9

Figure 5

Stability of 8-2 TBA in the soil extract of non-sterile SK961089 soil following adsorption for 24 h at 20°C determined by TLC

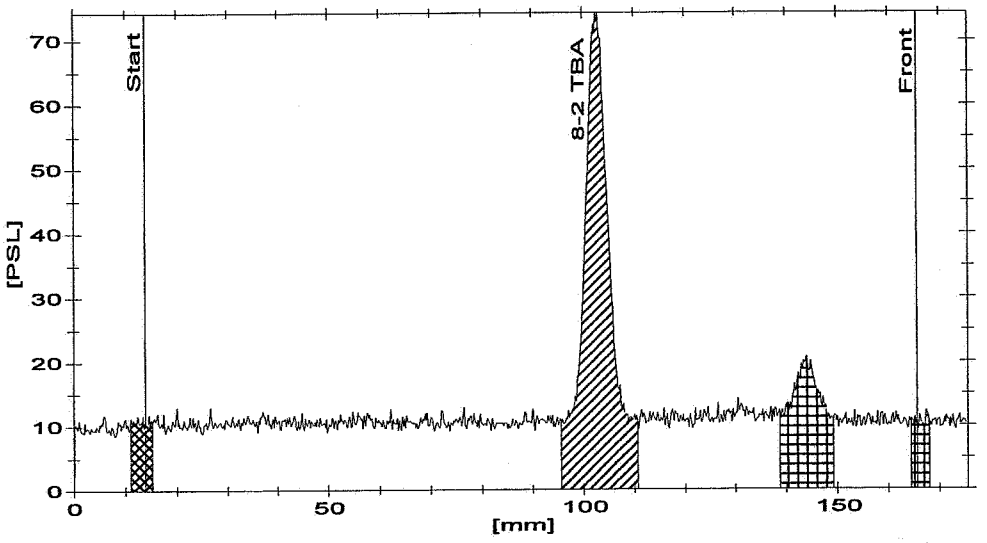


Plate 15, track 1

No	Name	Type	Width [mm]	Pos [mm]	RF	PSL	PSL-Bkg	%(PSL-Bkg)
1	8-2 TBA		15.00	102.80	0.59	2266.23	1467.26	86.14
2			10.40	143.80	0.86	781.36	227.41	13.35
3		Bkg	3.60	166.20	--	191.43	0.00	0.00
4		Bkg	4.20	13.00	--	224.04	0.00	0.00
-		Sum	25.40	--	--	3047.60	1694.66	99.49
-		Rem	150.20	--	--	8009.06	8.62	0.51
-		Ttl	175.60	--	--	11056.66	1703.28	100.00*

*normalised to this value

Figure 6

Stability of 8-2 TBA in the soil extract of non-sterile SK566696 soil following adsorption for 24 h at 20°C determined by TLC

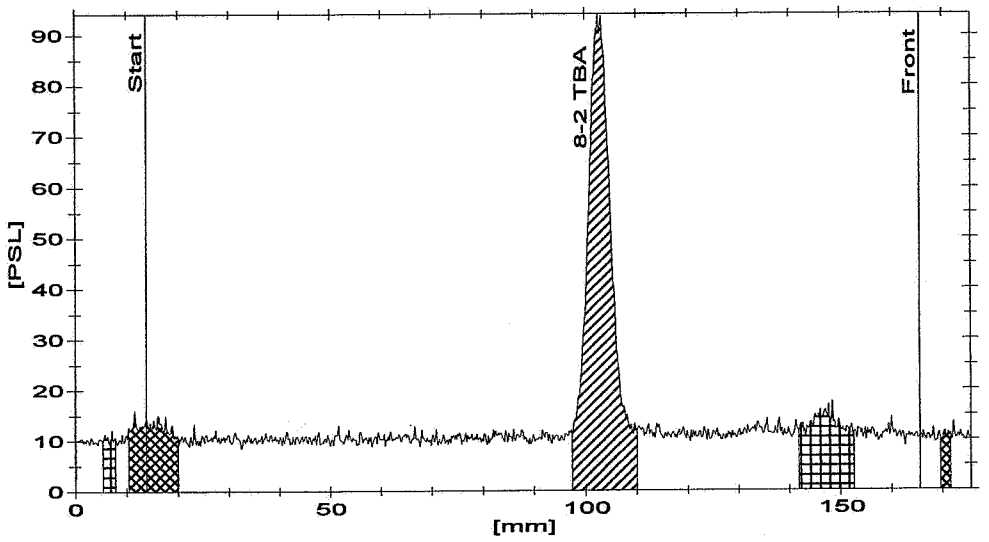


Plate 15, track 3

No	Name	Type	Width [mm]	Pos [mm]	RF	PSL	PSL-Bkg	%(PSL-Bkg)
1	8-2 TBA		12.60	103.00	0.59	2644.03	1971.68	88.09
2			10.80	147.00	0.88	717.25	140.95	6.30
3			9.60	15.00	0.01	611.78	99.52	4.45
4		Bkg	2.40	6.60	--	127.12	0.00	0.00
5		Bkg	2.00	170.60	--	107.67	0.00	0.00
-		Sum	33.00	--	--	3973.06	2212.16	98.83
-		Rem	142.60	--	--	7635.39	26.16	1.17
-		Ttl	175.60	--	--	11608.44	2238.31	100.00*

*normalised to this value

Figure 7

Stability of 8-2 TBA in the soil extract of non-sterile Emperor Son's Lake sediment following adsorption for 24 h at 20°C determined by TLC

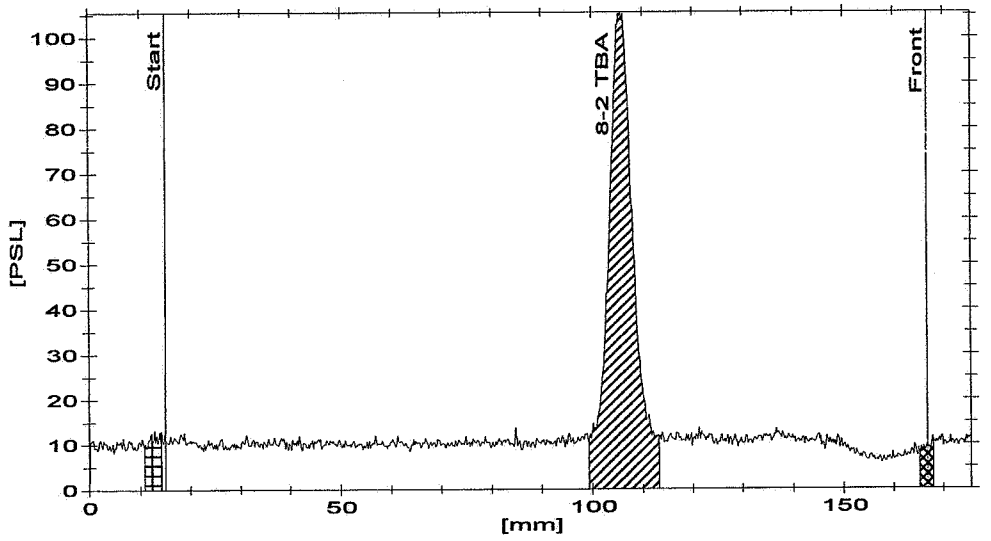


Plate 16, track 2

No	Name	Type	Width [mm]	Pos [mm]	RF	PSL	PSL-Bkg	%(PSL-Bkg)
1	8-2 TBA		13.80	106.00	0.60	3070.13	2365.81	99.41
2		Bkg	3.40	12.40	--	187.87	0.00	0.00
3		Bkg	2.80	166.60	1.00	128.56	0.00	0.00
-		Sum	13.80	--	--	3070.13	2365.81	99.41
-		Rem	161.80	--	--	8271.88	13.94	0.59
-		Ttl	175.60	--	--	11342.01	2379.74	100.00 *

*normalised to this value

Figure 8

Stability of 8-2 TBA in the soil extract of non-sterile SK179618 soil following adsorption for 24 h at 20°C determined by TLC

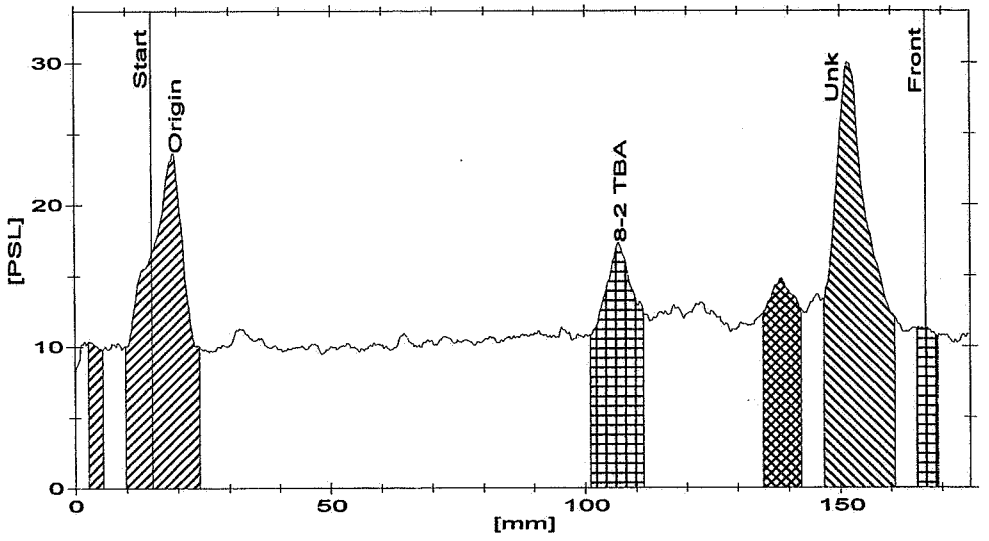


Plate 16, track 3

No	Name	Type	Width [mm]	Pos [mm]	RF	PSL	PSL-Bkg	%(PSL-Bkg)
1	Origin		14.40	17.00	0.01	1157.72	384.57	29.25
2	8-2 TBA		10.40	106.20	0.60	733.43	175.05	13.31
3			7.40	138.60	0.81	505.66	108.35	8.24
4	Unk		13.80	153.00	0.91	1360.35	619.41	47.11
5		Bkg	2.80	3.80	--	141.69	0.00	0.00
6		Bkg	4.20	167.00	--	234.14	0.00	0.00
-		Sum	46.00	--	--	3757.16	1287.38	97.91
-		Rem	129.60	--	--	6985.75	27.43	2.09
-		Ttl	175.60	--	--	10742.91	1314.82	100.00 *

*normalised to this value

Figure 9

Stability of 8-2 TBA in the soil extract of non-sterile SK961089 soil following adsorption for 3 h at 4°C determined by TLC

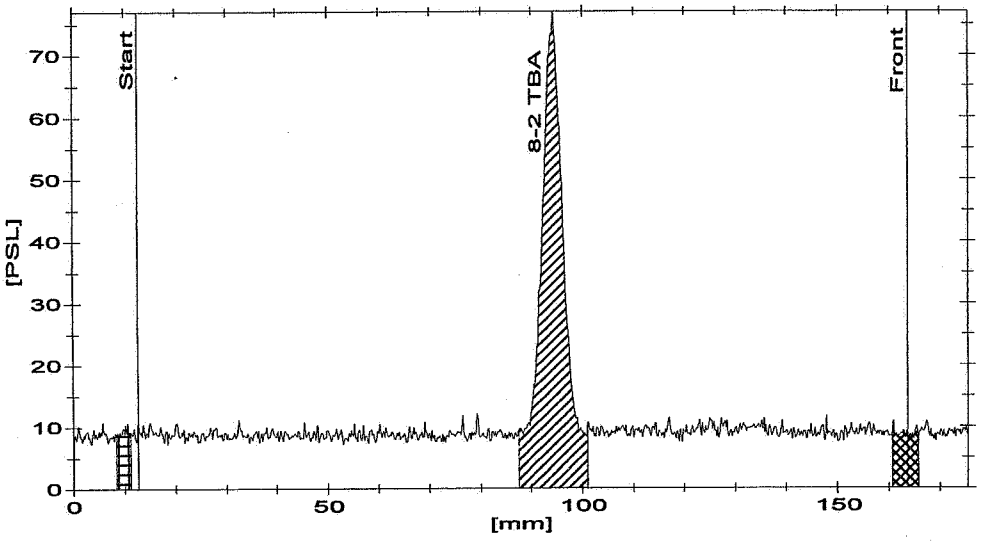


Plate 18, track 1

No	Name	Type	Width [mm]	Pos [mm]	RF	PSL	PSL-Bkg	%(PSL-Bkg)
1	8-2 TBA		13.40	94.20	0.54	2010.28	1410.08	99.92
2		Bkg	2.80	9.80	--	128.47	0.00	0.00
3		Bkg	5.20	163.00	1.00	229.86	0.00	0.00
-		Sum	13.40	--	--	2010.28	1410.08	99.92
-		Rem	162.20	--	--	7266.32	1.19	0.08
-		Ttl	175.60	--	--	9276.60	1411.26	100.00 *

*normalised to this value

Figure 10

Stability of 8-2 TBA in the soil extract of non-sterile SK566696 soil following adsorption for 3 h at 4°C determined by TLC

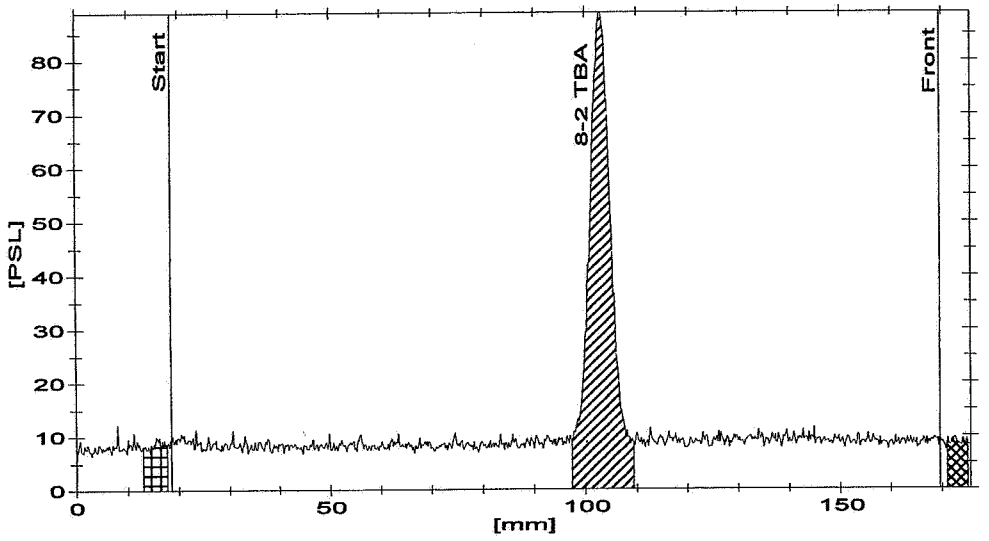


Plate 18, track 3

No	Name	Type	Width [mm]	Pos [mm]	RF	PSL	PSL-Bkg	%(PSL-Bkg)
1	8-2 TBA		12.00	103.00	0.56	2266.70	1748.24	99.77
2		Bkg	4.80	15.40	--	203.68	0.00	0.00
3		Bkg	4.00	172.80	--	176.52	0.00	0.00
-		Sum	12.00	--	--	2266.70	1748.24	99.77
-		Rem	163.60	--	--	7072.23	3.96	0.23
-		Ttl	175.60	--	--	9338.93	1752.21	100.00 *

*normalised to this value

Figure 11

Stability of 8-2 TBA in the soil extract of non-sterile Emperor Son's Lake sediment following adsorption for 3 h at 4°C determined by TLC

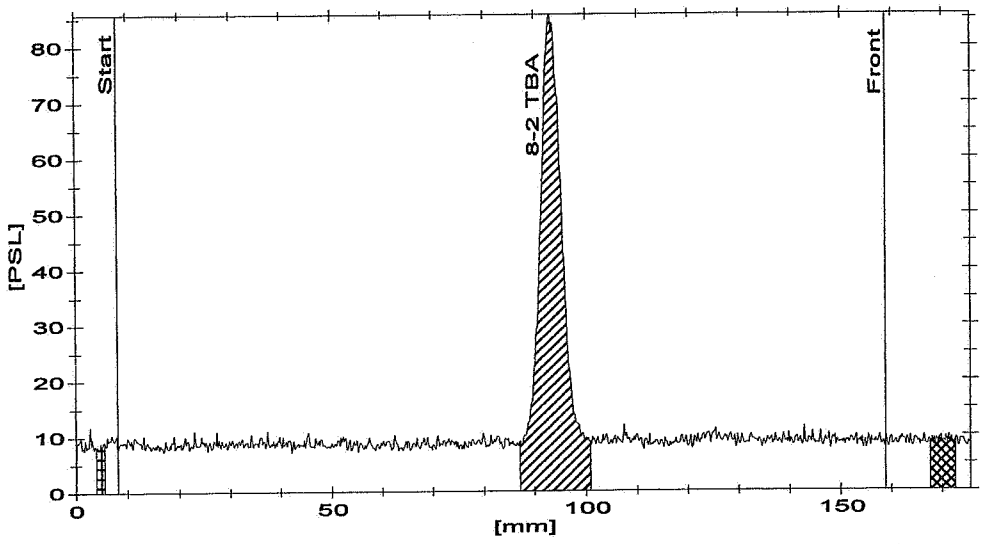


Plate 19, track 1

No	Name	Type	Width [mm]	Pos [mm]	RF	PSL	PSL-Bkg	%(PSL-Bkg)
1	8-2 TBA		13.60	93.60	0.57	2322.16	1722.79	98.70
2		Bkg	1.60	4.80	--	66.60	0.00	0.00
3		Bkg	5.00	170.00	--	224.27	0.00	0.00
-		Sum	13.60	--	--	2322.16	1722.79	98.70
-		Rem	162.00	--	--	7162.19	22.64	1.30
-		Ttl	175.60	--	--	9484.36	1745.44	100.00 *

*normalised to this value

Figure 12

Stability of 8-2 TBA in the soil extract of non-sterile SK179618 soil following adsorption for 3 h at 4°C determined by TLC

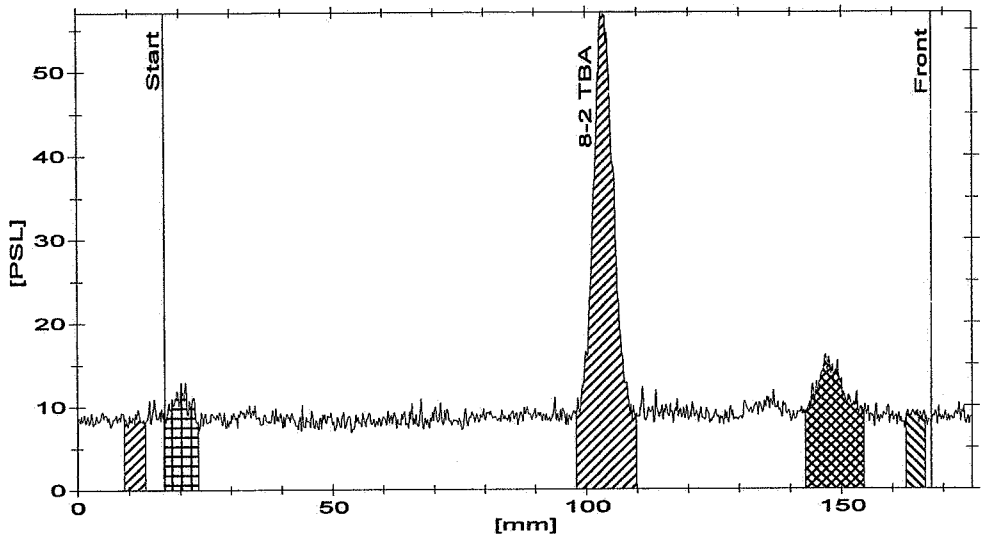


Plate 19, track 3

No	Name	Type	Width [mm]	Pos [mm]	RF	PSL	PSL-Bkg	%(PSL-Bkg)
1	8-2 TBA		11.80	103.60	0.58	1599.66	1086.36	80.39
2			6.80	20.20	0.02	350.20	54.40	4.03
3			11.40	148.40	0.87	684.24	188.34	13.94
4		Bkg	3.80	164.40	0.98	169.85	0.00	0.00
5		Bkg	4.00	11.20	--	169.45	0.00	0.00
-		Sum	30.00	--	--	2634.09	1329.11	98.36
-		Rem	145.60	--	--	6355.74	22.21	1.64
-		Ttl	175.60	--	--	8989.83	1351.32	100.00 *

*normalised to this value

Figure 13

Stability of 8-2 TBA in the soil extract of sterile SK179618 soil following adsorption for 3 h at 4°C determined by TLC

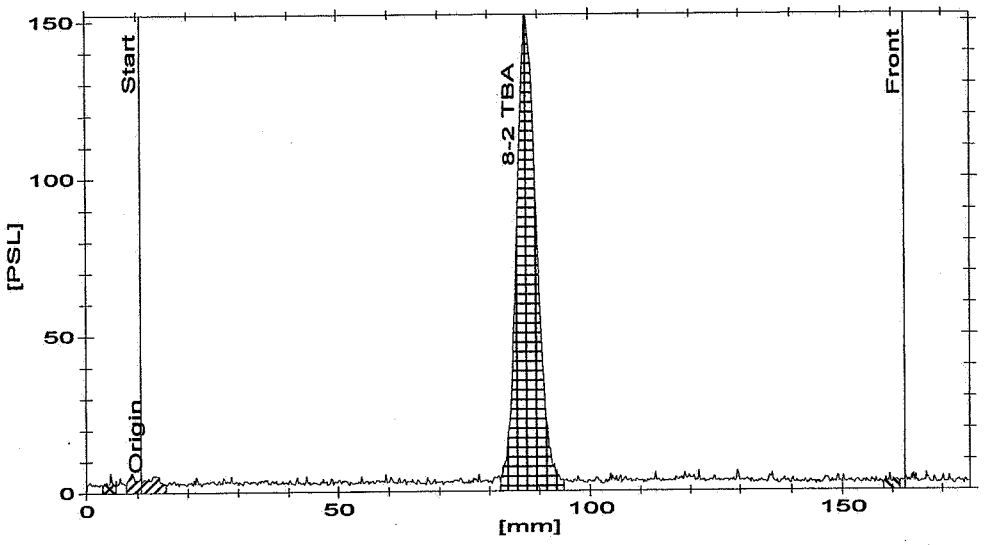


Plate 21, track 1

No	Name	Type	Width [mm]	Pos [mm]	RF	PSL	PSL-Bkg	%(PSL-Bkg)
1	Origin		7.80	11.40	0.01	169.26	45.65	1.34
2	8-2 TBA		12.60	87.80	0.51	3545.26	3345.58	98.17
3		Bkg	2.60	4.40	--	42.61	0.00	0.00
4		Bkg	3.20	159.60	0.98	49.30	0.00	0.00
-		Sum	20.40	--	--	3714.53	3391.24	99.51
-		Rem	155.20	--	--	2476.17	16.64	0.49
-		Ttl	175.60	--	--	6190.70	3407.87	100.00 *

*normalised to this value

Figure 14

Stability of 8-2 TBA in the soil extract of sterile Eurosoil 5 soil following adsorption for 3 h at 4°C determined by TLC

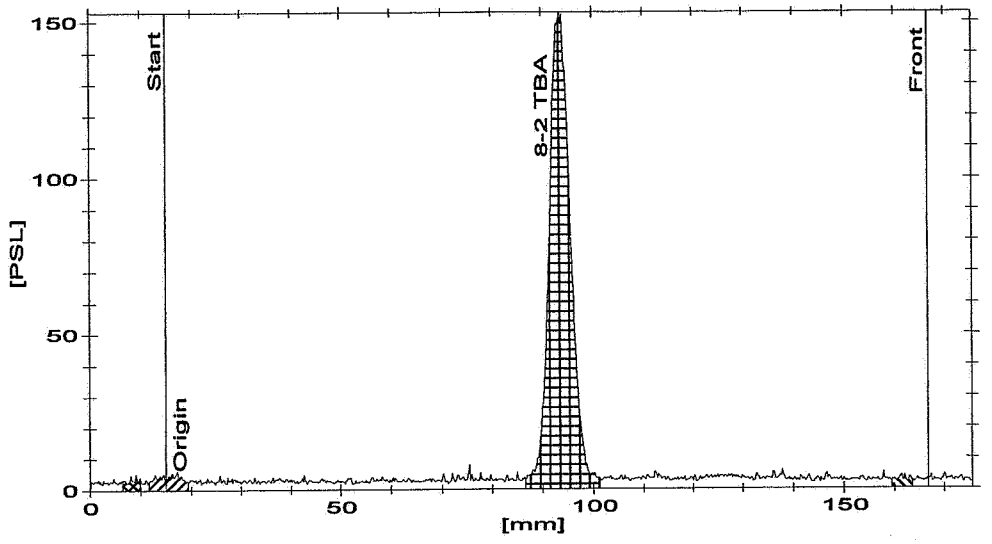


Plate 21, track 3

No	Name	Type	Width [mm]	Pos [mm]	RF	PSL	PSL-Bkg	%(PSL-Bkg)
1	Origin		7.80	15.40	0.00	158.93	39.19	1.09
2	8-2 TBA		14.60	93.60	0.52	3735.09	3510.96	98.01
3		Bkg	3.40	8.00	--	54.71	0.00	0.00
4		Bkg	4.20	161.40	0.96	61.96	0.00	0.00
-		Sum	22.40	--	--	3894.02	3550.15	99.10
-		Rem	153.20	--	--	2384.03	32.25	0.90
-		Ttl	175.60	--	--	6278.05	3582.40	100.00 *

*normalised to this value

Figure 15

Adsorption and desorption isotherms for 8-2 TBA on sterile soil SK961089 at 4°C

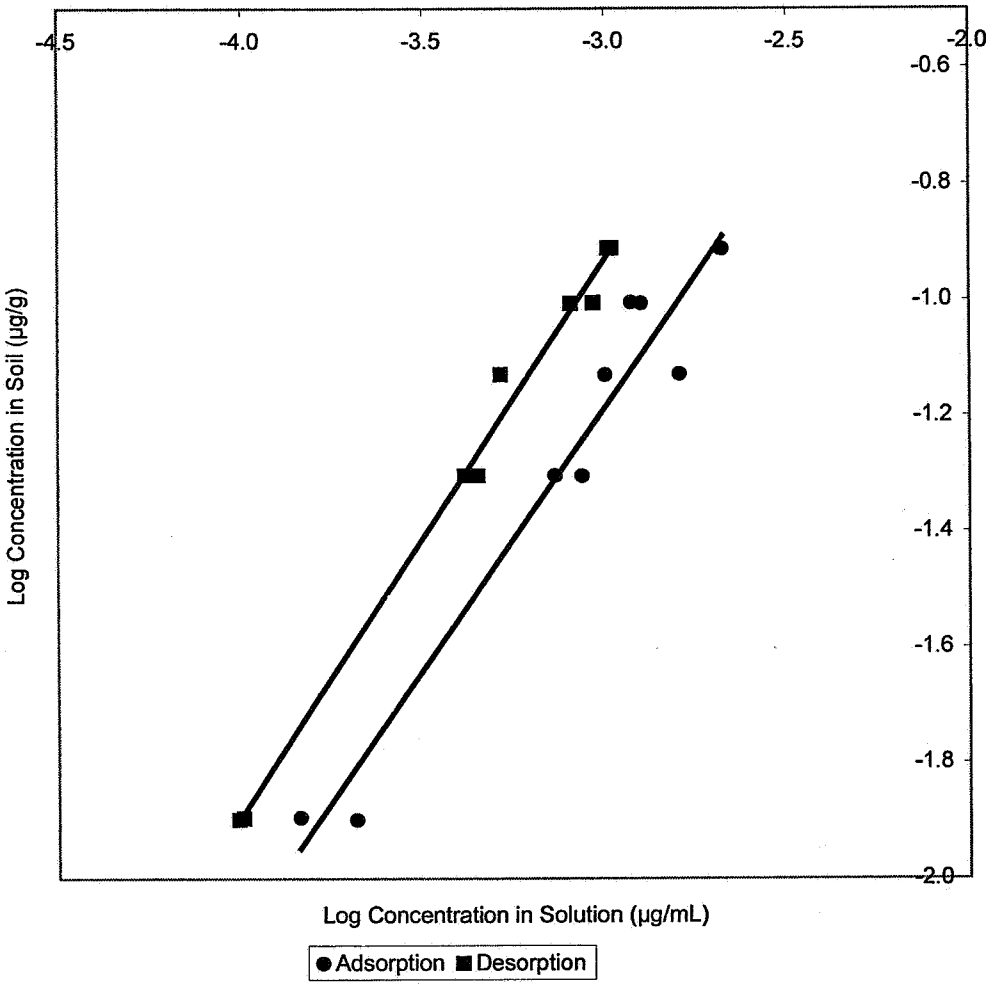


Figure 16

Adsorption and desorption isotherms for 8-2 TBA on sterile soil SK566696 at 4°C

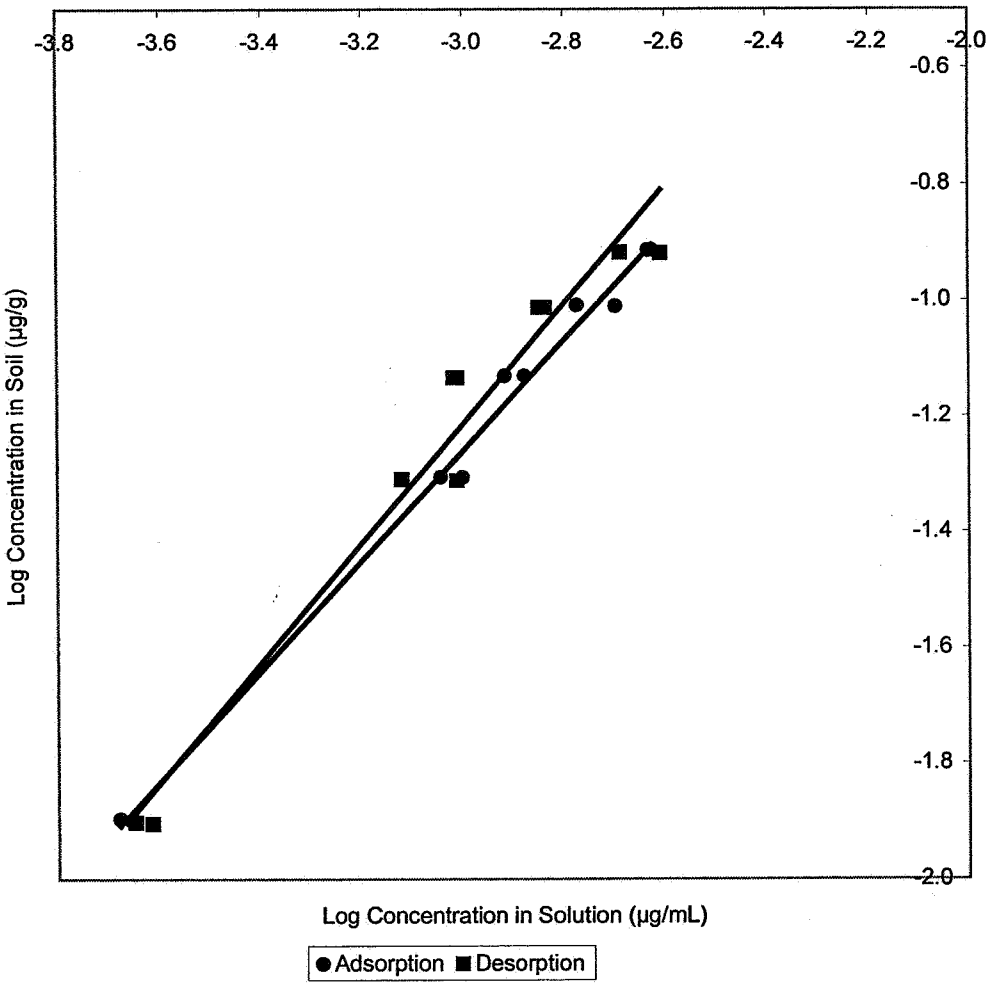


Figure 17

Adsorption and desorption isotherms for 8-2 TBA on sterile Emperor Son's Lake sediment at 4°C

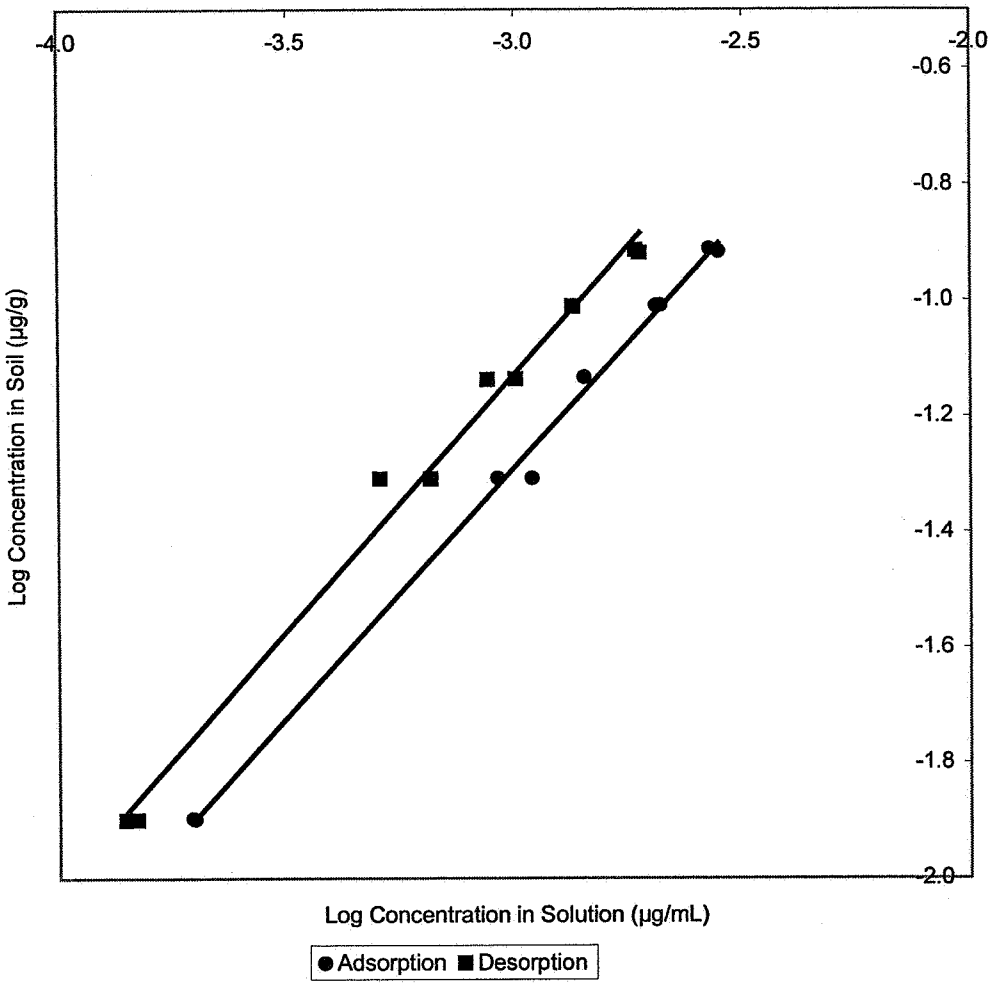


Figure 18

Adsorption and desorption isotherms for 8-2 TBA on sterile soil SK179618 at 4°C

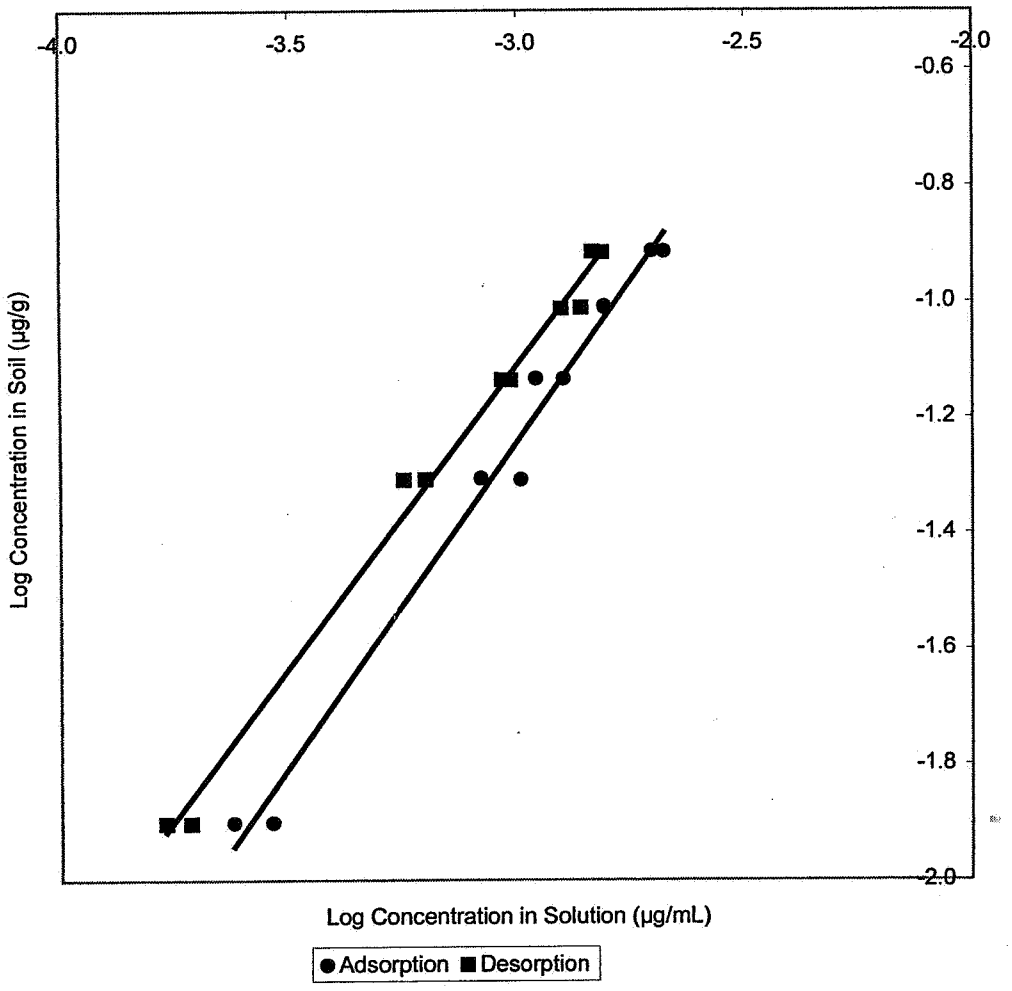
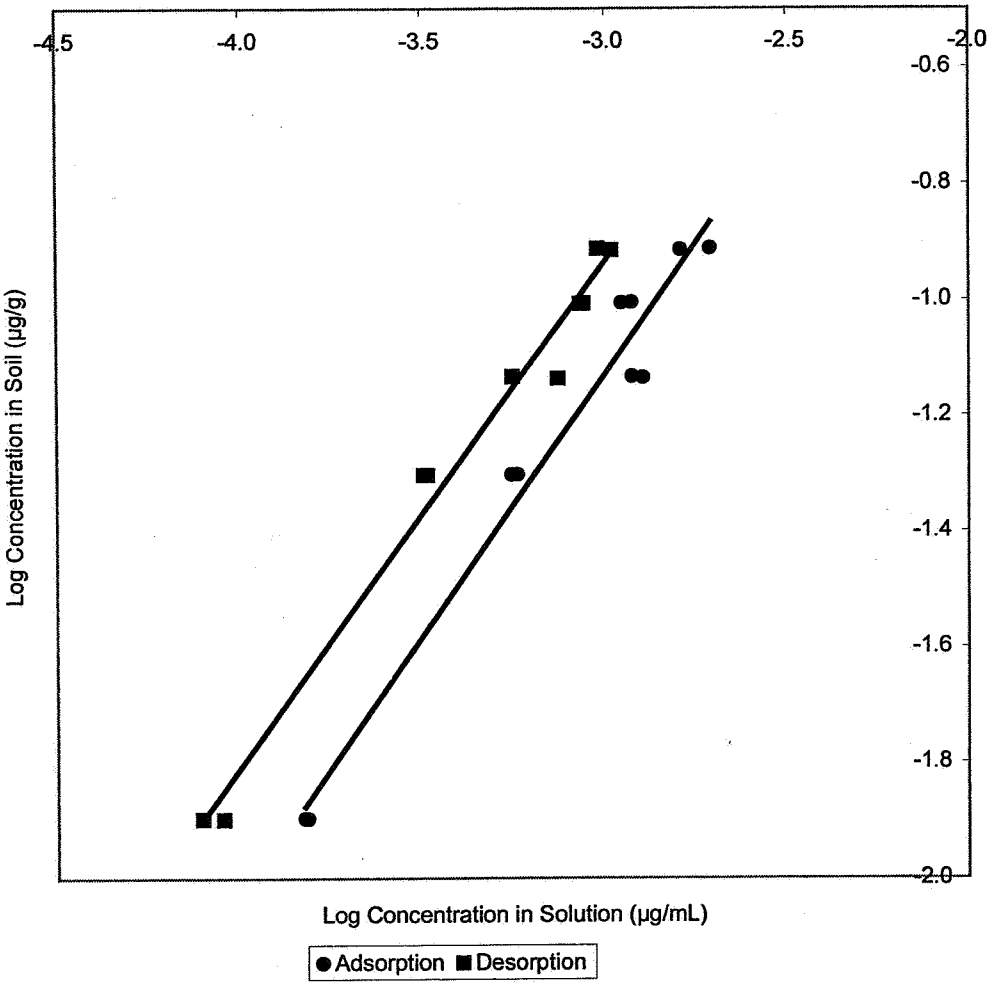


Figure 19

Adsorption and desorption isotherms for 8-2 TBA on sterile Eurosoil 5 soil at 4°C



APPENDICES

Appendix 1
Certificates of analysis

Certificate of analysis for (^{14}C)-8-2 TBA

CERTIFICATE OF ANALYSIS

Name:	1H, 1H, 2H, 2H-Perfluoro-1-decanol, [1,2-^{14}C]- DuPont Sample #H-25628
Lot Number:	3489-169
Molecular Formula:	$C_{10}H_5F_{17}O$
Formula Weight:	464
Date of Synthesis:	September 17, 2002 (Lot number 3489-165)
Date of Purification:	January 15, 2003
Last Solvent:	Dichloromethane
Physical Appearance:	Ethanolic solution
Structure:	

$C_8F_{17}{}^{14}CH_2{}^{14}CH_2OH$

ANALYTICAL METHOD	RESULT
Specific Activity (mass spectrometry on Lot No. 3489-165)	33 mCi/mmol (0.071 mCi/mg)
Radiochemical purity (HPLC; January 29, 2003)	>99%; Column: Vydac Protein C4; Mobile Phase: 0.1% TFA in Water/ Acetonitrile (50/50); Flow rate: 1 mL/min. Detector: Radioactivity

Issued by: _____ **Manager** **Date:** 3/19/03

Certificate of analysis for non-radiolabelled TBA

Clariant GmbH, Werk Gendorf
CQG / Qualitätsmanagement
D-84504 Burgkirchen
Fax: ++49 8679 / 2676

Clariant

Inspection certificate Date: 13.11.2001
according to EN10204-3.1B Page: 1 / 1

Our consignment
Material : 1-Decanol, 3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluoro
Material-no. : 104705
Batch No. : P00/001

On the batch, of which the consignment is a part, the following values were
determined. They conform to the agreed product specification.

Inspection characteristic/-method	Specification	Result
C8-Perfluoralkylethanol Capillary GC	>= 97,5	99,3%[a/a]
Sum Perfluoroalkylethanol Capillary GC	>= 98,0	99,9%[a/a]
Sum Perfluoralkylbutanol Capillary GC	<=1,0	< 0,1%[a/a]
Sum Perfluoralkylethene Capillary GC	<=0,5	< 0,1%[a/a]
Water content Karl Fischer DIN 51777	<=0,2	< 0,1%
N-Methylpyrrolidone content Capillary GC	<=0,2	< 0,1%

The above particulars do not release the customer from the obligation to carry out an
inspection of goods received.

Ms. Baumgärtner - Worksinspector

This report is not to be signed

Appendix 2
Soil certificates of analysis

Soil SK961089

TEST SYSTEM CERTIFICATE OF ANALYSIS

Test system code	CS17/00-628
Test system name	SK961089
Collected by	Land Research Associates, Lockington, Derby, DE74 2RH
Sampling location	Chapel Hill Farm, Empingham, Rutland.
Date of field sampling	02-August-2000
Pesticide history	No pesticide use in coppice
Land use	Long-established deciduous coppice, used for pheasant-rearing
Depth of sampling	Topsoil from 15 to 30 cm
Method of soil collection	In accordance with ISO10381-6
Date of arrival at CLE	04-August-2000
Handling	The soil was given a unique code, thoroughly mixed and passed through a 2mm mesh sieve. Prior to use the soil was stored air-dry, in the dark, at room temperature, in loosely tied plastic bags.

UK & BBA Particle Size Distribution		pH in H_2O	8.0
Sand % (2000 – 63 µm)	37	(Commenced: 16-8-01)	
Silt % (63 – 2 µm)	29		
Clay % (< 2 µm)	34	pH in 1M KCl	7.6
		(Commenced: 16-8-01)	
USDA Particle Size Distribution			
Sand % (2000 – 50 µm)	38	pH in 0.01M $CaCl_2$	7.6
Silt % (50 – 2 µm)	28	(Commenced: 16-8-01)	
Clay % (< 2 µm)	34		
		Cation Exchange Capacity	38.2
UK Textural Class	Clay loam	mEq/100g	
BBA Textural Class	sandiger toniger Lehm	mmol/Kg	382
USDA Textural Class	Clay loam	(Commenced: 22-8-01)	
Water Holding Capacity at pF 0 (0.001 bar) %	65.4	Nitrogen Content % * (Commenced: 28-3-01)	0.59
Water Holding Capacity at pF 2.5 (0.33 bar) %	29.4	Carbon/Nitrogen Ratio (Commenced: 28-3-01)	1: 0.13
Organic Carbon % (Commenced: 15-8-01)	4.6	Exchangeable Cations * mEq/100g	
		Exchangeable Ca	NA
Organic Matter %	7.9	Exchangeable Mg	NA
(Commenced: 15-8-01)		Exchangeable Na	NA
		Exchangeable K	NA
Exchangeable acidity *	NA	Exchangeable Mn	NA
mEq/100g		(Commenced: 28-03-01)	
(Commenced: 28-03-01)			
		$CaCO_3$ eq. mg/Kg *	187.6
		(Commenced: 28-03-01)	

The analytical procedures* undertaken at Covance Laboratories Ltd., Harrogate were performed in accord with the current national UK GLP Regulations and the current OECD Principles of GLP.
All data is retained in the GLP archive of Covance Laboratories Ltd., Harrogate.
* Analysis performed by National Soil Resource Institute, Silsoe, Bedford (formally known as the Soil Survey and Land Research Centre) (Laboratory Analysis Plan No. SSHQ/034/01 & SSHQ/035/01). No claim is being made for the compliance status of this work.

Analysed by: 15 May 2003

Approved by: 21 May 2003

Quality Assurance: 27 May 2003

Soil SK566696

TEST SYSTEM CERTIFICATE OF ANALYSIS

Test system code	CS01/03
Test system name	SK566696
Collected by	Land Research Associates, Lockington, Derby, DE74 2RH
Sampling location	Grid Reference: SK566696 Warsop, Nottinghamshire
Date of field sampling	14-January-2003
Pesticide history	No pesticide use in coppice
Land use	Grassy clearing at edge of established wood of silver birch, oak etc
Depth of sampling	Topsoil from 12 to 20 cm
Method of soil collection	In accordance with ISO10381-6
Date of arrival at CLE	16-January-2003
Handling	The soil was given a unique code, thoroughly mixed and passed through a 2mm mesh sieve. Prior to use the soil was stored air-dry, in the dark, at room temperature, in loosely tied plastic bags.

UK & BBA Particle Size Distribution		pH in H_2O	5.1
Sand % (2000 – 63 µm)	84	(Commenced: 18-1-03)	
Silt % (63 – 2 µm)	5		
Clay % (< 2 µm)	11	pH in 1M KCl	4.2
		(Commenced: 18-1-03)	
USDA Particle Size Distribution			
Sand % (2000 – 50 µm)	85	pH in 0.01M $CaCl_2$	4.2
Silt % (50 – 2 µm)	4	(Commenced: 18-1-03)	
Clay % (< 2 µm)	11		
		Cation Exchange Capacity	13.4
UK Textural Class	Loamy sand	mEq/100g	
BBA Textural Class	schwach toniger Sand	mmol/Kg	134
USDA Textural Class	Loamy sand	(Commenced: 06-2-03)	
Water Holding Capacity at pF 0 (0.001 bar) %	29.0	Nitrogen Content % * (Commenced: 07-02-03)	0.8
Water Holding Capacity at pF 2.5 (0.33 bar) %	7.1	Carbon/Nitrogen Ratio (Commenced: 07-02-03)	1 : 1
Organic Carbon % (Commenced: 17-1-03)	0.8		
Organic Matter % (Commenced: 17-1-03)	1.4		

The analytical procedures* undertaken at Covance Laboratories Ltd., Harrogate were performed in accord with the current national UK GLP Regulations and the current OECD Principles of GLP.
All data is retained in the GLP archive of Covance Laboratories Ltd., Harrogate.
* Analysis performed by National Soil Resource Institute, Silsoe, Bedford (formally known as the Soil Survey and Land Research Centre) (Laboratory Analysis Plan No. SSHQ/011/03). No claim is being made for the compliance status of this work.

Analysed by: 15 May 2003

Approved by: 21 May 2003

Quality Assurance: 27 May 2003

Soil SK179618

TEST SYSTEM CERTIFICATE OF ANALYSIS

Test system code	CS37/00-628
Test system name	SK179618
Collected by	Land Research Associates, Lockington, Derby, DE74 2RH
Sampling location	Kenslow Farm, Middleton, Derbyshire
Date of field sampling	09-November-2000
Pesticide history	No pesticide use
Land use	Permanent grass field
Depth of sampling	Topsoil from 5 to 20 cm
Method of soil collection	In accordance with ISO10381-6
Date of arrival at CLE	01-December-2000
Handling	The soil was given a unique code, thoroughly mixed and passed through a 2mm mesh sieve. Prior to use the soil was stored air-dry, in the dark, at room temperature, in loosely tied plastic bags.

UK & BBA Particle Size Distribution		pH in H_2O	6.0
Sand % (2000 – 63 µm)	33	(Commenced: 16-8-01)	
Silt % (63 – 2 µm)	47		
Clay % (< 2 µm)	20	pH in 1M KCl	5.8
		(Commenced: 16-8-01)	
USDA Particle Size Distribution			
Sand % (2000 – 50 µm)	34	pH in 0.01M $CaCl_2$	5.6
Silt % (50 – 2 µm)	46	(Commenced: 16-8-01)	
Clay % (< 2 µm)	20		
		Cation Exchange Capacity	24.9
UK Textural Class	Clay loam	mEq/100g	
BBA Textural Class	schwach sandiger Lehm	mmol/Kg	249
USDA Textural Class	Loam	(Commenced: 22-8-01)	
Water Holding Capacity at pF 0 (0.001 bar) %	75.9	Nitrogen Content % * (Commenced: 28-03-01)	0.33
Water Holding Capacity at pF 2.5 (0.33 bar) %	27.3	Carbon/Nitrogen Ratio (Commenced: 28-03-01)	1:0.09
Organic Carbon % (Commenced: 15-8-01)	3.8		
Organic Matter % (Commenced: 15-8-01)	6.6		

The analytical procedures* undertaken at Covance Laboratories Ltd., Harrogate were performed in accord with the current national UK GLP Regulations and the current OECD Principles of GLP.
All data is retained in the GLP archive of Covance Laboratories Ltd., Harrogate.
* Analysis performed by National Soil Resource Institute, Silsoe, Bedford (formally known as the Soil Survey and Land Research Centre) (Laboratory Analysis Plan No. SSHQ/034/01). No claim is being made for the compliance status of this work.

Analysed by: 15 May 2003

Approved by: 21 May 2003

Quality Assurance: 27 May 2003

Emperor Son's Lake

TEST SYSTEM CERTIFICATE OF ANALYSIS

Test system code	CS04/03
Test system name	Site C1 (Emperor's Son Lake)
Collected by	Land Research Associates, Lockington Hall, Lockington, Derby
Sampling location	SK 268699 Emperor's Son Lake, Chatsworth, Derbyshire
Date of field sampling	10-February-2003
Pesticide history	No pesticides used in the catchment
Method of soil collection	Water: Collected into container.
	Sediment: Collected by hand into container.

Water Temperature (°C) just below the water surface	4.2	Water Temperature (°C) 5cm above the sediment	4.3
Oxygen Content (%) just below the water surface	99.0	Oxygen Content (%) 5cm above the sediment	98.8
Water pH	6.34	Redox potential (mV) of water	416.0
Depth of water (cm) above sediment	15-20cm		
Depth of sediment sampled (cm)	0-10cm	Redox potential (mV) of sediment	225

Storage prior to despatch	Stored at 3 - 5°C
Date of arrival at CLE	12-February-2003
Handling	The water-sediment system was given a unique code. Prior to use/despatch the sediment was sieved to 2mm and stored in the dark, in an incubator routinely maintained at 4 ± 2°C.

Sediment Parameters:

UK & BBA Particle Size Distribution		pH in 1M KCl	6.1
Sand % (2000 – 63 µm)	74	(Commenced: 24.02.03)	
Silt % (63 – 2 µm)	9		
Clay % (< 2 µm)	17	pH in 0.01M $CaCl_2$	6.0
		(Commenced: 24.02.03)	
USDA Particle Size Distribution			
Sand % (2000 – 50 µm)	75	Cation Exchange Capacity	13.6
Silt % (50 – 2 µm)	8	mEq/100g	
Clay % (< 2 µm)	17	mmol/Kg	136
		(Commenced: 25.03.03)	
USDA Textural Class	Sandy loam		
UK Textural Class	Sandy loam	Microbial Biomass µg C/g	192.99
BBA Textural Class	mittel toniger Lehm or schwach toniger Sand	(Commenced: 27.03.03)	
		Total Nitrogen mg/kg *	882.0
Organic Carbon %	1.2	%	0.09
(Commenced: 19.02.03)		(Commenced: 04.04.03)	
Organic Matter %	2.1	Total Phosophorus	382.9
(Commenced: 19.02.03)		mg/kg *	
		(Commenced: 04.04.03)	
Water Holding Capacity at pF 0	42.6		
(0.001 bar) %		Carbon/Nitrogen Ratio	1: 0.08
		(Commenced: 19.02.03)	
Water Holding Capacity at pF 2.5	8.1		
(0.33 bar) %		pH in H_2O	6.8
		(Commenced: 24.02.03)	

TEST SYSTEM CERTIFICATE OF ANALYSIS

Test system code	CS04/03
Test system name	Site C1 (Emperor's Son Lake)
Associated Water Parameters:	
Total Nitrogen mg/L * (Commenced: 04.04.03)	7.0
Total Phosophorus mg/L * (Commenced: 04.04.03)	0.6
Dissolved Organic Carbon (DOC) mg/L * (Commenced: 04.04.03)	35.9
Water Hardness mg/L as $CaCO_3$ * (Commenced: 04.04.03)	41.0
Suspended Solids mg/L * (Commenced: 04.04.03)	210.0

The analytical procedures* undertaken at Covance Laboratories Ltd., Harrogate were performed in accord with the current national UK GLP Regulations and the current OECD Principles of GLP.
All data is retained in the GLP archive of Covance Laboratories Ltd., Harrogate.
* Analysis performed by National Soil Resource Institute, Silsoe, Bedford (formally known as the Soil Survey and Land Research Centre) (Laboratory Analysis Plan No. SSHQ 025/03). No claim is being made for the compliance status of this work. NSRI is a member of the UK GLP compliance programme and the work supplied to Covance complies with the UK GLP regulations.

Analysed by: 27-1-04

Approved by: 30 January 2004

Quality Assurance: 11·2·2004

Eurosoil 5

TEST SYSTEM CERTIFICATE OF ANALYSIS

Test system code	CS55/03 and CS57/03
Test system name	Eurosoil 5 (IRMM-443-5)
Collected by	IRRM (Institute for Reference Materials and Measurements)
Date of arrival at CLE	22/10/03 and 30/10/03
Handling	The soil was given a unique code on arrival. The soil was supplied as an air dried fine soil <2mm in batches of 200g. Each batch supplied by IRMM is from the same bulk sample collected in 1991. The two batches received have been treated as being the same despite having different Covance batch numbers.

UK & BBA Particle Size Distribution		pH in H_2O^+ (Commenced: Dec 2001)	4.1
Sand % (2000 – 63 µm)	86		
Silt % (63 – 2 µm)	9		
Clay % (< 2 µm)	5	pH in 0.01M $CaCl_2^+$ (Commenced: Dec 2001)	3.1
USDA Particle Size Distribution			
Sand % (2000 – 50 µm)	87	Cation Exchange Capacity mEq/100g (Commenced: 18/12/03)	26.8
Silt % (50 – 2 µm)	8		
Clay % (< 2 µm)	5		
UK Textural Class	Loamy sand	Nitrogen Content % * (Commenced: 13-01-04)	0.2
BBA Textural Class	schwach toniger Sand or reiner Sand		
USDA Textural Class	Loamy sand		
Water Holding Capacity at pF 0 (0.001 bar) %	46.9	Carbon/Nitrogen Ratio (Commenced: 16/12/03)	1 : 0.03
Water Holding Capacity at pF 2.5 (0.33 bar) %	10.3		
Organic Carbon % (Commenced: 16/12/03)	6.7		
Organic Matter % (Commenced: 16/12/03)	11.6		

The analytical procedures*+undertaken at Covance Laboratories Ltd., Harrogate were performed in accord with the current national UK GLP Regulations and the current OECD Principles of GLP.
All data is retained in the GLP archive of Covance Laboratories Ltd., Harrogate.

* Analysis performed by National Soil Resource Institute, Silsoe, Bedford (Laboratory Analysis Plan No. NSRI/001/04). No claim is being made for the compliance status of this work. NSRI is a member of the UK GLP compliance programme and the work supplied to Covance complies with the UK GLP regulations.

+Analysis supplied by IRMM. No claim is being made for the compliance status of this work.

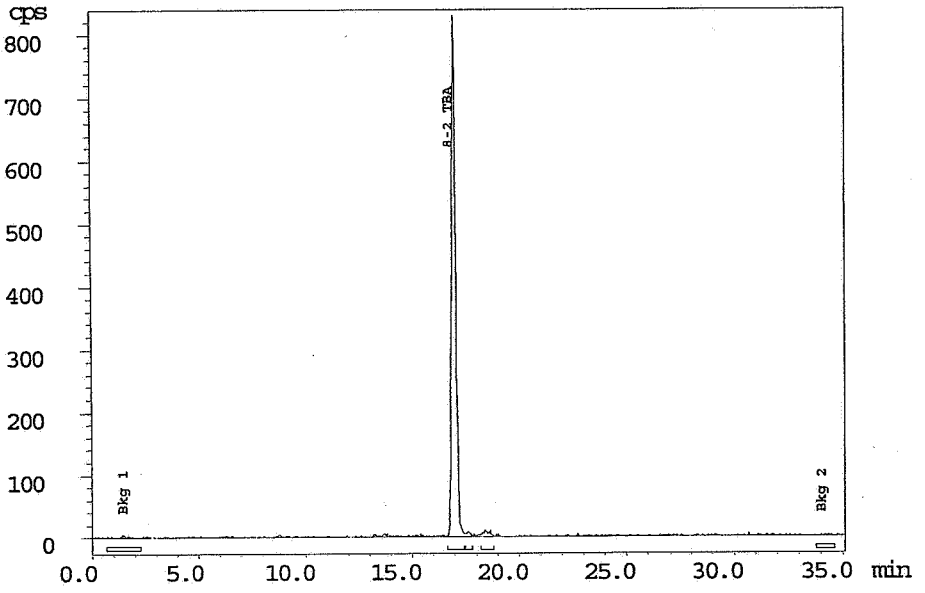
Analysed by: 11-02-04

Approved by: 11 February 2004

Quality Assurance: 11·2·2004

Appendix 3
Radiochemical purity of (^{14}C)-8-2 TBA and formulation stability

Radiochemical Purity by HPLC (stock solution 1)



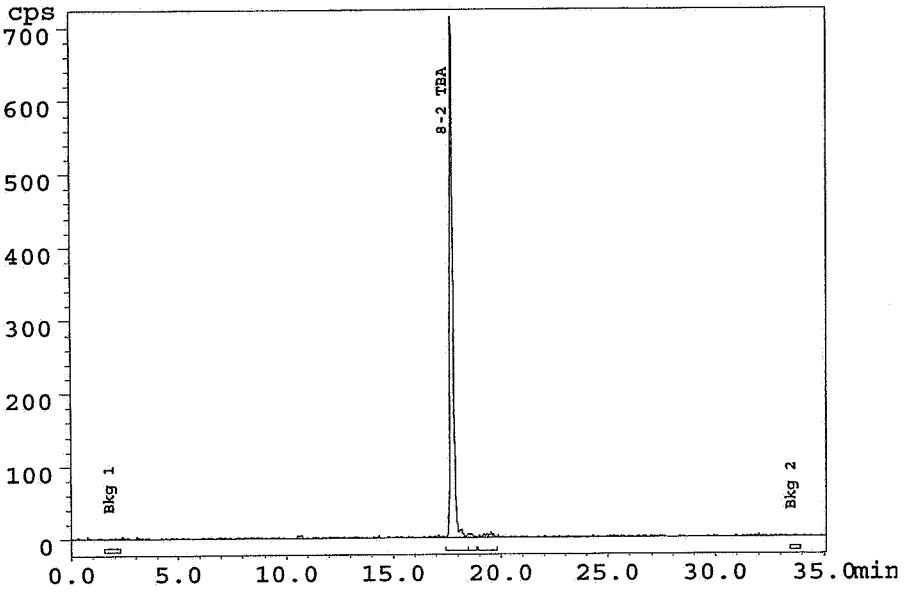
Method: METHOD1 Filename: 4CHR001.R01 Evaluation: A User: S.Swales
Channel: C-14 Detector:

Name	Start - End (m)	RT (m)	Height (cps)	Area (Counts)	%Total (%)
Bkg 1	0.7- 2.3	1.5	0.3		
TBA	16.6- 17.4	17.0	854.8	10931.4	98.03
	17.5- 17.8	17.6	7.8	55.6	0.50
	18.2- 18.8	18.4	10.8	155.4	1.39
Bkg 2	33.7- 34.6	34.1	0.1		
3 Peaks				11142.4	99.92

Bkg Area	395.5	Counts
Total Area	11151.5	Counts
Unallocated	9.0	Counts (0.08 %)

Date of analysis: 4 April 2003

Radiochemical Purity by HPLC (stock solution 1)



Method: METHOD1 Filename: CHR014.R01 Evaluation: A User: S.Swales
Channel: C-14 Detector:

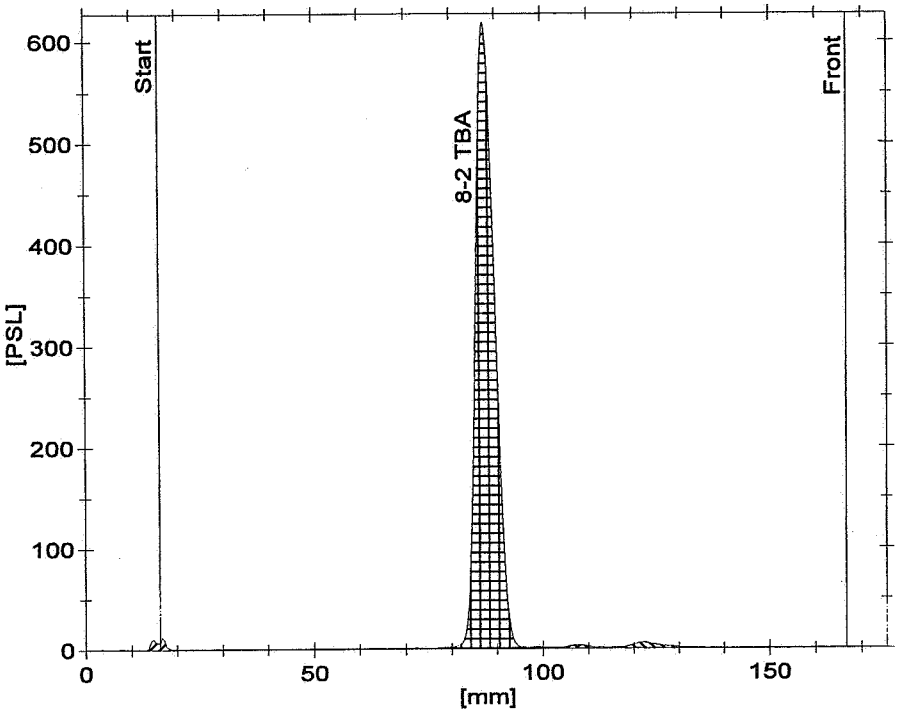
Name	Start	End (m)	RT (m)	Height (cps)	Area (Counts)	%Total (%)
Bkg 1	1.6-	2.3	1.9	0.2		
8-2 TBA	17.4-	18.5	17.7	718.8	7338.1	98.14
	18.5-	18.9	18.5	5.8	48.9	0.65
	18.9-	19.8	19.5	6.8	86.1	1.15
Bkg 2	33.4-	33.9	33.6	0.2		
3 Peaks					7473.1	99.94

Bkg Area	429.6 Counts	
Total Area	7477.4 Counts	
Unallocated	4.4 Counts	(0.06 %)

Date of analysis: 19 December 2003
Prior to formulation of dose formulations for the isotherms test.

Radiochemical Purity by TLC (stock solution 1)

Plate 6, track 3

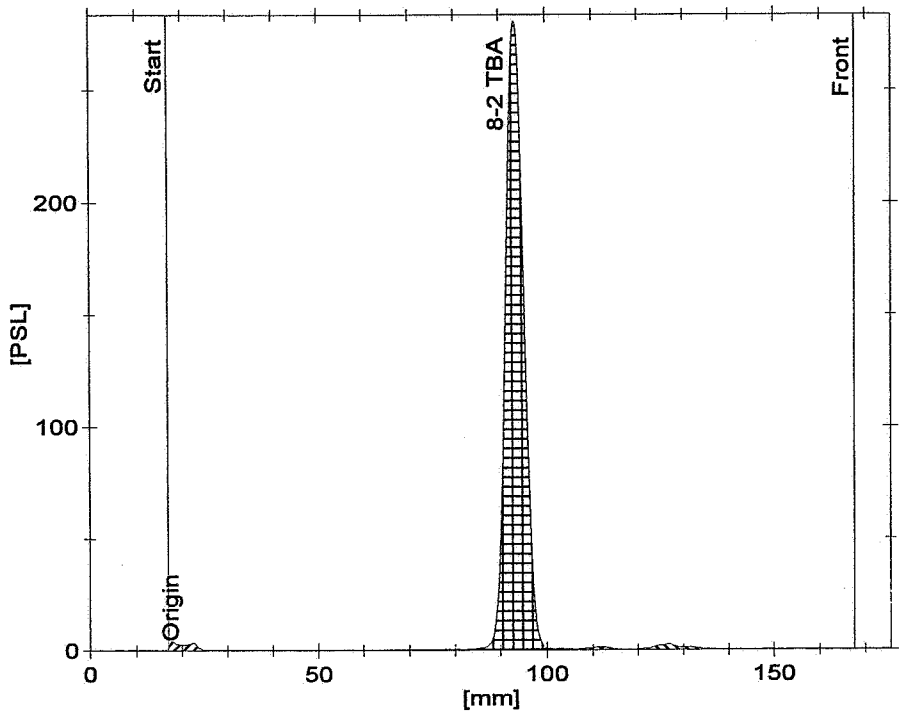


No	Name	Type	Pos [mm]	RF	PSL	PSL-Bkg	%(PSL-Bkg)
1			16.00	--	174.46	167.34	1.07
2	8-2 TBA		87.80	0.48	15044.37	15014.08	96.14
3			107.80	0.61	94.37	84.95	0.54
4			123.40	0.71	227.07	210.27	1.35
5		Bkg	6.00	--	8.45	0.00	0.00
6		Bkg	169.60	--	6.57	0.00	0.00
-		Sum	--	--	15540.27	15476.64	99.10
-		Rem	--	--	300.72	140.87	0.90
-		Ttl	--	--	15840.99	15617.51	100.00

Date of analysis: 7 April 2003

Radiochemical Purity by TLC (stock solution 1)

Plate 23, track 3



No	Name	Type	Pos [mm]	RF	PSL	PSL-Bkg	%(PSL-Bkg)
1	Origin		20.20	0.02	104.36	97.58	1.47
2	8-2 TBA		93.40	0.51	6424.60	6399.84	96.33
3			112.20	0.63	46.38	36.44	0.55
4			127.60	0.73	102.61	88.89	1.34
5		Bkg	5.80	--	5.25	0.00	0.00
6		Bkg	169.20	--	4.05	0.00	0.00
-		Sum	--	--	6677.96	6622.76	99.69
-		Rem	--	--	104.00	20.73	0.31
-		Ttl	--	--	6781.95	6643.49	100.00

Date of analysis: 19 December 2003
Prior to formulation of dose formulations for the isotherms test.

Appendix 4
Chromatography of reference standard

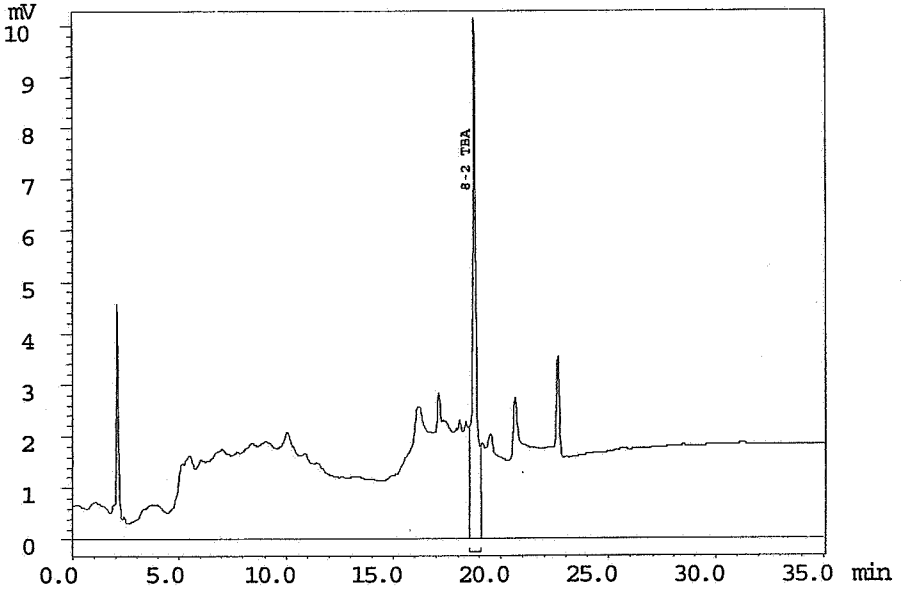
Retention times and Rf values of 8-2 TBA by HPLC and TLC

Reference Standard	Retention time by UV detection (min)	Rf
TBA	18.7	0.49

[1] HPLC Chromatogram used is 03CHR002.R01(UV)
[2] TLC Plate used is Plate 7 (non-radiolabelled values quoted)

HPLC and TLC chromatographic conditions are described on page 27.

The retention time of the UV peak was *ca* 0.1 min before the retention time of the corresponding ^{14}C peak due to the positioning of the detectors. The retention time of the reference standard varied slightly depending on the nature of the samples and the amount of material chromatographed. HPLC chromatogram 03CHR002.R01 is shown below:



Appendix 5
Minor deviations from protocol

The study supervisor changed from J Cross to K Dixon and the study co-ordinator changed from S Irlam to C Unsworth.

Soil SK179618 was classified as being a clay loam in the protocol. However, this was the UK textural classification not USDA as stated. The USDA textural classification is loam as indicated by the certificate of analysis.

The amount of test substance in the supernatant was not determined during the stability test because the radioactivity remaining in the adsorption supernatants was too low to enable any form of analysis to be performed. The amount of test substance was determined in the soil extracts only.

The five concentrations selected for the adsorption isotherms test did not cover two orders of magnitude. The test compound had a low water solubility (0.14 mg/L) and was highly adsorbed to the solid phase (<3% remaining in the aqueous phase at the adsorption equilibrium time). These parameters made it impossible to cover two orders of magnitude and still be able to detect radioactivity in the adsorption supernatants. The adsorption isotherms test was , therefore, conducted over one order of magnitude.

These deviations do not affect the scientific integrity of the study.

Appendix 6
Example calculations

Dose solution concentration calculations

The spreadsheet below shows how the concentration of (^{14}C)-8-2 TBA was determined in the dose solutions applied to soils:

Source file: EF_DOSE.XLS Version 1
File name: I:\D2142\Study Data\2304-001\Data\[SpAc 16.xls]Report

Study details

Study number: 2304/1
Experiment type:Adsorption/ Desorption
Dosing occasion:Main Isotherms- highest concentration
Dosing vehicle:Ethanol
Radiolabelled test article HD and Lot number(s):319/03-2304 Lot 1
Analysis date: 22/12/03

Assay details

Test article specific radioactivity (MBq/mg):	**2.63**						
Formulation quantities in weight / volume:	**V**						
LSC aliquot quantities in weight / volume:	**V**	Volume of LSC aliquots (mL):			**0.25**		
Number of LSC replicates:	3	Number of flasks:			6		
Flask number (identity):	**Spac 1**	**Spac 2**	**Spac 3**	**Spac 4**	**Spac 5**	**Spac 6**	
Volume of formulation aliquot (mL):	**0.05**	**0.05**	**0.05**	**0.05**	**0.05**	**0.05**	
Diluted to volume (mL):	**5**	**5**	**5**	**5**	**5**	**5**	
Radioactivity in aliquots (dpm) 1:	**51901**	**52575**	**51561**	**51438**	**50954**	**50836**	
2:	**51857**	**52454**	**51393**	**51221**	**50857**	**51035**	
3:	**51544**	**52393**	**51527**	**51440**	**50827**	**50752**	
Background radioactivity (dpm):	**32**						
Background corrected dpm/mL 1:	207476	210172	206116	205624	203688	203216	
2:	207300	209688	205444	204756	203300	204012	
3:	206048	209444	205980	205632	203180	202880	
kBq/mL of formulation:	345	350	343	342	339	339	
SD:	1	1	1	1	0	1	
CV:	0.38%	0.18%	0.17%	0.25%	0.13%	0.29%	
Flasks omitted from mean marked with X:							
Values used:	345	350	343	342	339	339	
n:	6						
Sum:	2058						
Mean kBq/mL of formulation:	343						
SD:	4						
CV:	1.17%						

Volume of formulation applied (mL):	**0.0086**
Radioactivity applied (µCi):	0.079715
Radioactivity applied (kBq):	2.949446
Weight of test article applied (mg):	0.001121

Determination of applied radioactivity

The following spreadsheet shows how the percent of applied radioactivity was determined in soil extracts. The same spreadsheet was used for quantification of combusted residues.

Source file: R_RCALC.XLS Version 1
File name: I:\D2142\Study Data\2304-001\Data\Isotherms\[Adsorb supernatant DG B.xls]Sheet1

ASSAY DETAILS
Covance study number: **2304/1**
Sample description: **Adsorb supernatant DG B**
Number of LSC replicates: **2**
Flag variability at x% of mean: **10%**
LOD (times background): **1.5** LOD based on mean counts
Number of samples (10 max) 10

LSC DATA INPUT (dpm) Subject code:	B1	B2	B3	B4	B5	B6	B7	B8	B9	B10
SDATA file input										
1	0	0	0	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0	0	0	0
Background	0	0	0	0	0	0	0	0	0	0
Manual input										
1	397	393	336	279	214	223	251	207	72	69
2	384	403	356	313	205	238	256	246	74	71
Background	25	25	32	32	20	20	24	24	24	24
WEIGHT DATA INPUT (g)										
Weight of empty pot										
Weight of pot + sample	6.167	6.192	6	6.192	6.079	6.176	6.047	6.148	6.123	6.036
Total assayed sample weight	6.167	6.192	6	6.192	6.079	6.176	6.047	6.148	6.123	6.036
LSC aliquot weights										
1	0.997	1.001	0.994	0.993	0.995	0.995	1.477	1.367	1.395	1.397
2	0.999	1.004	0.996	0.993	0.986	1.003	1.437	1.466	1.433	1.393
REFERENCE DATA										
Dose / Reference (kBq) ... (A)	2.949	2.949	2.366	2.366	1.783	1.783	1.194	1.194	0.3054	0.3054
Weight of sub-sample (g)										
Initial sample weight (g)										
Equivalent dose (kBq) ... (B)										
Specific activity (MBq/mg):	2.63	2.63	2.63	2.63	2.63	2.63	2.63	2.63	2.63	2.63
RESULTS										
Background corrected dpm										
1	372	368	304	247	194	203	227	183	48	45
2	359	378	324	281	185	216	232	222	50	47
Dpm per g										
1	373.119358	367.632368	305.83501	248.741188	194.974874	204.020101	153.689912	133.869788	34.4086022	32.2118826
2	359.359359	376.494024	325.301205	282.980866	187.626775	215.353938	161.44746	151.432469	34.8918353	33.7401292
Mean dpm per g	366	372	316	266	191	210	158	143	35	33
Concentration (µg/g)	2.321E-03	2.358E-03	2.000E-03	1.685E-03	1.212E-03	1.329E-03	9.985E-04	9.040E-04	2.196E-04	2.090E-04
Variability limit exceeded?	NO	NO	NO	NO	NO	NO	NO	NO	NO	NO
LSC input method	Manual	Manual	Manual	Manual	Manual	Manual	Manual	Manual	Manual	Manual
Radioactivity recovered										
dpm in total sample	2259	2304	1893	1646	1163	1295	953	877	212	199
kBq in total sample	0.0376	0.0384	0.0316	0.0274	0.0194	0.0216	0.0159	0.0146	0.0035	0.0033
dpm in sub-sample	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
kBq in sub-sample	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
% Recovery of radioactivity	1.3	1.3	1.3	1.2	1.1	1.2	1.3	1.2	1.2	1.1
LOD (%)	0	0	0.1	0.1	0.1	0.1	0.1	0.1	0.3	0.3
based on reference	A	A	A	A	A	A	A	A	A	A
Mean % recovery	1.3		1.3		1.2		1.3		1.2	
Sample concentration (µg/g)	2.321E-03	2.358E-03	2.000E-03	1.685E-03	1.212E-03	1.329E-03	9.985E-04	9.040E-04	2.196E-04	2.090E-04

Adsorption coefficient calculations

The following spreadsheet shows how the adsorption coefficients were calculated:

AdsorptionDesorption.XLS Version 3.0 (Microsoft Excel 97)
File name:
Study number: **2304-001**
Treatment: **Soil B**
% Organic carbon: **0.8**
% Organic matter: **1.4**
Assumed solution density (g/mL): **1**

Concentration	Replicate	m	V_0	G	W_2	W_3	C_e	C_1	V_2	V_3	X	%A	X_1	%D	%ND	X/m	X_1/m	K_d	K_{doc}
0.125	**B1**	**9.014**	**9.232**	**1.121**	**6.167**	**6.136**	**0.002321**	**0.002045**	6.167	6.136	1.09957253	98.08854	1.08787035	1.06424812	98.9357519	0.12198497	0.12068675	52.5570749	6569.63437
0.125	**B2**	**8.999**	**9.266**	**1.121**	**6.192**	**6.275**	**0.002358**	**0.002457**	6.192	6.275	1.09915077	98.050916	1.08342877	1.43037711	98.5696229	0.12214143	0.12039435	51.7987425	6474.84281
0.1	**B3**	**9.019**	**9.12**	**0.9**	**6**	**6.028**	**0.002**	**0.001459**	6	6.028	0.88176	97.973333	0.87465307	0.80599392	99.1940061	0.09776694	0.09697894	48.8834682	6110.43353
0.1	**B4**	**9.017**	**9.33**	**0.9**	**6.192**	**6.216**	**0.001685**	**0.001419**	6.192	6.216	0.88427895	98.253217	0.87629315	0.90308562	99.0969144	0.09806798	0.09718234	58.2005799	7275.07249
0.075	**B5**	**9.002**	**9.24**	**0.678**	**6.079**	**6.122**	**0.001212**	**0.0009592**	6.079	6.122	0.66680112	98.348248	0.661728	0.7608148	99.2391852	0.07407255	0.073509	61.1159676	7639.49595
0.075	**B6**	**8.993**	**9.256**	**0.678**	**6.176**	**6.165**	**0.001329**	**0.0009755**	6.176	6.165	0.66569878	98.18566	0.6607736	0.73985077	99.2601492	0.0740241	0.07347644	55.6991008	6962.3876
0.05	**B7**	**8.993**	**9.244**	**0.454**	**6.047**	**5.93**	**0.0009985**	**0.0009743**	6.047	5.93	0.44476987	97.966931	0.43906963	1.28161371	98.7183863	0.04945734	0.04882349	49.5316383	6191.45478
0.05	**B8**	**8.996**	**9.31**	**0.454**	**6.148**	**6.175**	**0.000904**	**0.0007586**	6.148	6.175	0.44558376	98.146203	0.44135916	0.94810462	99.0518954	0.04953132	0.04906171	54.7912838	6848.91048
0.0125	**B9**	**9.033**	**9.337**	**0.116**	**6.123**	**6.092**	**0.0002196**	**0.0002418**	6.123	6.092	0.11394959	98.232409	0.1124052	1.35533295	98.6446671	0.01261481	0.01244384	57.4444981	7180.56226
0.0125	**B10**	**8.991**	**9.22**	**0.116**	**6.036**	**6.35**	**0.000209**	**0.0002241**	6.036	6.35	0.11407302	98.33881	0.11260191	1.28962431	98.7103757	0.01268747	0.01252385	60.705586	7588.19825

Bookmark code	Description	Bookmark
bm01	Study Number	2304/001
bm02	Study Title	(^{14}C)-8-2 Telomer B Alcohol: Adsorption/Desorption using a Batch Equilibrium Method
bm03	Report Status	Draft
bm04	Study Director	S Swales
bm05	Company name of sponsor	Katie Smythe, Administrator
bm06	Sponsor address	Telomers Research Program The Rand Corporation 1200 South Hayes Street Arlington, VA22202 USA
bm08	Test article	8-2 TBA
bm08a	test article	8-2 TBA
bm09	chemical name	2-perfluorooctylethanol
bm01a	OECD Guideline	
bm13	UK GLP Guideline	United Kingdom Statutory Instrument 1999 No. 3106, The Good Laboratory Practice Regulations 1999
bm14	OECD GLP Guideline	OECD Principles of Good Laboratory Practice (revised 1997, Issued Jan 1998) ENV/MC/CHEM(98)17
bm16	Report issue date	
bm17	Protocol signed date	5 November 2001
bm18	First practical work date	23 January 2002
bm19	Last practical work date	