

DIOXIN/FURAN  
ANALYSIS REPORT  
NUMBER D7910



This analysis is based on Northumbrian Water Scientific Services Organics Laboratory method O-36 which is accredited under the **UKAS** accreditation scheme.

**DIOXIN/FURAN**  
**ANALYSIS REPORT**  
**NUMBER D7910**

**Laboratory Address:**

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**Report Ref:** D7910

**Client Address:**

Victoria Smith  
Hanson Cement  
Padeswood  
Mold  
Flintshire  
CH7 4HB

**Prepared by:** Sophie Wright

**Signed:**

A handwritten signature in black ink, appearing to be 'Sophie Wright'.

**Date:** 18/05/16

**Issued under the authority of Steve Wilson – Laboratory Manager (Howdon Organics)**

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## SECTION 1

## SUMMARY

One sample taken on behalf of Hanson Cement was analysed for dioxin and furan contamination. Analysis of the sample gave the following TEQ values on an air dried basis (to 2 significant figures).-

Sample	Toxic Equivalent Results ng/kg				
	NATO/CCMS ITEQ	WHO (1998) HUMANS TEQ	WHO (2005) HUMANS TEQ	WHO (1998) FISH TEQ	WHO (1998) BIRDS TEQ
BYPASS DUST	6.9	6.6	6.5	7.5	6.4

The total (tetra to octa chlorinated) dioxin/furan content of the sample is indicated below on an air dried basis (to 2 significant figures):-

Sample	Total Results ng/kg
BYPASS DUST	770

Full results for individual congeners and homologue group totals together with information on any deviations from methodology/quality systems is shown in section 2.

## INTRODUCTION

One sample was submitted to Northumbrian Water Scientific Services by Hanson Cement for analysis to determine the levels of polychlorinated dibenzo-p-dioxins (PCDD's) and polychlorinated dibenzofurans (PCDF's) present.

The analysis covered the 17 PCDD and PCDF congeners containing chlorine substitution at the 2,3,7 and 8 positions so that Toxicity Equivalents could be calculated based on the NATO/CCMS and WHO systems.

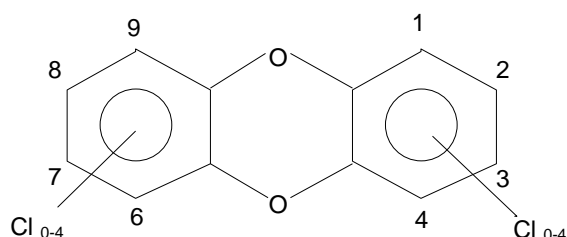
The sample was received on 25/05/16. Details of the sample are shown below.

**CONTRACT** - HANSONCEM-00244

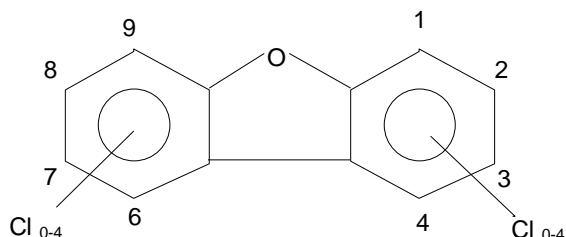
DATE REC'D	SAMPLE	LAB N°	REF
25/05/16	BYPASS DUST	1208210	D7910

## DIOXINS AND FURANS

PCDDs and PCDFs form a group of 210 closely related substances. The structural formulae are shown below. A dioxin or furan molecule can have as few as one or as many as eight chlorine atoms attached to the molecule at any of eight locations. The combination of number and position of the chlorine atoms gives rise to 75 possible dioxins and 135 furans.

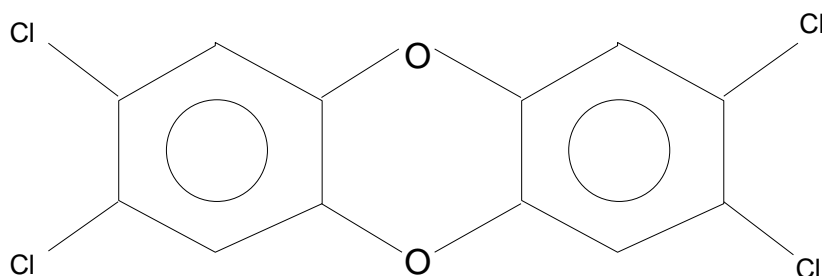


A Dioxin Molecule



A Furan Molecule

It is both the number of chlorine atoms and position in the molecule that determines the physical and chemical properties, as well as the toxicity of a given dioxin or furan. The most hazardous dioxin has chlorine substitution at the 2,3,7 and 8 positions and is therefore called 2,3,7,8-tetrachlorodibenzo-p-dioxin.



2,3,7,8-Tetrachlorodibenzo-p-dioxin

## TOXIC EQUIVALENTS

In order to assess the toxicity of complex mixtures of PCDDs, PCDFs and PCBs the concept of toxic equivalents was devised. Toxic Equivalent Factors (TEF) are assigned to individual dioxins, furans and PCBs on the basis of how toxic they are in comparison with 2,3,7,8-TCDD, the most potent dioxin which has been assigned a value of 1.0. By comparison, animal and cell tests show that 2,3,7,8-TCDF is approximately one-tenth as toxic as 2,3,7,8-TCDD. Consequently its toxic equivalent factor is 0.1.

Of the 210 dioxins and furans, 17 contribute most to the toxicity of a complex mixture and are of most concern. Of the 209 PCBs 12 contribute most to the toxicity of a complex mixture and are of most concern. Therefore it is these 29 compounds that have TEFs assigned shown in the table below for various schemes.

TEF tables	NATO/CCMS	WHO (1998)	WHO (2005)	WHO (1998)	WHO (1998)
Congener		Humans/ mammals	Humans/ mammals	Fish	Birds
2,3,7,8-TCDF	0.1	0.1	0.1	0.05	1
2,3,7,8-TCDD	1.0	1	1	1	1
1,2,3,7,8-PeCDF	0.05	0.05	0.03	0.05	0.1
2,3,4,7,8-PeCDF	0.5	0.5	0.3	0.5	1
1,2,3,7,8-PeCDD	0.5	1	1	1	1
1,2,3,4,7,8-HxCDF	0.1	0.1	0.1	0.1	0.1
1,2,3,6,7,8-HxCDF	0.1	0.1	0.1	0.1	0.1
2,3,4,6,7,8-HxCDF	0.1	0.1	0.1	0.1	0.1
1,2,3,7,8,9-HxCDF	0.1	0.1	0.1	0.1	0.1
1,2,3,4,7,8-HxCDD	0.1	0.1	0.1	0.5	0.05
1,2,3,6,7,8-HxCDD	0.1	0.1	0.1	0.01	0.01
1,2,3,7,8,9-HxCDD	0.1	0.1	0.1	0.01	0.1
1,2,3,4,6,7,8-HpCDF	0.01	0.01	0.01	0.01	0.01
1,2,3,4,7,8,9-HpCDF	0.01	0.01	0.01	0.01	0.01
1,2,3,4,6,7,8-HpCDD	0.01	0.01	0.01	0.001	<0.001*
OCDF	0.001	0.0001	0.0003	0.0001	0.0001
OCDD	0.001	0.0001	0.0003	<0.0001*	0.0001
PCB BZ 81	-	0.0001	0.0003	0.0005	0.1
PCB BZ 77	-	0.0001	0.0001	0.0001	0.05
PCB BZ 123	-	0.0001	0.00003	<0.000005*	0.00001
PCB BZ 118	-	0.0001	0.00003	<0.000005*	0.00001
PCB BZ 114	-	0.0005	0.00003	<0.000005*	0.0001
PCB BZ 105	-	0.0001	0.00003	<0.000005*	0.0001
PCB BZ 126	-	0.1	0.1	0.005	0.1
PCB BZ 167	-	0.00001	0.00003	<0.000005*	0.00001
PCB BZ 156	-	0.0005	0.00003	<0.000005*	0.0001
PCB BZ 157	-	0.0005	0.00003	<0.000005*	0.0001
PCB BZ 169	-	0.01	0.03	0.00005	0.001
PCB BZ 189	-	0.0001	0.00003	<0.000005*	0.00001

\* NB Where < figure is quoted for TEF the actual figure is used in all calculations in this report giving a worst case scenario.



## **METHOD**

The analytical method used for the analysis is based on the American EPA Method 1613.

### **SPIKING AND EXTRACTION**

Air dried soil samples are ground, spiked with a mixture of 16 stable isotopically labelled standards, (see following page for spiking scheme), mixed and allowed to equilibrate. The samples are then soxhlet extracted for a minimum of 16 hours with toluene. Ash samples are pre-treated with hydrochloric acid before being spiked and extracted as above.

### **CLEANUP AND ANALYSIS**

The extracts are washed with concentrated sulphuric acid and aqueous potassium hydroxide if particularly dirty before being dried and concentrated. Impurities are removed from the extracts by column chromatography using silica and alumina columns.

The resulting extracts are concentrated and solvent exchanged to give a final volume of 10 µl in nonane.

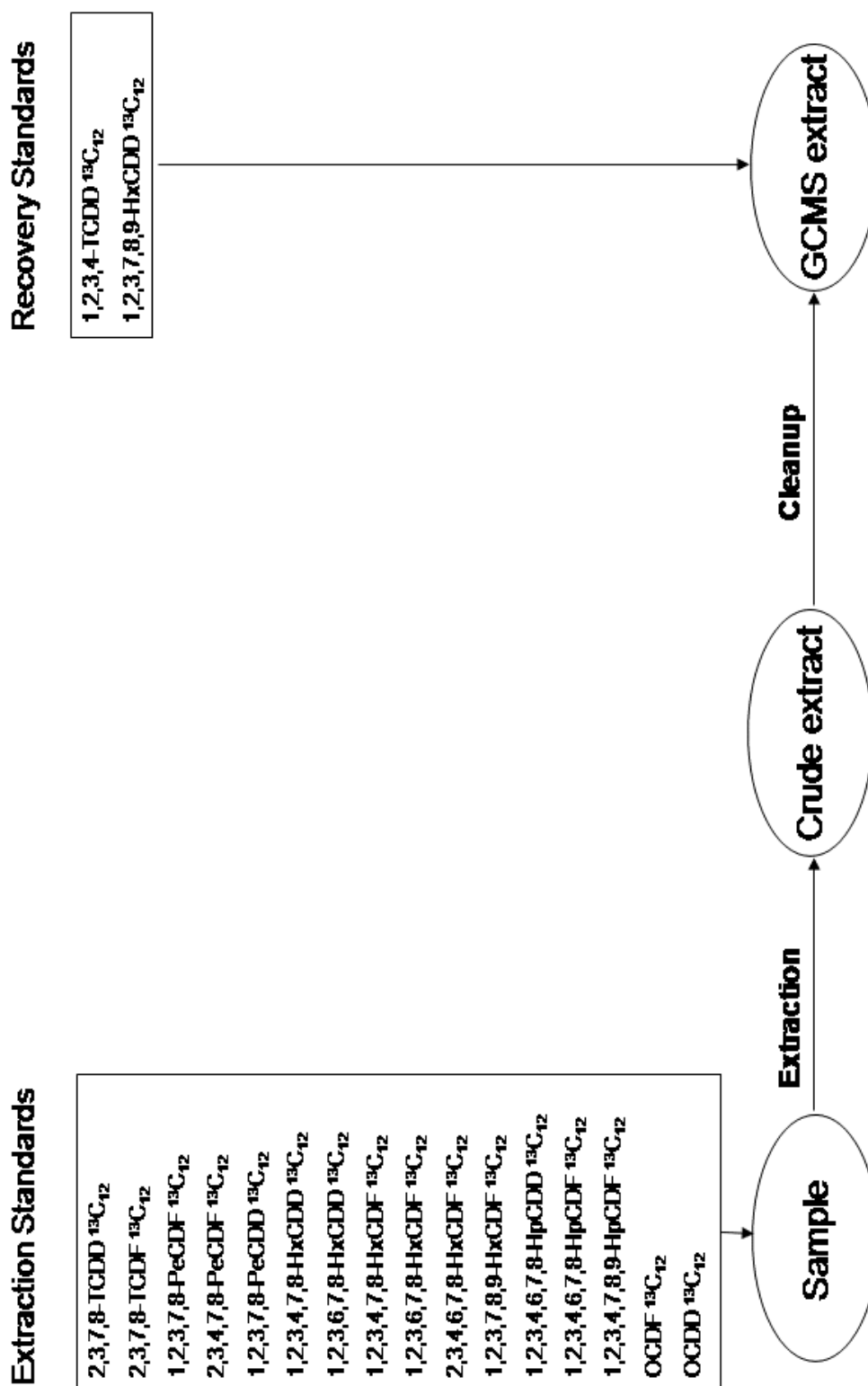
Two isotopically labelled internal standards are added before analysis by high resolution gas chromatography- high resolution mass spectrometry (HRGC-HRMS) using an isotope dilution method for those 2,3,7,8 isomers for which labelled analogues are available and an internal standard method for the non 2,3,7,8 substituted isomers and totals.

Identification of the dioxins and furans is based on comparison of GC retention time ranges and the ion abundance ratios of the m/z's with the corresponding retention time ranges of authentic standards and the theoretical ion abundance ratios of the exact m/z's.

If results for individual targeted dioxins/furans (i.e. 2,3,7,8 substituted) exceed the calibration range of the instrument then these results are flagged in the analytical report.

The mass spectrometer is operated at a resolution of  $\geq 10000$  to minimize the potential for interference. Selected ions characteristic of the PCDDs and PCDFs are monitored together with ions characteristic of polychlorinated diphenyl ethers which may interfere with the PCDFs. The mass spectrometer is continuously calibrated during acquisition to correct for any mass drift by allowing a reference compound FC43 to bleed into the system.

## EPA 1613 LABELLED STANDARD ADDITIONS (AES O36)



## SECTION 2

## RESULTS

The following pages contain the detailed analytical results for the isomer specific analysis for each sample and blank along with homologue group totals and recovery information for the labelled standards.

A matrix blank is analysed alongside the samples to show any possible contamination. This is normally a sample of dioxin-free quartz sand for solid samples.

The following points should be noted.

- All results are on a dry weight basis.
- Results have not been blank corrected. Results have not been rounded. This is to permit further processing if necessary and does not imply the level of accuracy. Summary results on page 5 have been rounded to two significant figures.
- n.d. - not detected – Detection limits for the analysis are calculated on a sample specific basis by the GCMS software, and are based on a signal to noise value of 2.5 to 1
- Results marked \* are over the normal calibration limit of the method.
- The total results for TCDD and TCDF homologue groups do not include the 1,2,8,9 isomers due to the fact that these components elute prior to the first of the PeCDFs on the DB5-MS column used. Acquisition windows have been set up to acquire the first eluting PeCDF in preference to the two tetrachlorinated isomers identified above.
- All quality criteria in the method O36 have been met with any deviations outlined below.

**Deviations from methodology/quality criteria/comments: -none.**

## DIOXIN-FURAN RESULTS

CLIENT HANSON CEMENT

CONTRACT HANSONCEM-00244

SAMPLE LABORATORY BLANK

LAB NO N/A

REF D7910

CONGENER	ng/kg	NATO	WHO TEQ			
		I-TEQ	Humans <sup>a</sup>	Humans <sup>b</sup>	Fish <sup>a</sup>	Birds <sup>a</sup>
2,3,7,8-TCDF	<0.082	n.d.	n.d.	n.d.	n.d.	n.d.
2,3,7,8-TCDD	<0.064	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,7,8-PeCDF	<0.127	n.d.	n.d.	n.d.	n.d.	n.d.
2,3,4,7,8-PeCDF	<0.132	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,7,8-PeCDD	<0.138	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,4,7,8-HxCDF	<0.132	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,6,7,8-HxCDF	<0.125	n.d.	n.d.	n.d.	n.d.	n.d.
2,3,4,6,7,8-HxCDF	<0.128	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,7,8,9-HxCDF	<0.17	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,4,7,8-HxCDD	<0.179	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,6,7,8-HxCDD	<0.186	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,7,8,9-HxCDD	<0.18	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,4,6,7,8-HpCDF	<0.069	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,4,7,8,9-HpCDF	<0.104	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,4,6,7,8-HpCDD	<0.253	n.d.	n.d.	n.d.	n.d.	n.d.
OCDF	<0.093	n.d.	n.d.	n.d.	n.d.	n.d.
OCDD	<0.217	n.d.	n.d.	n.d.	n.d.	n.d.
	<b>TOTAL</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.0000</b>

TOTALS	ng/kg
TCDFs	0.1452
TCDDs	n.d.
PeCDFs	n.d.
PeCDDs	n.d.
HxCDFs	0.3007
HxCDDs	n.d.
HpCDFs	n.d.
HpCDDs	n.d.
OCDF	n.d.
OCDD	n.d.
<b>TOTAL</b>	<b>0.4459</b>

<sup>a</sup> WHO 1998 TEQ values, <sup>b</sup> WHO 2005 TEQ values

**LABELLED STANDARD RECOVERIES**

**CLIENT** HANSON CEMENT

**CONTRACT** HANSONCEM-00244

**SAMPLE** LABORATORY BLANK

**LAB NO** N/A

**REF** D7910

EXTRACTION STANDARDS	RECOVERY %
<sup>13</sup> C <sub>12</sub> 2378 TCDF	79
<sup>13</sup> C <sub>12</sub> 2378 TCDD	81
<sup>13</sup> C <sub>12</sub> 12378 PeCDF	111
<sup>13</sup> C <sub>12</sub> 23478 PeCDF	116
<sup>13</sup> C <sub>12</sub> 12378 PeCDD	113
<sup>13</sup> C <sub>12</sub> 123478 HxCDF	85
<sup>13</sup> C <sub>12</sub> 123678 HxCDF	86
<sup>13</sup> C <sub>12</sub> 234678 HxCDF	88
<sup>13</sup> C <sub>12</sub> 123789 HxCDF	87
<sup>13</sup> C <sub>12</sub> 123478 HxCDD	88
<sup>13</sup> C <sub>12</sub> 123678 HxCDD	88
<sup>13</sup> C <sub>12</sub> 1234678 HpCDF	86
<sup>13</sup> C <sub>12</sub> 1234789 HpCDF	73
<sup>13</sup> C <sub>12</sub> 1234678 HpCDD	72
<sup>13</sup> C <sub>12</sub> OCDF	57
<sup>13</sup> C <sub>12</sub> OCDD	56

All of the recoveries quoted above are within the acceptance limits of method O36.

## DIOXIN-FURAN RESULTS

CLIENT HANSON CEMENT

CONTRACT HANSONCEM-00244

SAMPLE BYPASS DUST

LAB NO 1208210

REF D7910

CONGENER	ng/kg	NATO	WHO TEQ			
		I-TEQ	Humans <sup>a</sup>	Humans <sup>b</sup>	Fish <sup>a</sup>	Birds <sup>a</sup>
2,3,7,8-TCDF	<0.243	n.d.	n.d.	n.d.	n.d.	n.d.
2,3,7,8-TCDD	<0.169	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,7,8-PeCDF	8.2672	0.4134	0.4134	0.2480	0.4134	0.8267
2,3,4,7,8-PeCDF	<0.44	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,7,8-PeCDD	<0.342	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,4,7,8-HxCDF	10.9635	1.0964	1.0964	1.0964	1.0964	1.0964
1,2,3,6,7,8-HxCDF	<0.365	n.d.	n.d.	n.d.	n.d.	n.d.
2,3,4,6,7,8-HxCDF	21.7016	2.1702	2.1702	2.1702	2.1702	2.1702
1,2,3,7,8,9-HxCDF	8.6329	0.8633	0.8633	0.8633	0.8633	0.8633
1,2,3,4,7,8-HxCDD	4.4881	0.4488	0.4488	0.4488	2.2441	0.2244
1,2,3,6,7,8-HxCDD	<0.511	n.d.	n.d.	n.d.	n.d.	n.d.
1,2,3,7,8,9-HxCDD	5.7771	0.5777	0.5777	0.5777	0.0578	0.5777
1,2,3,4,6,7,8-HpCDF	38.4134	0.3841	0.3841	0.3841	0.3841	0.3841
1,2,3,4,7,8,9-HpCDF	15.2912	0.1529	0.1529	0.1529	0.1529	0.1529
1,2,3,4,6,7,8-HpCDD	50.3583	0.5036	0.5036	0.5036	0.0504	0.0504
OCDF	68.3346	0.0683	0.0068	0.0205	0.0068	0.0068
OCDD	188.5525	0.1886	0.0189	0.0566	0.0189	0.0189
	<b>TOTAL</b>	<b>6.8672</b>	<b>6.6360</b>	<b>6.5220</b>	<b>7.4581</b>	<b>6.3717</b>

TOTALS	ng/kg
TCDFs	33.6136
TCDDs	28.1149
PeCDFs	76.1072
PeCDDs	54.4221
HxCDFs	73.5758
HxCDDs	73.8426
HpCDFs	81.0017
HpCDDs	95.4772
OCDF	68.3346
OCDD	188.5525
<b>TOTAL</b>	<b>773.0422</b>

<sup>a</sup> WHO 1998 TEQ values, <sup>b</sup> WHO 2005 TEQ values

**LABELLED STANDARD RECOVERIES**

**CLIENT** HANSON CEMENT

**CONTRACT** HANSONCEM-00244

**SAMPLE** BYPASS DUST

**LAB NO** 1208210

**REF** D7910

EXTRACTION STANDARDS	RECOVERY %
<sup>13</sup> C <sub>12</sub> 2378 TCDF	85
<sup>13</sup> C <sub>12</sub> 2378 TCDD	74
<sup>13</sup> C <sub>12</sub> 12378 PeCDF	98
<sup>13</sup> C <sub>12</sub> 23478 PeCDF	94
<sup>13</sup> C <sub>12</sub> 12378 PeCDD	99
<sup>13</sup> C <sub>12</sub> 123478 HxCDF	101
<sup>13</sup> C <sub>12</sub> 123678 HxCDF	96
<sup>13</sup> C <sub>12</sub> 234678 HxCDF	94
<sup>13</sup> C <sub>12</sub> 123789 HxCDF	88
<sup>13</sup> C <sub>12</sub> 123478 HxCDD	100
<sup>13</sup> C <sub>12</sub> 123678 HxCDD	100
<sup>13</sup> C <sub>12</sub> 1234678 HpCDF	93
<sup>13</sup> C <sub>12</sub> 1234789 HpCDF	75
<sup>13</sup> C <sub>12</sub> 1234678 HpCDD	80
<sup>13</sup> C <sub>12</sub> OCDF	51
<sup>13</sup> C <sub>12</sub> OCDD	54

All of the recoveries quoted above are within the acceptance limits of method O36.



## SECTION 3

## **EXPLANATION OF APPENDICES**

### **APPENDIX 1 CHAIN OF CUSTODY FORMS/EXTRACTION/AUTOSAMPLER LISTS**

These pages show copies of forms that document the progress of the sample from the sampling stage through all analysis stages.

The chain of custody form documents the date that the sample was taken and contains sample identification information together with records of the transfer of the sample prior to analysis.

The extraction log shows the dates of all extraction and cleanup processes, including details of the spiking standards used for analysis. The final extract volume after addition of internal standards is also shown.

The autosampler list shows the run order of the samples GCMS analysis together with the datafile names under which data is stored.

### **APPENDIX 2 GLOSSARY**

This is a list of abbreviations used in this report.

## APPENDIX 1



### SAMPLE EXTRACTION LOG

LAB NO	✓	1208210	1208337	1208338	1208339	1208340	1208341
PCBs REQD ?	✓	✓	✓	✓	✓	✓	✓
SAMPLE ID	LAB BLANK	BYPASS DUST	WS 2061	WS 2062	WS 2063	WS 2064	WS 2065
WT/VOL/TRAP	1.015g	1.010g	1.029g	1.030g	1.028g	1.002g	1.030g
SIGN	<i>[Signature]</i>						

#### <sup>13</sup>C<sub>12</sub> STD ADDITIONS

DATE/TIME	11.5.16 / 17:00						
STD	LS2180216 / PCBES1110214						
VOL	20ul / 20ul						
SIGN	<i>[Signature]</i>						

#### EXTRACTION

EXT DATES & TIMES	11.5.16 17:30	12.5.16 09:00					
GLASSWARE N°	1	2	3	4	5	6	7

#### ALUMINA CLEANUP

DATE	12.5.16						
SIGN	<i>[Signature]</i>						
PCB KEEP?	✓	✓	✓	✓	✓	✓	✓

#### MIXED SILICA CLEANUP

DATE	12.5.16						
SIGN	<i>[Signature]</i> / see						

#### OTHER CLEANUPS 1

TYPE							
DATE	N/A						
SIGN							

#### OTHER CLEANUPS 2

TYPE							
DATE	N/A						
SIGN							

#### CONCENTRATION INTO NONANE

DATE	15.5.16						
VOL	10ul						
SIGN	<i>[Signature]</i>						

#### RECOVERY STD ADDITION

DATE/TIME	15.5.16 / 14:00						
STD	182120516						
VOL	10ul						
Final VOL	20ul						
SIGN	<i>[Signature]</i>						

### SAMPLE EXTRACTION LOG

LAB NO	1208692	✓					
PCBs REQD ?	✓	✓					
SAMPLE ID	W8 2066	DX3					
WT/VOL/TRAP	1.030g	1.060g					
SIGN	<i>[Signature]</i>						

#### <sup>13</sup>C<sub>12</sub> STD ADDITIONS

DATE/TIME	11.5.16 / 17:00						
STD	LS2180216/PCBES1110214						
VOL	20ul / 20ul						
SIGN	<i>[Signature]</i>						

#### EXTRACTION

EXT DATES & TIMES	11.5.16 17:30	12.5.16 09:00					
GLASSWARE N°	8	9					

#### ALUMINA CLEANUP

DATE	12.5.16						
SIGN	<i>[Signature]</i>						
PCB KEEP?	✓						

#### MIXED SILICA CLEANUP

DATE	12.5.16						
SIGN	<i>[Signature]</i>						

#### OTHER CLEANUPS 1

TYPE							
DATE	N/A	→					
SIGN							

#### OTHER CLEANUPS 2

TYPE							
DATE	N/A	→					
SIGN							

#### CONCENTRATION INTO NONANE

DATE	15.5.16						
VOL	10ul						
SIGN	<i>[Signature]</i>						

#### RECOVERY STD ADDITION

DATE/TIME	15.5.16/16:00						
STD	LS2120516						
VOL	10ul						
Final VOL	20ul						
SIGN	<i>[Signature]</i>						

## AUTOSAMPLER RUN LIST

Date of Run 16/5/16

A/S POSITION	DATA FILE NAME	LAB NUMBER	SAMPLE DETAILS	NOTES
1	gec001	—	6CWD2 column check	
2	02	—	CS1	
3	03	—	CS2	
4	04	—	CS3	
5	05	—	CS4	
6	06	—	CS5	
7	07	—	N	
8	08	—	N	
9	09	—	N	
10	10	✓	LAB BLANK 11.5.16	
11	11	1208210	BYPASS CUST	
12	12	—	N	
13	13	1208337	WS 2061	
14	14	—	N	
15	15	1208338	WS 2062	
16	16	—	N	
17	17	1208339	WS 2063	
18	18	—	N	
19	19	1208390	WS 2064	
20	20	—	N	
21	21	1208391	WS 2065	
22	22	—	N	
23	23	1208392	WS 2066	
24	24	—	N	
25	25	—	DX3 11.5.16	
26	26	—	N	
4	✓ 27	✓	CS3	

COMMENTS N = NO WASH

## APPENDIX 2



## GLOSSARY

The following terms and abbreviations are used throughout this report.

PCDD	Polychlorinated dibenzo-p-dioxin
PCDF	Polychlorinated dibenzofuran
TCDD	Tetrachlorodibenzo-p-dioxin
TCDF	Tetrachlorodibenzofuran
PeCDD	Pentachlorodibenzo-p-dioxin
PeCDF	Pentachlorodibenzofuran
HxCDD	Hexachlorodibenzo-p-dioxin
HxCDF	Hexachlorodibenzofuran
HpCDD	Heptachlorodibenzo-p-dioxin
HpCDF	Heptachlorodibenzofuran
OCDD	Octachlorodibenzo-p-dioxin
OCDF	Octachlorodibenzofuran
HxCdPE	Hexachlorodiphenyl ether
HpCdPE	Heptachlorodiphenyl ether
OCdPE	Octachlorodiphenyl ether
NCDPE	Nonachlorodiphenyl ether
DCdPE	Decachlorodiphenyl ether
TEF	Toxic Equivalent Factor
TEQ	Toxic Equivalent
I-TEF	International Toxic Equivalent Factor (NATO/CCMS)
I-TEQ	International Toxic Equivalent (NATO/CCMS)