WHO PCB

ANALYSIS REPORT

NUMBER D7911P

1181

This analysis is based on Northumbrian Water Scientific Services Organics Laboratory method O084 which is accredited under the **UKAS** accreditation scheme.

WHO PCB

ANALYSIS REPORT

NUMBER D7911P

Laboratory Address:

Northumbrian Water Scientific Services
Northumberland Dock Road
Howdon
Wallsend
Tyne & Wear
NE28 0QD

Report Ref: D7911P

Client Address:

Victoria Smith
Hanson Cement
Padeswood
Mold
Flintshire
CH7 4HB

Prepared by: Sophie Wright **Signed:**  **Date:**22/05/16

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

CONTENTS

SECTION 1

SUMMARY

One sample taken on behalf of Hanson Cement was analysed for WHO PCB contamination. Analysis of the sample gave the following TEQ values (to 2 significant figures).

	Toxic Equivalent Results ng/kg			
Sample	**WHO (1998) HUMANS TEQ**	**WHO (2005) HUMANS TEQ**	**WHO (1998) FISH TEQ**	**WHO (1998) BIRDS TEQ**
BYPASS DUST	0.85	0.85	0.047	2.2

Full results for individual congeners together with information on any deviations from methodology/quality systems are shown in section 2.

INTRODUCTION

One sample was submitted to Northumbrian Water Scientific Services by Hanson Cement for analysis to determine the levels of the 12 polychlorinated biphenyls assigned TEQ values by the World Health Organisation (WHO PCBs).

The sample was received on 25/04/16. Details of the sample are shown below.

CONTRACT - HANSONCEM-00244

DATE REC'D	SAMPLE	LAB N^{o}	REF
25/04/16	BYPASS DUST	1208210	D7911P

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 SUMMARY

The analytical method used for this analysis, O084, is based on US EPA 1668.

Air dried soil samples are ground, spiked with a mixture of 12 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.

Impurities are removed from the extracts by acid/base back-extraction and column chromatography using silica and alumina absorbents and HPLC, any or all of the techniques being used.

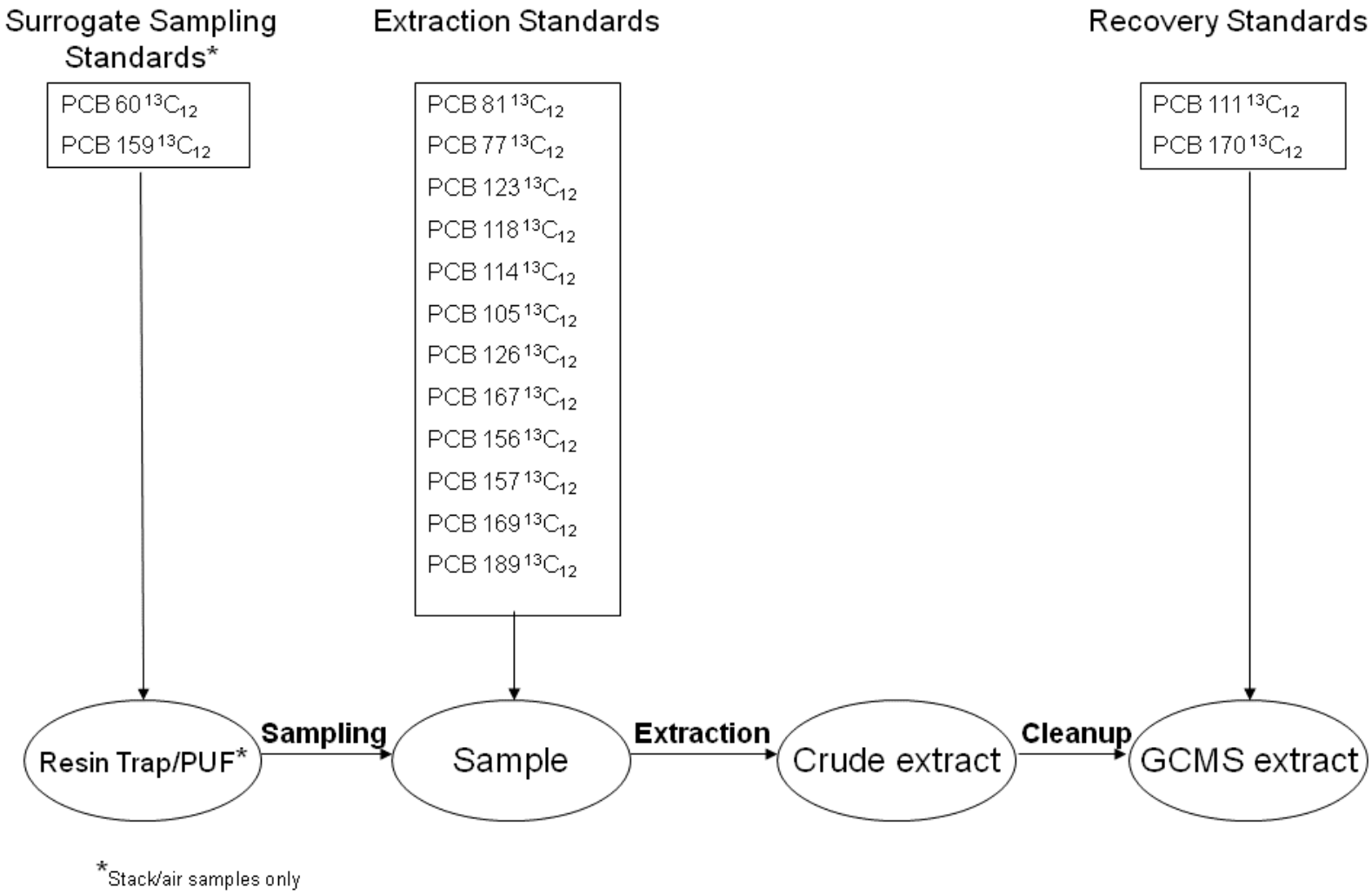
The resulting extracts are concentrated and solvent exchanged to give a final volume of 10 µl in nonane.

Two stable isotopically labelled internal standards are added before analysis by high resolution gas chromatography- high resolution mass spectrometry (HRGC-HRMS) using a DB5-MS column.

Identification of the WHO PCBs is based on comparison of GC retention times and the ion abundance ratios of the monitored mass signals with the corresponding retention times of authentic standards and the theoretical ion abundance ratios.

If results for individual targeted WHO PCBs exceed the calibration range of the instrument then these results are flagged in the analytical report. The mass spectrometer is operated at a resolution in excess of 10000 to minimize the potential for interference. Selected ions characteristic of the WHO PCBs are monitored. The mass spectrometer is continuously calibrated during acquisition to correct for any mass drift using mass signals from a reference compound FC43 (heptacosafluorotributylamine).

WHO PCB LABELLED STANDARD ADDITIONS (Method O084)



*Stack/air samples only

SECTION 2

RESULTS

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

A matrix blank is analysed alongside the samples to show any possible contamination. This consists of a sample of quartz sand.

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 – Limits of detection (LOD) 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.

All quality criteria in the method O84 have been met with any deviations outlined below.

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

WHO PCB RESULTS

CLIENT HANSON CEMENT **CONTRACT** HANSONCEM-00244

SAMPLE LABORATORY BLANK

LAB NO N/A **REF** D7911P

		Recovery of	WHO TEQ			
CONGENER	ng/kg	$^{13}C_{12}$ %	Humans[a]	Humans[b]	Fish[a]	Birds[a]
PCB BZ 81	<0.51	73	n.d.	n.d.	n.d.	n.d.
PCB BZ 77	9.36	65	0.000936	0.000936	0.000936	0.468000
PCB BZ 123	<0.2	70	n.d.	n.d.	n.d.	n.d.
PCB BZ 118	17.62	65	0.001762	0.000529	0.000088	0.000176
PCB BZ 114	<0.35	40	n.d.	n.d.	n.d.	n.d.
PCB BZ 105	<0.36	44	n.d.	n.d.	n.d.	n.d.
PCB BZ 126	<0.36	45	n.d.	n.d.	n.d.	n.d.
PCB BZ 167	<0.32	120	n.d.	n.d.	n.d.	n.d.
PCB BZ 156	<0.28	136	n.d.	n.d.	n.d.	n.d.
PCB BZ 157	<0.27	135	n.d.	n.d.	n.d.	n.d.
PCB BZ 169	<0.28	89	n.d.	n.d.	n.d.	n.d.
PCB BZ 189	<0.17	94	n.d.	n.d.	n.d.	n.d.
	TEQ TOTAL		0.002698	0.001465	0.001024	0.468176

[a] WHO 1998 TEQ values, [b] WHO 2005 TEQ values

All of the recoveries quoted above are within the acceptance limits of method O84.

WHO PCB RESULTS

CLIENT HANSON CEMENT **CONTRACT** HANSONCEM-00244

SAMPLE BYPASS DUST

LAB NO 1208210 **REF** D7911P

CONGENER	ng/kg	Recovery of $^{13}C_{12}$ %	WHO TEQ Humans[a]	WHO TEQ Humans[b]	WHO TEQ Fish[a]	WHO TEQ Birds[a]
PCB BZ 81	6.17	79	0.000617	0.001851	0.003085	0.617000
PCB BZ 77	15.01	78	0.001501	0.001501	0.001501	0.750500
PCB BZ 123	<0.34	99	n.d.	n.d.	n.d.	n.d.
PCB BZ 118	14.32	99	0.001432	0.000430	0.000072	0.000143
PCB BZ 114	1.94	95	0.000970	0.000058	0.000010	0.000194
PCB BZ 105	7.16	100	0.000716	0.000215	0.000036	0.000716
PCB BZ 126	8.46	109	0.846000	0.846000	0.042300	0.846000
PCB BZ 167	<0.25	108	n.d.	n.d.	n.d.	n.d.
PCB BZ 156	<0.25	108	n.d.	n.d.	n.d.	n.d.
PCB BZ 157	<0.25	108	n.d.	n.d.	n.d.	n.d.
PCB BZ 169	<0.2	93	n.d.	n.d.	n.d.	n.d.
PCB BZ 189	2.84	91	0.000284	0.000085	0.000014	0.000028
	TEQ TOTAL		0.851520	0.850140	0.047017	2.214582

[a] WHO 1998 TEQ values, [b] WHO 2005 TEQ values

All of the recoveries quoted above are within the acceptance limits of method O84.

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

PAGE No. 1/3

NWSS AIR E[illegible]ISSIONS SAMPLE SUBMISSION [illegible]EET

NORTHUMBRIAN WATER Scientific Services

CLIENT: MANSON CEMENT **SITE:** PADESWOOD	**NWSS CONTRACT REF:** MANSONCEM-00244		
CLIENT PROJECT MANAGER: VICTORIA SMITH **TEL:**	**NWSS PROJECT MANAGER (+ MOBEX):** JOHN McBRIDE (73567)	**AGREED TURNROUND:** (No of working days):	Howdon (0191) 2968500 Cwmbran (01633) 862950

MATRIX				STACK, TEST & RUN NUMBER	SAMPLER	COMMENTS / SOLUTION / FILTER	ANALYSIS / TEST SCHEDULE REQUIRED	DATE SAMPLED	TIME SAMPLED	NWSS LAB NO
Liquid	Sludge	Soil	Gas							
			✓	COOLER STACK DUST BLANK	JM/MD	FILTER 16LD080	CEN PARTICULATES	18/04/16	13.00	1208178
			✓	COOLER STACK DUST BLANK	JM/MD	ACETONE / H_2O	CEN PARTICULATES	18/04/16	13.00	1208179
			✓	COOLER STACK DUST RUN 1	JM/MD	FILTER 16LD089	CEN PARTICULATES	18/04/16	13.20	1208180
			✓	COOLER STACK DUST RUN 1	JM/MD	ACETONE / H_2O	CEN PARTICULATES	18/04/16	13.20	1208181
			✓	COOLER STACK DUST RUN 2	JM/MD	FILTER 16LD081	CEN PARTICULATES	19/04/16	08.36	1208182
			✓	COOLER STACK DUST RUN 2	JM/MD	ACETONE / H_2O	CEN PARTICULATES	19/04/16	08.36	1208183
			✓	COOLER STACK PM10 BLANK	JM/MD	STAGE 1 FILTER 16LD ~~082~~ 117 JM	CEN PARTICULATES	19/04/16	09.45	1208184
			✓	COOLER STACK PM10 BLANK	JM/MD	STAGE 2 FILTER 16LD ~~121~~ 118 JM	CEN PARTICULATES	19/04/16	09.45	1208185
			✓	COOLER STACK PM10 BLANK	JM/MD	BACK UP FILTER 16LD ~~122~~ 119 JM	CEN PARTICULATES	19/04/16	09.45	1208186
			✓	COOLER STACK PM10 RUN 1	JM/MD	STAGE 1 FILTER 16LD120	CEN PARTICULATES	19/04/16	10.00	1208187
			✓	COOLER STACK PM10 RUN 1	JM/MD	STAGE 2 FILTER 16LD121	CEN PARTICULATES	19/04/16	10.00	1208188
			✓	COOLER STACK PM10 RUN 1	JM/MD	BACK UP FILTER 16LD122	CEN PARTICULATES	19/04/16	10.00	1208189
		✓		BYPASS DUST	SITE		WILSA 00409	19/04/16	09.30	1208210
			✓	CEMENT MILL 2 BLANK	JM/MD	FILTER 16LD078	CEN PARTICULATES	19/04/16	12.30	1208190
			✓	CEMENT MILL 2 BLANK	JM/MD	ACETONE / H_2O	CEN PARTICULATES	19/04/16	12.30	1208191
			✓	CEMENT MILL 2 RUN 1	JM/MD	FILTER 16LD084	CEN PARTICULATES	19/04/16	13.05	1208192

CHAIN OF CUSTODY

Relinquished By	Date	Received By
[signature]	22/4/16	[signature]
Relinquished By	Date	Received By
Relinquished By	Date	Received By

ADDITIONAL INFORMATION\HAZARD DATA

ENTERED BY:	[signature]
ON (DATE):	25/4/16
NO. OF SAMPLES CORRECT?	✓
IF NO THEN REPORTED TO:	

COMPLETED FORM TO BE SENT TO:	TICK BOX	ACTIONED BY
JOHN McBRIDE	✓	[signature]
CWMBRAN OFFICE (FAX 01633 865087)		

SAMPLE EXTRACTION LOG

LAB NO	—	1208210	1208587	1208588	1208589	1208590	1208591
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							

$^{13}C_{12}$ STD ADDITIONS

DATE/TIME	11.5.16/17:00						
STD	LSS2.180216/PCBES1110214						
VOL	20ul/20ul						
SIGN							

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							
PCB KEEP?	✓	✓	✓	✓	✓	✓	✓

MIXED SILICA CLEANUP

DATE	12.5.16						
SIGN	/Bee						

OTHER CLEANUPS 1

TYPE							
DATE	N/A						
SIGN							

OTHER CLEANUPS 2

TYPE							
DATE	N/A						
SIGN							

CONCENTRATION INTO NONANE

DATE	16.5.16						
VOL	10ul						
SIGN							

RECOVERY STD ADDITION

DATE/TIME	16.5.16/14:30						
STD	PCBRS22.11215						
VOL	10ul						
Final VOL	20ul						
SIGN							

SAMPLE EXTRACTION LOG

LAB NO	1208592	—					
PCBs REQD ?	✓	✓					
SAMPLE ID	WS 2066	DX3					
WT/VOL/TRAP	1.030g	1.086g					
SIGN							

$^{13}C_{12}$ STD ADDITIONS

DATE/TIME	11.5.16 / 17:00						
STD	LS21802216/PCBES1110214						
VOL	20µl / 20µl						
SIGN							

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							
PCB KEEP?	✓	✓					

MIXED SILICA CLEANUP

DATE	12.5.16						
SIGN							

OTHER CLEANUPS 1

TYPE	N/A →						
DATE							
SIGN							

OTHER CLEANUPS 2

TYPE	N/A →						
DATE							
SIGN							

CONCENTRATION INTO NONANE

DATE	16.5.16						
VOL	10µl →						
SIGN							

RECOVERY STD ADDITION

DATE/TIME	16.5.16 / 14:30						
STD	PCBRS2.211215						
VOL	10µl						
Final VOL	20µl						
SIGN							

AUTOSAMPLER RUN LIST

Date of Run 20/5/16

A/S POSITION	DATA FILE NAME	LAB NUMBER	SAMPLE DETAILS	NOTES
2	hec001	—	PCBCS1 COLUMN CHECK	
2	02		PCBCS1	
3	03		PCBCS2	
4	04		PCBCS3	
5	05		PCBCS4	
6	06		PCBCS5	
7	07		N	
8	08		N	
9	09		N	
10	10	↓	LAB BLANK 11.5.16	
11	11	1208210	BYPASS DUST	
12	12	—	N	
13	13	1208587	WS 2061	
14	14	—	N	
15	15	1208588	WS 2062	
16	16	—	N	
17	17	1208589	WS 2063	
18	18	—	N	
19	19	1208590	WS 2064	
20	20	—	N	
21	21	1208591	WS 2065	
22	22	—	N	
23	23	1208592	WS 2066	
24	24	—	N	
25	25		DX3 11.5.16	
26	26		N	
4	27		PCBCS3	
4	28		PCBCS3	
29	29	↓	N	
11	30	1208210	BYPASS DUST	REPEAT
31	31	—	N	
17	32	1208589	WS 2063	REPEAT

COMMENTS N = NONANE WASH.

AUTOSAMPLER RUN LIST

Date of Run 20/5/16

A/S POSITION	DATA FILE NAME	LAB NUMBER	SAMPLE DETAILS	NOTES
33	hec033	—	N	
4	↓ 034	↓	PCBCS3	

COMMENTS N = NONANE WASH.

APPENDIX 2

GLOSSARY

The following terms and abbreviations are used throughout this report.

WHO	World Health Organisation
PCB	Polychlorinated biphenyl
TEF	Toxic Equivalent Factor
TEQ	Toxic Equivalent
I-TEF	International Toxic Equivalent Factor (NATO/CCMS)
I-TEQ	International Toxic Equivalent (NATO/CCMS)
LOQ	Limit of Quantitation